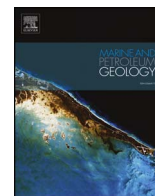




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Research paper

Pore structure, wettability, and spontaneous imbibition of Woodford Shale, Permian Basin, West Texas

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ABSTRACT

Even after more than 30 years of exploration and production, low total recovery of shale gas (< 30%) and tight oil (5%–10%) constrains sustainable shale hydrocarbon development in the United States. Since the Woodford Shale is one of the principal source rocks of the Permian Basin, West Texas, this study uses core samples of Woodford Shale from the Reliance Triple Crown (RTC) #1 well in Pecos County, Texas, to examine mineralogy, pore structure, wetting characteristics, imbibition behavior, and edge-accessible porosity with the following complementary tests: X-ray diffraction (XRD), mercury injection capillary pressure (MICP), contact angle measurement of various fluids, fluid imbibition into initially-dry shale, and edge-accessible pore connectivity from vacuum saturation-high pressure impregnation with tracers-containing n-decane. The wettability and imbibition tests use both polar (hydrophilic, deionized water) and nonpolar (hydrophobic, n-decane) fluids. Our results indicate that Woodford Shale samples (Upper, Middle, and Lower Members) have different geologic (mineralogy) and reservoir (e.g., total organic carbon, porosity, and permeability) characteristics. MICP analyses show that the median pore-throat diameters for the Woodford Shale are 3.7–5.4 nm, and almost 70–80% of pore-throats by volume are smaller than 100 nm, with high tortuosity for fluid flow and mass movement. Spontaneous fluid imbibition into Woodford Shale exhibits imbibition slopes (from the plots of log imbibition vs. log time) close to ¼ for deionized water and ½ for n-decane, consistent with contact angle measurements that indicate a marginally water-wet, but very strong oil-wet, nature for these shales. Edge-accessible pore connectivity tests indicate that well-connected hydrophobic pore networks (primarily organic matter-hosted pores) have pore-throat sizes of approximately 5 nm and experience molecular entanglement with nm-sized tracers used in vacuum saturation tests. Our analyses suggest that the middle Woodford member will be the best interval for stimulation and hydrocarbon production. The findings from these complementary experimental approaches suggest low connectivity of tortuous nanopore networks and mixed-wet characteristics, which could have implications for total hydrocarbon recovery in Woodford Shale.

1. Introduction

Shale hydrocarbon exploration and production from various basins in the United States have experienced significant growth due to scientific and technological improvements in hydraulic fracturing and horizontal drilling; this has been changing the oil and gas industry worldwide (EIA, 2014). Contributing to 44% of the total gas production, in 2016, U.S. shale gas production was approximately 14 trillion cubic feet (Tcf), a sharp increase compared to the 1.6 tcf produced in 2007 (EIA, 2016). Similarly, since 2009, U.S. oil production saw the highest level of oil production in 2016, mostly from the growth in shale plays (“tight oil formations”), with close to 3.5 billion barrels per year

(9.4 million barrels per day) (EIA, 2016).

The Woodford Shale is considered an important source of hydrocarbons within the Permian Basin (Galley, 1958; Comer, 1991; Cardott, 2012; Hackley and Cardott, 2016). Based on its geographic location and depositional characteristics, this Shale has been discovered in three different basins: the Woodford (Arkoma Basin) and Cana-Woodford (Anadarko Basin) of Oklahoma, and the Barnett Woodford (Delaware Basin) of Texas. These three Woodford Shales are projected to contain technically recoverable shale gas resources of 22.21 tcf, 5.72 tcf, and 32.15 tcf, respectively (Gupta et al., 2013; EIA, 2014).

