



Initial water saturation and imbibition fluid affect spontaneous imbibition into Barnett shale samples



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ABSTRACT

Spontaneous imbibition has been widely investigated due to its significant impact on gas/oil recovery from conventional sandstone and carbonate reservoirs. Unlike conventional reservoir rocks with relative high porosity and permeability, shale is characterized by its low porosity (<10%), ultra-low permeability ($\sim 10^{-18}$ – 10^{-23} m²), complex wettability, and strong heterogeneity at multiple scales. This study is to understand the effects of initial water saturation and imbibing fluid on spontaneous imbibition of Barnett shale core samples taken from three different depths of Blakely #1 well in Wise County of Texas. Two partial, and yet uniform, water saturations of shale samples were achieved by equilibrating the samples inside closed containers at specific relative humidity controlled with over-saturated salts. The effect of imbibing fluids (water or oil-phase n-decane) on spontaneous imbibition behavior is evaluated by conducting replicate experiments. Our results show that the spontaneous imbibition behavior of shale samples could be affected by the initial water saturation conditions, and the effects are different for shale samples with different mineral compositions. The imbibition curves of shale samples with a high clay content show a poor reproducibility during water imbibition experiments due to the water sensitivity of clay minerals. In addition, n-decane could not be completely removed after its imbibition experiment. The interaction between remnant n-decane and shale matrix may change the sample wettability towards more oil-wet, which is indicated by a higher imbibition slope of second n-decane imbibition experiment on the same sample. Overall, this work helps understand the mechanism controlling fluid loss and ultimate gas/oil recovery in unconventional hydrocarbon exploration, which is significant for optimizing the selection of fracturing and treatment fluids to enhance recovery rate and minimize environmental risk.

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1. Introduction

With the development of hydraulic fracturing technology and multi-stage horizontal wells, a successful exploration of natural gas from shale reservoirs first occurred in US around 2000. During the exploration process of shale gas/oil, a large volume of fracturing fluid containing chemicals and proppants is injected into the shale formation to stimulate and keep the induced fractures open for gas/oil recovery in tight shale formations. However, only a small fraction of fracturing fluid could be recovered, and some studies have been conducted to investigate the fluid loss mechanism and

evaluate the environmental risk of lost fluid (e.g., Roychaudhuri et al., 2013; Ghanbari and Dehghanpour, 2016). Capillary-force controlled spontaneous imbibition (SI) is an important mechanism controlling the fluid loss and ultimate gas/oil recovery (Bahrami et al., 2012; Dehghanpour et al., 2013), and the increase of initial gas production rate after a long shut-in period is probably related to the effective imbibition of fracturing fluid into shale matrix (Cheng, 2012). Furthermore, spontaneous imbibition by itself is defined for a wetting fluid on a wetting porous media, while shales exhibit different wetting characteristics (Hu et al., 2015). Consequently, it is essential to investigate the imbibition characteristics of shale and the controlling factors of SI process related to water saturation and shale wettability (Dullien, 1998; Civan, 2010; Gao and Hu, 2015).

Compared to conventional reservoirs, the SI characteristics of

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shale, mainly composed of brittle minerals, clay minerals and organic matter, is more complicated. The fluid-shale interactions, including clay swelling, mineral dissolution and water/oil adsorption, could alter the pore structure of shale and then affect the gas/oil recovery process (Dehghanpour et al., 2012).

Recently, an increasing number of studies have been published with a focus on the SI characteristics of shale samples. Roychaudhuri et al. (2013) conducted SI experiments of shale samples from Appalachian Basin using water and surfactant solutions to investigate the role of capillarity in fluid-loss mechanism, and they concluded that fluid-loss during hydraulic fracturing could be explained at least partly by imbibition processes. Dehghanpour et al. (2013) investigated the SI of deionized water, brine (potassium chloride solution of various concentrations) and oil (kerosene and iso-octane) in gas shales, and they attributed the observed water intake excess to water adsorption by clay minerals and adsorption-induced microfractures. Gao and Hu (2016) characterized the wettability characteristics of Barnett shale at different depths using the directional SI experiments, and they correlated the derived wettability to pore structure and mineralogy of shale samples.

The rate of spontaneous imbibition could be significantly affected by the initial water saturation due to its impact on capillary pressure and viscous resistance to flow of reservoir fluids (Hu et al., 2002; Li et al., 2006; Viksund et al., 1998; Akin et al., 2000; Cai et al., 2010). The effect of initial water saturation on SI process of conventional reservoir rocks has been widely investigated, and different experimental results were observed for different rocks (Cil et al., 1998; Zhou et al., 2000). However, very few studies investigated the effect of initial water saturation on SI process of shale. The in-situ state of water saturation is about 25% for organic-rich Barnett shale (Bowker, 2003) and 20% for the Horn River shale (Apache Corp, 2014). Ghanbari and Dehghanpour (2015) found that initial water saturation decreased the water uptake while did not affect oil imbibition process for shale samples from Otter Park and Evie formations, whose clay mineralogy was not identified. The mixed layer clay minerals (illite/smectite, I/S) have a larger water-swelling potential compared with illite (Hensen and Smit, 2002). The swelling of smectite clays could significantly affect shale stability during well drilling operations and many studies about clay swelling and inhibition have been conducted for this reason (Anderson et al., 2010).

