



## Permeability evolution of shale during spontaneous imbibition



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### ABSTRACT

Shales have small pore and throat sizes ranging from nano to micron scales, low porosity and limited permeability. The poor permeability and complex pore connectivity of shales pose technical challenges to (a) understanding flow and transport mechanisms in such systems and, (b) in predicting permeability changes under dynamic saturation conditions. This study presents quantitative experimental evidence of the migration of water through a generic shale core plug using micro CT imaging. In addition, in-situ measurements of gas permeability were performed during counter-current spontaneous imbibition of water in nano-darcy permeability Marcellus and Haynesville core plugs. It was seen that water blocks severely reduced the effective permeability of the core plugs, leading to losses of up to 99.5% of the initial permeability in experiments lasting 30 days. There was also evidence of clay swelling which further hindered gas flow. When results from this study were compared with similar counter-current gas permeability experiments reported in the literature, the initial (base) permeability of the rock was found to be a key factor in determining the time evolution of effective gas permeability during spontaneous imbibition. With time, a recovery of effective permeability was seen in the higher permeability rocks, while becoming progressively detrimental and irreversible in tighter rocks. These results suggest that matrix permeability of ultra-tight rocks is susceptible to water damage following hydraulic fracturing stimulation and, while shut-in/soaking time helps clearing-up fractures from resident fluid, its effect on the adjacent matrix permeability could be detrimental.

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### 1. Introduction

The fusion of horizontal well and hydraulic fracture technology in the early 2000s was crucial to unlocking the vast oil and gas resource that shales are. Shales now contribute over 40% of the total US natural gas production and its share is expected to rise to 55% by 2040 (EIA, 2016). But despite this massive production boom, the interpretation and management of shales as productive hydrocarbon deposits is still relatively new. In the past, petrophysical assessment of shales was done to evaluate their efficacy as reservoir caps and seals (Katsube et al., 1991) or to address wellbore integrity issues (Horsrud et al., 1998). Today, we possess the tools to produce from these formations but still have scant understanding of the fundamental physics of flow in ultra-tight and nano-porous materials. As a consequence, about one in three shale gas wells have poor production characteristics (Kovscek and Majumdar, 2015). A

big opportunity, therefore, exists to adapt and improve current reservoir characterization and interpretation in terms of storage and deliverability of shale formations.

Recent studies have focused on characterizing shales at the pore scale and they suggest a complex quad-media porous structure with different porosity, permeability, and wettability domains (e.g., Civan et al., 2012). Experimental work can build on this knowledge to help understand the interactions between these different structural domains, in order to build an appropriate effective medium description of shale. In addition, multiphase flow in shales is an important area of study to understand fluid migration, and shed light on the fate of unrecovered fracturing fluids from stimulation operations and their effect on productivity (Lan et al., 2014; Ghanbari and Dehghanpour, 2016). Spontaneous fracturing fluid (mostly water) uptake by shales can be attributed to capillary forces, adsorption, or osmosis (Zhou et al., 2016; Ghanbari and Dehghanpour, 2016). This may cause time-dependent reduction of gas permeability (Bahrami et al., 2011; Pagels et al., 2013), or improve permeability by fracturing caused by clay swelling (Morsy

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and Sheng, 2014).

Simulation tests of fracture-face damage due to fluid invasion, by Holditch (1979), concluded that only when the fracture-face permeability reduction is greater than 99%, will there be any significant productivity impairment in conventional gas wells. Cho et al., 2013 set this threshold at 95% for shale wells. Both Holditch, 1979 and Li et al., 2012 are of the opinion that this level of permeability loss is unlikely to be caused by fracture fluid and leak off. Numerous experimental studies have shown that the permeability in tight rocks can fall by 50%–90% due to the introduction of water blocks (Odumabo et al., 2014; Bostrom et al., 2014; Yan et al., 2015). Some studies on tight sandstones indicate that the lost permeability can recover over time, after injection is ceased (Odumabo et al., 2014; Bostrom et al., 2014). This is mainly due to the redistribution of fluids via spontaneous imbibition that results in a reduction of water saturation within the invaded zone. However, a recent experimental study by Yan et al., (2015) found little-to-no permeability improvement in the case of shale.

To our knowledge, there is no work in the literature correlating the evolution of saturation fronts during spontaneous imbibition in shales and their impact on effective permeability to gas. Micro-computed tomography (CT) technology can be used to monitor fluid migration in shales, dynamically and non-destructively. Therefore, this paper presents an experimental study of spontaneous water imbibition in ultra-tight shales, as well as time-dependent gas permeability measurements to assist in filling this knowledge gap. Results from this work are also compared against other in-situ measurements of time-dependent permeability during imbibition (Yan et al., 2015; Odumabo et al., 2014) to investigate permeability evolution relative to the initial, absolute permeability of the rock, hereafter referred to as “base” permeability.

