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# Rock characterization of Fayetteville shale gas plays

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#### HIGHLIGHTS

### G R A P H I C A L A B S T R A C T

- Geological properties of Fayetteville shale gas samples were measured using multiple disciplinary techniques.
- Micro-features of shale rocks were characterized using the dual beam system (FIB/SEM).
- 3D pore structure of a shale rock was reconstructed using FIB/SEM slides.
- The properties of a shale rock, including permeability, porosity, were calculated using the reconstructed 3D model.

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#### 1. Introduction

Unconventional natural gas resources have grown in importance as a complement to conventional fossil fuels as world energy demand has increased [6]. Shale gas is the second largest unconventional energy resource after heavy oil. Recently, the United States Geological Survey (USGS) estimated that tight sands and gas shale in the United States may hold up to 460 Tcf of gas. There

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3-D pore structure model for Fayetteville shale

### ABSTRACT

Multiple techniques were used to characterize the petrophysical properties of the rock samples from the Fayetteville shale gas play, including clay mineralogy, wettability, organic matter and their maturation, submicron pore structure, and 3-D pore structure. X-ray diffraction (XRD) mineralogy analysis showed high quartz and low clay content in the Fayetteville shale. Wettability tests revealed that the shale surface is originally intermediate-wet and the additives used in hydraulic fracturing fluids can alter shale gas surfaces toward water-wet conditions. The kerogen analysis suggested kerogen type IV and a high level of maturation in the tested samples. Three types of pores were observed through SEM images and the majority of the pores in organic matters were submicron sized (5–100 nm). A three-dimensional pore structure model was reconstructed from 200 two-dimensional SEM/FIB images, and the rock petrophysical properties, including porosity, permeability, and tortuosity, were calculated from the model. In addition, a good agreement was found between the total organic carbon (TOC) computed from SEM images and the TOC measured in the laboratory.

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are about 200,000 unconventional gas wells in low-permeability sands, coal-bed methane deposits, and shale gas in the lower 48 states. Shale gas is more environmentally friendly and attractive compared to other energy resources due to its ecological advantages (low levels of carbon dioxide CO<sub>2</sub> emission) and safety qualities (insignificant sulfur dioxide contents, H<sub>2</sub>S%).

The major cause of productivity impairment in gas wells during drilling, completion and fracturing or workover operation is the retention of the injected fluids and the specially designed additives, such as friction reducers, viscosity modifiers. It is increasingly important for shale gas development to select proper fluids

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and associated additives that have no damage to the formation. Knowing the fluid flow behavior is of major importance for the proper selection of fracturing fluids and additives. However, many petro-physical properties of shale gas formation are significantly different from those of conventional reservoirs because of nanoscale pores and channels, unique pore structure, and the resulted wettability, transport, and storage properties. In this study, some core analysis methods such as mineralogy measurements, kerogen analysis, and high resolution rock imaging were attempted to characterize the micro-structure of shale gas rocks.

The Fayetteville shale is located in the Arkoma basin of northern Arkansas. It consists of black, organic-rich rock ranging in thickness from 50 to 550 feet at depths of 1,500-6,500 feet. It contains Mississippian-age shale bounded by limestone (Pitkin limestone) above and sandstone (Batesville sandstone) below [7]. The gasin-place accumulation is enormous (more than 40 Tcf). The bottomhole temperature ranges from 120 to 220 °F. The formation water salinity (in terms of total dissolved solids concentrations) is measured at 150,000-230,000 ppm. Only in recent years, though, has the true potential of the Fayetteville shale been demonstrated, primarily because of the access to new drilling and hydraulic fracturing technologies, high gas quality, and its geographic location. Development of the Fayetteville began in the early 2000s. Between 2004 and 2007, the number of gas wells drilled there annually jumped from 13 to more than 600, and the annual gas production for the shale increased from just over 100 MMcf to approximately 88.85 Bcf [2].

Although much production has occurred in the Fayetteville shale, it remains difficult to accurately estimate its size. This could be attributed to the fact that the Fayetteville shale has not been evaluated thoroughly, and limited public information is available about its organic content, petrophysical properties and potential response to horizontal drilling and hydraulic fracturing. Therefore, this study tested the shale samples using the methods of shale mineralogy, wettability testing, kerogen analysis, and micro-pore structure analysis. The aim of this work is to investigate the petrophysical properties of the shale play, which will provide us the fundamentals to evaluate the flow behavior of gas and hydraulic fluids in the nano-level pore sized porous media. The sample tested in this study was taken in Van Buren, Arkansas at a depth of 2,351 feet. The shale mineralogy and contact angles of the rock samples were measured, and the effect of various fracturing fluid additives on their wettability was tested. Kerogen analysis was provided for the information about the kerogen type and its degree of thermal maturation. An in situ dual-beam microscope (Scanning Electron Microscope and Focused Ion Beam, also called SEM-FIB) was exploited to image and analyze the microstructure of the Fayetteville shale samples.

#### 2. Clay mineralogy

Rock mineralogy is critical for shale evaluation [22,17] and can greatly affect the brittleness of rocks [9]. The proportion of quartz-

Table 1

XRD analysis summary for Fayetteville shale.

Minerals	Component	wt.%
Clay minerals	Chlorite Illite	<5 20–25
Non-clay minerals	Calcite Dolomite Quartz	5–10 5–10 45–50

carbonates-clays could lead to very different mechanical properties in rocks. Shales with abundant quartz usually are very brittle and have a high Young's modulus, while shales with high clay content are less brittle [16]. Therefore, the rock mineralogy of shale plays is essential for drilling and hydraulic fracturing operations.

In this work, the specimens were examined on the XRD instrument (Philips X-Pert Diffractometer) in different circumstances. The sample was treated chemically, heated and then reexamined. To eliminate the possibility of interference between the peaks of different clay minerals, the following procedure was implemented. Initially, the samples were examined before applying any treatment, and the results were marked as "Untreated." Then, the samples were put in a glycolation vessel for 24 h to widen the clay mineral layers for better mineral identification resolution. This run was called the "Glycolated" run [12]. The last run was performed after heating the samples to 375 °C for 1 h, and it was called the "Heated" run. And each specimen was measured three times in order to ensure the accuracy of the results.

Fig. 1 presents the combined plot of the XRD results for the Fayetteville shale. Table 1 summarizes the quantitative analysis of the XRD results for the shale gas samples. The range of mineral content percentages from all three measurements is given in the table. It shows that the Fayetteville shale has a high quartz content. Deville et al. [5] also evaluated the Fayetteville mineralogy using an XRD analysis of the cuttings and core samples and obtained an average composition of 40% quartz and 24% illite. This agrees with the presented XRD results. The high quartz and low clay content in our tested samples indicate that the Fayetteville shale has a relatively high Young's modulus and low Poisson's ratio, making the rocks more brittle and prone to natural fractures [9]. Therefore, this shale is a good candidate for fracture stimulation.

#### 3. Wettability measurements

The wettability of a shale rock surface is of major importance to the gas flow in the porous media and the flow-back behavior of hydraulic fracturing fluids. The chemical additives such as friction reducers and viscosifiers often are added into the fracturing fluids, which may impair fracture permeability and alter rock wettability, thus influencing gas flow. Therefore, it is important to study the wettability of shale rocks and the interaction between the rock's surface and the fluid composition.



Fig. 1. X-ray diffraction results of Fayetteville shale.

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