This study appears to be the first attempt to investigate the effect of initial water saturation on SI process of Barnett shale of different clay types, and to clarify the effect of oil/water phase on SI process of Barnett shale by conducting replicate imbibition experiments.

2. Materials and methods

Barnett shale samples were obtained from the core repository of the Texas Bureau of Economic Geology (BEG). And our samples were taken from Blakely 1 well at depths below ground surface of 7136, 7169 and 7219 ft (2175, 2185, and 2200 m, respectively); they are termed as B7136, B7169, and B7219 in this paper. Loucks and Ruppel (2007) presented the location and description of Blakely 1 well, and X-ray Diffraction analyses of these samples were provided by Gao and Hu (2016).

Firstly, we conducted contact angle measurements and qualitative drop tests to assess the wettability characteristics of our Barnett samples. During the contact angle measurement, a sample of about 15 mm size across was divided into a grid system at about 3.0 mm × 2.5 mm for each grid unit. The contact angle measurements with a deionized water drop in each grid were performed using VCA Optima system (AST Products Inc., Billerica, MA). Data

analyses performed using ImageJ (<http://imagej.nih.gov/ij/>) and the “Drop analysis - LB-ADSA” plugin (<http://bigwww.epfl.ch/demo/dropanalysis/>), and the results were plotted using Python and Matplotlib. During the fluid drop test, a small drop (about 0.015 mL and 3 mm in diameter) of DI water or n-decane was applied onto a saw-cut flat piece of slab sample at about 10 mm × 10 mm cross-section, and its spreading behavior was documented by still pictures and videos. On the scale of 1–10, the degree of spreading of a fluid was assessed. “10” stands for a “perfect” spreading which covers the entire field of view and “1” means no movement outwards from the drop.

The SI apparatus was illustrated in Gao and Hu (2012) with a semi-microbalance at 0.01 mg resolution (Model AUW220D, Shimadzu), and the experimental procedure and data processing method follow Hu et al. (2001). Core samples were cut into rectangular prisms of about 1.5 cm, and all sides except bottom and top were coated with quick-cure transparent epoxy to generate co-current imbibition, during which displaced air moves in the same direction with imbibed liquid. The exact dimensions of tested samples and other important properties are listed in Table 1. Either deionized water or n-decane (a representative oil phase) was used as the imbibing fluid to displace air in pore spaces. For replicate water or n-decane SI experiments, each cube sample was dried at least 48 h under 60 °C inside an oven to obtain an initially dry state before starting another SI experiment. Triplicate water imbibition experiments were conducted on the same sample to evaluate the effect of water imbibition on pore structure.

In order to investigate the effect of initial water saturation on SI process, three samples from each depth were prepared under three different initial saturations: drying at 60 ± 2 °C and relative humidity (RH) of about 5.9 ± 0.4% (with a corresponding capillary pressure of 435 MPa, according to Kelvin's equation), partial water-saturation by equilibrating the samples at 75% (38.7 MPa) or 98% (2.75 MPa) under 22.5 °C. Partial initial water saturation was achieved by hydrating the 60 °C oven-dried shale samples inside a closed chamber with a constant RH value. The chamber RH was controlled by an over-saturated salt solution, and monitored by a portable RH/temp datalogger (Model 42270, EXTECH). These RH chambers were held at a constant temperature of 22.5 °C inside an incubator. In this study, over-saturated NaCl and CaSO₄ solutions were selected for a reported equilibrium RH of 75.3% (Rockland, 1960) and 98% (ASTM, 1971), respectively. The samples in these controlled RH chambers were biweekly weighed until they reached a stable weight (i.e., uniform saturation inside the sample). After 54 days, equilibrium (weight change less than ±0.01%) was attained and these partially-saturated shale samples were used for SI tests after more than 4 months' water vapor equilibration; only one imbibition test was conducted for these partially but uniformly saturated samples.

3. Theory of spontaneous imbibition

Spontaneous imbibition is a capillary-force controlled process during which a wetting phase displaces a non-wetting phase under the influence of capillary force only.

Handy (1960) proposed the following equation (Eq. (1)) to describe the SI process in a water-air system during which imbibition occurs vertically upward,

$$Q_w^2 = \left(\frac{2P_c k_w \phi A^2 S_w}{\mu_w} \right) t \quad (1)$$

where Q_w is the volume of water imbibed into the sample (m³); P_c

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