In the research area for this study, in Pecos County, Texas, the Barnett Woodford (Delaware Basin) is an organic-rich formation with

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2–6 wt% TOC (total organic carbon). This formation has solid bitumen, which is an organic assemblage, and $\sim 1.35\%$ R_o maturity indicates that the formation is within the condensate-wet gas window (Hackley and Cardott, 2016). Similarly, the Woodford Shale of the Cana-Woodford (Anadarko Basin) and Woodford (Arkoma Basin) in Oklahoma has been well known as oil- and condensate-rich plays. The thermal maturity of the Woodford Shale in the Arkoma Basin is 0.55% R_o at depths of approximately 2300 ft (700 m) in the western portion of the Basin, while the eastern part of the Basin is overmature (6.0% R_o) at depths up to 17000 ft (5200 m); the average thermal maturity is 1.67% R_o for the Arkoma Basin Woodford Shale (Cardott, 2012). For the Cana-Woodford (Anadarko Basin), the thermal maturity varies from 0.49% R_o at a depth of 3600 ft (1100 m) to 4.89% R_o at 22,500 ft (6800 m) (Cardott et al., 1990).

There are numerous publications with an attempt to understand the Woodford Shale as a source rock, yet most of these studies focus on the geochemical signatures of stratigraphic sequences (Ellison, 1950; Comer, 1991; Mnich, 2009; Rowe et al., 2012; Harris et al., 2013; Hemmesch et al., 2014), or the mechanical properties and fracturing potential (Aoudia, 2009; Harris et al., 2011). This research investigates pore structure and wettability and their effects on fluid flow, which have rarely been studied for the Permian Basin's Woodford Shale; the work of Mnich (2009) and Aoudia (2009) only reported the measurements of porosity and permeability. This study focuses on an area in the southwestern part of the Permian Basin (Fig. 1), where the Woodford Shale is reported to produce dry gas, condensate, and sometimes oil, from an averaged 46 m-thick formation (Comer, 1991). In this work, we quantify pore structure with mercury injection capillary pressure (MICP), evaluate wettability characteristics from the contact angle measurement, and assess pore connectivity by spontaneous imbibition; both contact angle and imbibition tests involve both polar and non-polar (e.g., deionized water and n-decane) fluids to examine hydrophilic and hydrophobic surfaces and pore networks. Using tracers in n-decane, we also employ a vacuum saturation & high-pressure impregnation approach to identifying the distribution of edge-accessible connected pore spaces. This work will bridge the knowledge gap of pore size distribution, pore connectivity, wettability, and stimulation interval implicated in the hydrocarbon extraction for the Woodford Shale; the approaches and findings are also beneficial for other shale reservoirs.

2. Geological background

Occupying western Texas and southeastern New Mexico, the Permian Basin has become the common location name used for a significant oil and gas province (Dutton et al., 2005). The Woodford Shale is one of the major hydrocarbon source rocks in the western Texas Permian Basin area. The Tobosa Basin is the basin below the Permian Basin within West Texas where the Woodford Shale was deposited, while the Delaware and Midland Basins are two subdivisions of the younger Permian Basin (Galley, 1958; Comer, 1991). Formed in Late Devonian time, the Tobosa Basin is bounded by the siliciclastic Pederal Massif in the northern area, the Concho Arch (a shallow water platform) borders the eastern side, and the Diablo platform (another shallow platform) borders the western side (Fig. 1). The highest burial depth of the Woodford Shale in the Delaware and Val Verde Basins is approximately 6400 m, while the burial depth in the Midland Basin and Central Basin Platform is lower (Comer, 1991).

The thickness of the Woodford Shale in the basin center is 180 m and decreases to less than 30 m around basin margins (Comer, 1991). Based on the well log patterns, the Woodford Shale can be divided into three stratigraphic members: Upper, Middle, and Lower Woodford (Comer, 1991; Harris et al., 2011; Hemmesch et al., 2014). The Lower and Upper Woodford Shale Members show high gamma-ray amplitude differences over a scale of 3–10 m. With a range of 400–800 API units, the Middle Woodford Shale also shows steadily higher gamma-ray

values (Comer, 1991; Aoudia, 2009; Harris et al., 2011; Hemmesch et al., 2014).