## 2. Methodology

### 2.1. Sample preparation and mineral composition characterization

Samples of three different shales were extracted from slabbed well cores of 2.75 cm radius. Smaller plugs were cored for gas permeability measurements (2.5 cm in diameter and ranging 1.75–6.25 cm in length) and for monitoring of saturation profiles during imbibition (1.27 cm in diameter and 2.8 cm in length). All samples were heated in an oven for 24 h at 60 °C in order to dry out any water that might have been introduced during the coring process. The physical properties and dimensions of the tested shale samples are given in Table 1. While we have attempted to measure matrix permeability without inducing artifacts such as cracks, it was difficult to completely prevent induced fracturing during sample preparation. However, no fractures larger than 9.6 μm (CT voxel resolution) were observed, with the exception of samples G1 and M2.

X-ray diffraction (XRD) tests for Haynesville and Marcellus samples are also shown in Fig. 1. Samples were crushed and analyzed in a PANalytical Xpert Pro MPD instrument. These

revealed Calcite, Quartz, Muscovite, Pyrite and clay minerals – particularly albite – to be the main constituent minerals for both rocks, which suggests limited clay swelling during exposure to water. However, based on experiments and modelling of unconsolidated bead packs with dispersed clays, Aksu et al., 2015 found that both increasing clay content and lowering porosity serves to exacerbate the permeability reduction due to imbibition related swelling. It is reasonable to assume that given their extremely low effective porosity, shales are also susceptible to similar permeability reduction even with modest clay content or minor swelling.

### 2.2. Experiment 1 - Gravity assisted imbibition

A gravity assisted imbibition experiment was set up to obtain saturation profiles as a function of time, along the length of the sample. This was done to get a general sense of the time scale of liquid migration in ultra-tight shales. As the first step, the shale sample G1 was jacketed and placed vertically in a glass vial (Fig. 2). The dry sample was then scanned at a voxel resolution of 9.6 μm, using X-ray micro CT imaging in a GE Phoenix V Tome-X scanner running at 100 kV voltage and 100 μA current. Thereafter, a column of water, with 5% dissolved KI as the X-ray contrast enhancer, was placed above the sample and the vial was sealed. X-ray micro CT scanning during imbibition was used to monitor the migration and spreading of the water front at selected time periods: immediately after the introduction of water, after 5 h, 48 h and 7 days into the experiment. Each scan lasted 3 h.

### 2.3. Experiment 2 – Permeability evolution during spontaneous imbibition

Experiment 2 consisted of initiating a spontaneous imbibition front in selected core samples while countercurrent gas permeability measurements were performed as a function of time. Fig. 3 illustrates the experimental procedure in three sequential steps.

The first step of the process was to measure the permeability of the dry core plug in order to obtain a base permeability using Argon gas. Next, 5% KI solution was injected at ~13.8 MPa (2000psi) for several hours in order to establish a zone of high water saturation near the downstream face of the sample. These conditions allowed for a comparable depth of penetration of the KI solution to those reported in previous experiments using tight sands and shale cores, which were used for comparison purposes. The water injection time was approximately 18hr, 2.5min, and 4.5 h for samples M1, M2 and H1, respectively. Injection was then stopped and there were no further pressure gradients applied across the samples to force liquid flow. This allowed for all subsequent imbibition and redistribution of water to be spontaneous. Counter current gas permeability measurements were taken periodically at intervals of ~24 h.

Attempts to accurately measure the introduced water saturation, both volumetrically and via imaging, failed because of the ultra-low porosity of the samples and poor X-ray signal-to-noise ratio within the pressure vessel. Therefore, these are not reported

**Table 1**  
Physical properties and dimensions of tested shale samples.

Rock Type	Porosity <sup>a</sup>	Sample	Flow vs Lamination	Diameter (cm)	Length (cm)	Base Permeability (nd)
Marcellus	1.50%	M1	Parallel	2.54 (1")	6.25 (2.5")	58.6
		M2	Parallel		4.75 (1.9")	19098 (19.1 μD)
		M3	Perpendicular		1.75 (0.7")	Too Low to Measure
		M4	Perpendicular		0.20 (0.08")	≤50
Haynesville	3.20%	H1	Parallel		4.50 (1.8")	173
Generic	N/A	G1	Parallel	1.27 (0.5")	2.8 (1.1")	N/A

<sup>a</sup> Average porosity of multiple rock fragments using Mercury Intrusion Porosimetry.

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