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Sealing efficiency of caprocks: Experimental investigation of entry pressure measurement methods

P.F. Boulin*, P. Bretonnier, V. Vassil, A. Samouillet, M. Fleury, J.M. Lombard

IFP Energies nouvelles, 1 et 4 avenue du bois prau, 92852 Rueil-Malmaison Cedex, France

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ABSTRACT

Caprocks play a key role in hydrocarbon entrapment and in the geological storage of gas. Top seals inhibit vertical migration due to their low permeability and high entry pressure (PE). This study investigated four different techniques for measuring PE: (1) step by step method; (2) dynamic approach; (3) racking method; (4) residual pressure method. This article reports results on two samples: a carbonate (1.5 μDarcy (1.5 10^{-18} m^2)) and a claystone (15 nDarcy (1.5 10^{-20} m^2)). On the carbonate sample, methods 1, 2 and 3 gave a PE value of 1.1 MPa, whilst method 4 gave a PE of 0.4 MPa. On the claystone sample, methods 1 and 2 gave a PE value around 12 MPa. The data allow us to consider best practices for PE measurements on caprocks. Methods 2 and 3 are the quickest and most accurate methods but show limitations in very low permeability porous media. Method 2 required three days to measure PE in the 15 nD claystone and experiments on 1 nD (10^{-21} m^2) materials would take longer. Additional issues on mechanical stresses impact the result reliability since in methods 2 and 3 effective stress can significantly change during the experiment. Method 4 measures a snap-off pressure that is lower than the entry pressure value. Method 1 is a long experiment but is the most representative of *in situ* hydrocarbon migration though caprocks.

1. Introduction

Caprocks form barriers above hydrocarbon reservoirs, inhibiting upwards migration due to their low permeability and high entry pressures (PE). PE is the pressure of a non-wetting fluid required to displace water within the rock. If the pressure does not overcome capillary forces generated by the caprock pores, non-wetting fluids such as hydrocarbons, CO_2 and H_2 cannot penetrate further into the formation and do not leak (Foh et al., 1979; Ingram et al., 1997; Al-Bazali et al., 2005; Bachu, 2008; Fleury et al., 2010; Galle, 2000; Busch and Muller, 2011; Naylor et al., 2010). In this article, the concept of PE will be discussed for gas, but it is equally relevant to oil.

The capillary pressure (P_c) in a cylindrical tube with radius r is given by Laplace's law:

$$P_c = P_g - P_w = 2\sigma_w \frac{\cos(\theta)}{r} \tag{1}$$

where P_g and P_w are the gas (i.e. non-wetting) phase and the water (i.e. wetting) phase pressure (Pa), respectively. Both the contact angle (θ) and the surface tension (σ_w, N m^{-1}) depend mainly on the liquid/gas couple. Laplace's law describes the local mechanical equilibrium between the gas and water phases. If the gas pressure increases at one side of the tube, water will be expelled. We can therefore define a PE value:

$$\mathrm{PE} = 2\sigma_w \frac{\cos(\theta)}{r} \tag{2}$$

If the pressure values of each side of the tube give $P_c > \mathrm{PE}$, the gas will start to displace the water. On the other hand, if $P_c < \mathrm{PE}$, the tube remains water-filled. If the porous media is made of parallel cylindrical pores of different sizes, PE is simply related to the largest pore size. Real porous media are far more complex so that pore connectivity, pore shape, the scale of observation and sedimentological heterogeneity all influence the PE at which a non-wetting fluid breaches the rock sample (Carruthers and Ringrose, 1998). The PE at which a rock volume is breached has been given several names in the literature, including threshold pressure, breakthrough pressure, displacement pressure and critical pressure. The precise meaning of these terms varies according on the authors and on the experiments carried out, and a good overview is available in

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* Corresponding author. Tel.: +33 1 47 52 54 45; fax: +33 1 47 52 70 72.
E-mail address: pierre.boulin@ifpen.fr (P.F. Boulin).

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Hildenbrand (2003). Although a variety of definitions for PE exists, here we differentiate two main concepts: threshold pressure and breakthrough pressure. The threshold pressure is the gas pressure at which water is first displaced from the largest pores of a water-saturated sample. From an experimental point of view, the pore network invaded by the gas should be large enough for the volume of water expelled from the sample to be observed. At this stage, however, due to local heterogeneities or to the scale of observation, the network invaded by the gas may not reach the other side of the sample, so that there is no breakthrough. Experimentally, gas pressure can be increased in order to measure a breakthrough pressure, which corresponds to the capillary pressure of the largest interconnected pores across the rock volume of interest. In this paper, we assumed a monomodal and homogeneous porous medium where breakthrough pressure and threshold pressure are equal and defined as PE. This issue will be discussed later.