3. Materials and methods

3.1. Core samples and preparation

This work uses core samples collected from the Reliance Triple Crown (RTC) #1 well (103.46°W, 30.79°N; API 42-371-37790), completed by Pioneer Natural Resources in 2007, in Pecos County, Texas (Fig. 1). The total thickness of the retrieved core is approximately 100 m, with the Woodford Shale alone covering 85% of the total section. In this core, the Upper Woodford Member is irregularly thin, and the top of the Woodford Shale Mississippian Lime has unconformable intervals (Harris et al., 2011). In our study, we use the following five core samples at different depths below ground surface (Fig. 2; Table 1): 3912-U (for 12835 ft or 3912.11 m), 3931-U (3930.70 m), 3942-M (3941.67 m), 3955-M (3955.39 m), and 3967-L (3967.28 m). Here, letters U, M, and L denote Upper, Middle, and Lower Woodford Members.

For MICP and spontaneous imbibition (SI) tests, the core slab was cut into cubes with a linear dimension for each side of 10–11 mm, using a 6" Lapidary trim saw (Kingsley North Inc., Norway, MI) and minimal use of cooling water. To investigate the contact angle, the cubes were further cut into thin slabs (10 mm \times 10 mm \times 3 mm) using a circular saw (IsoMet Low-Speed Saw, Buehler, Lake Bluff, IL). After that, the slabbed samples were polished on a 240-grit sandpaper. To investigate edge-accessible connected porosity, 3931-U sample was dried in an oven at $60 \pm 2^\circ\text{C}$ for at least two days, then subject to vacuum saturation, followed by high-pressure impregnation, a process with n-decane containing tracers (details are provided in the Section "vacuum saturation & high-pressure impregnation"). The powdered shale sample fraction ($< 75 \mu\text{m}$) was used to determine mineralogical composition with a X-ray diffraction (XRD) (Maxima XRD-7000, Shimadzu Corporation, Kyoto, Japan), and TOC using Shimadzu TOC-VWS Wet Chemical Total Organic Carbon Analyzer.

3.2. Mercury injection capillary pressure (MICP) analysis

The MICP method is one of the most efficient and cost-effective ways to characterize pore structure for porous solids such as shales (Gao and Hu, 2013). The porosity, particle density, bulk density, and pore-throat size distribution of a porous medium can be directly measured by MICP tests (Webb, 2001; Gao and Hu, 2013; Zhang et al., 2016). In addition, permeability (Katz and Thompson, 1986, 1987; Gao and Hu, 2013) and tortuosity (Hager, 1998; Webb, 2001; Hu et al., 2015) can be indirectly derived from MICP results.

Utilizing a mercury intrusion porosimeter (AutoPore IV 9510, Micromeritics Instrument Corporation, Norcross, GA), liquid mercury was incrementally injected with an increasing hydrostatic pressure to occupy the pore spaces of increasingly smaller pore-throat sizes in shale. Considering the high surface energy and non-wetting characteristics of mercury and assuming that the pores are cylindrical, Washburn (1921) determined pore throat diameters from equivalent pressures.

Before the MICP tests, a cube-sized shale sample was placed in an oven at 60°C for at least two days to remove moisture. Then, the samples were kept in a desiccator with a relative humidity of less than 10% at room temperature (23°C) for cooling. At the beginning of MICP test, the connected pore spaces in a sample are evacuated until $50 \mu\text{m}$ Hg pressure (at 6.7 Pa or 99.993% vacuum). Then, the sample is subjected to low-pressure conditions, followed by a high-pressure test up to 413 MPa or 60,000 psi, which corresponds to a pore throat diameter of 2.8 nm using the corrected Washburn equation to consider variable contact angle and surface tension in nm-sized pore spaces (Wang et al., 2016). A penetrometer (narrow-bore sample holder), used for samples with less than 5% porosity, recorded the largest pore throat diameter at

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