The accurate evaluation of PE values in low permeability materials can be a very long process (Busch and Muller, 2011). Reviews of existing methods is available in Hildenbrand (2003), Egermann et al. (2006) and Busch and Muller (2011). The main technique used to determine PE in low permeability media is the step by step approach initially proposed by Thomas et al. (1968). Gas is placed in contact with the upstream surface of a water-saturated sample. Initially, the gas pressure is equal to the pore pressure and is then increased in steps. Choices of pressure steps and duration depend on sample permeability and on the accuracy required for the PE estimate. When the capillary pressure (gas pressure minus pore pressure) is higher than PE, water is displaced out of the sample. A continuous flow of water results, ultimately followed by a free gas phase (Tonnet et al., 2010). A similar experiment consists of closing the downstream reservoir and observing pressure increases due to gas penetration (Al-Bazali et al., 2005). In very low permeability porous media, the pressure increase is controlled by the compressibility of the downstream reservoir and the pressure change observation is more challenging than a direct measurement of the water flow rate (Boulin et al., 2012). An advanced technique involves the upstream injection of gas at a constant rate (Rudd and Pandey, 1973; Horseman et al., 1999). Inlet gas pressure increases until gas breakthrough. This has the advantage of including the whole pressure range and is very effective in high permeability porous media when PE values of less than 0.1 MPa have to be measured. In very low permeability porous media, such as caprocks, the gas flow rate must be low enough to ensure that the gas has the time to displace water. Otherwise, the gas pressure will be higher than PE when water outflow is noticed (Boulin, 2008).

PE values can be evaluated roughly using correlations such as the relationship between CEC and PE (Al-Bazali et al., 2005) or the relationship, based on empirical data, between permeability and PE (Thomas et al., 1968) (PE in psi and k in mD, PE between 0.2 and 4 MPa and permeability from 50 nD to 0.2 μD):

$$\mathrm{PE} = 7.37\left(\frac{1}{k}\right)^{0.45} \tag{3}$$

Mercury intrusion curves can also provide an estimate of the PE value (Monicard, 1981; Carles et al., 2007). The mercury intrusion test is a well established technique that has been used for years to provide pore diameter distributions in porous media. Mercury injection capillary pressure analysis has been used extensively in the petroleum industry to determine the effectiveness of the top seal in relation to hydrocarbon column height (Daniel and Kaldi, 2008); a review is available in Rootare (1970). The sample is surrounded by mercury. Mercury pressure is increased in steps, invading increasingly small pores, so that the volume intruded for a given pressure is representative of the diameter distribution of the porous structure. The mercury, as the non-wetting phase, invades the porous media as a gas would do when its pressure increases. The PE is obtained graphically; an example will be provided in the results section. It corresponds to the PE for the mercury/air couple and should be changed accordingly for the non-wetting/wetting phases studied. Equation (2) is used as a reference and PE has to be recalculated with the appropriate wettability and surface tension values. For CO_2 (Tonnet et al., 2010) and for hydrocarbons, pore water composition and pore surface affinity to the fluid (mineralogy, asphaltene deposition, …) can affect the rock wettability and should be assessed. In addition, for CO_2, surface tension is a function of pressure (Chiquet, 2006). MICP is a fast and simple method and can be used when retrieving preserved cores is difficult. Furthermore, it can provide a valuable PE order of magnitude so that experiments on cylinder cores can be designed properly. However, the method may not accurately assess PE (Egermann et al., 2006) since:

- samples can be affected by drying conditions;
- MICP is an isobaric experiment carried out at conditions of zero effective stress, so that the sample is in a mechanical state that is far from its *in situ* state. We show in this study that PE values are indeed affected by the stress state at which measurements are made.

The dynamic method, originally introduced by Egermann et al. (2006), is another technique for measuring the PE. Prior to testing, the sample is fully saturated. Gas is injected upstream at a constant pressure P_g. On the upstream reservoir, the gas displaces the water until the gas is in contact with the sample surface. Downstream pressure is maintained constant at P_w throughout the experiment. The order of magnitude of PE should be known as the gas pressure should be high enough to allow the gas to penetrate the sample. Two different flow rates are observed: before and after gas entry. Before gas entry, water is produced due to a pressure gradient within the sample equal to $\Delta P_1 = P_g - P_w$. When gas enters the sample, owing to the capillary forces, pore pressure drops. Upstream pressure drops from P_g to P_g − PE. P_g − PE should be higher than P_w, otherwise the water stops moving and no flow will be observed. The new pressure gradient is thus $\Delta P_2 = P_g - \mathrm{PE} - P_w$. The flow rate is measured continuously and since P_g, P_w are known values, using Darcy's law, PE can be estimated from Q_2 (m^3/s), the second observed flow rate:

$$Q_2 = -\frac{k_w}{\mu_w}\frac{S}{e}\Delta P_2 \tag{4}$$

where k_w is the sample permeability (m^2), S is the cross sectional area (m^2), e is the sample length (m) and μ_w is the water viscosity (Pa s). k_w can be estimated by the Darcy's law applied to the first flow rate Q_1 if the pressure gradient ΔP_1 is known.

The racking method (Meyn, 1999) is similar to the dynamic approach except that a pump placed downstream extracts water at a constant water flow rate. This method is a three-step process. First, water moves from the upstream reservoir to the downstream reservoir. As would be the case with a simple steady state experiment, downstream pressure reaches equilibrium. After that, when gas starts to come into contact with the sample surface, pore pressure is still too high to allow gas to enter the sample. Downstream pore pressure starts to decrease due to the constant flow rate extracted by the pump. Since pressure wave propagation is a fast process even in very low permeability rocks ((Boulin et al., 2012), this statement will be discussed later in the article), when downstream pressure starts to decrease, pore pressure within the sample decreases too. The third step happens when pore pressure