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The representative sample size in shale oil rocks and nano-scale characterization of transport properties

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ABSTRACT

The experimental determination of petrophysical properties for shale rocks is dependent on measurement techniques and often produces inconsistent results. Alternatively, high-resolution three-dimensional imaging techniques coupled with image analysis and direct numerical simulations have been employed to estimate these parameters in shale samples. Nevertheless, the application of these results at the core and reservoir scales are uncertain due to the limited size of imaged samples. Here, Focused Ion Beam milling and Scanning Electron Microscope tomography is employed to study transport properties for the three upper layers of the Bakken formation. The representative size of each shale sample is characterized. Pore types, their connectivities, and pore size distributions are studied using high-resolution micrographs and their implications to mass transport at the macro scale are discussed. Porosity and permeability of the samples are also calculated from three-dimensional images, and the results are compared to experimentally-measured values at the core scale. It is shown that the representative size of shale samples is dependent on the scale of analysis and could range from tens to hundreds of microns. We found that the upper Bakken layer is rich in clay and organic materials, the dominant pore type is pores associated with organic matter, and no connected porosity was observed in the preserved core samples. In contrast, the middle Bakken layers (upper and lower layers) have about 1% connected porosity mainly as intraplatelet pores within clay aggregates and interparticle pores, resulting in permeabilities between 4 and 30 μD. Moreover, comparison of pore types and modeled flow pathways suggests the presence of water-wet connected pores in the middle Bakken rocks, whereas mainly oil-wet pores are present in the upper Bakken shale.

1. Introduction

The ever-growing demand for energy, relatively high price of hydrocarbons, and recent advances in production technologies have brought tight hydrocarbon-bearing shale formations into attention as a vast source of energy. Shale rocks are defined as laminated fine-grained ($\leq$62.5 μm) argillaceous sedimentary rocks with varying compositions [Potter et al. (2005)]. Pores within these rocks are orders of magnitude smaller (nanometer scale) than those in conventional carbonate and sandstone samples (micrometer scale)[Nelson (2009); Bertoncello et al. (2013)]. Therefore, compared to conventional rocks, shales typically have low porosities (5–10%) and permeabilities (10 nano-Darcy to 10 micro-Darcy) [Bertoncello et al. (2013)]. The experimental measurements of petrophysical parameters in shale rocks using conventional techniques have reported inconsistent results [Bertoncello et al. (2013); (Sinha et al., 2013), (Lasswell, 2013)]. It was shown that the results are sensitive to the measurement techniques and experimental conditions. Alternatively, high-resolution three-dimensional images of rocks coupled with image analysis and direct numerical modeling of single- or two-phase flow has been used to characterize the shale rocks [Keller et al. (2013); (Gelb et al., 2011), (Al-Raoush and Papadopoulos, 2010), (Chen et al., 2013), (Suhrer et al., 2013)]. The currently available imaging tools for three-dimensional analysis of rock samples include non-destructive instruments, such as traditional medical X-ray computed tomography (CT) and modern higher resolution X-ray computed micro- and nanotomography, and destructive instruments such as dual beam Focused Ion Beam and Scanning Electron Microscope (FIB-SEM) tomography. X-ray based CT technique uses computer-processed X-ray intensities from various realizations, allowing the reconstruction of three-dimensional images of intact objects. In FIB-SEM tomography, a sequence of two-dimensional cross-sectional images, spaced evenly through a region of bulk specimen, is acquired by physical sectioning of an object (using FIB). This stack of two-dimensional images is then re-constructed into a three-dimensional digital gray-scale representation of the sample volume. Since the latter technique directly obtains an electron micrograph of the rock surface, less imaging artifacts are produced and smaller features can be resolved at higher resolutions (e.g., ~1 nm). However, higher resolutions provided by advanced imaging techniques such as FIB-SEM tomography and computed nanotomography inevitably limit the field of view under study. Meanwhile, the instruments with larger field of view such as conventional (medical) CT and micro-CT have much lower resolutions (e.g., ~250 μm and ~1 μm, respectively) and

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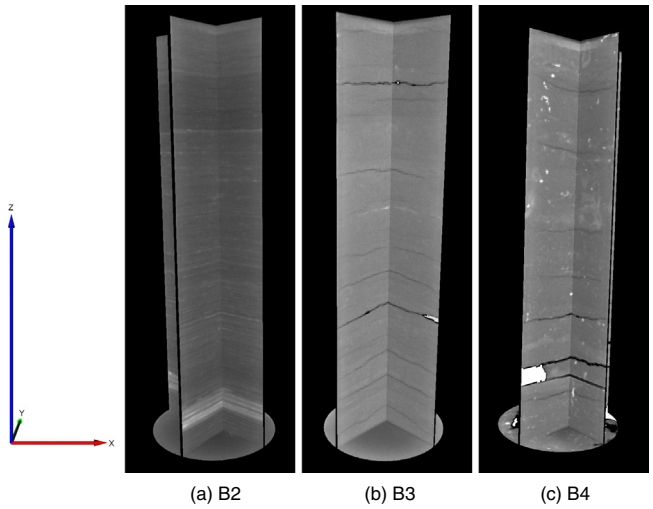

Fig. 1. 3D macro images of the preserved core samples (pixel resolutions of 235 μm in X–Y and 1 mm in the Z direction).

thus cannot resolve the majority of pores in shale rocks. Therefore, it is critical to determine the optimal sample size and resolution for obtaining flow properties in shale samples.

In order for quantitative analysis of porous rocks at nano or micro scales to be relevant at the macro scale, selection of a representative averaging volume is required. The concept of representative elementary volume (REV) for porous rocks was first introduced by (Bear, 1972). REV is defined as a minimum averaging volume over which the macroscopic measurable characteristics of a porous medium remain constant. Usually, porosity is used as the desired macroscopic parameter in this type of analysis, but other parameters such as specific surface area and permeability have also been used for this purpose [Zhang et al. (2000); (Hendrick et al., 2012), (Nordahl and Ringrose, 2008)]. (Zhang et al., 2000) proposed the use of statistical REV (sREV) for heterogeneous rocks, which is less restrictive compared to the deterministic REV. Later, this concept was used by other researchers to find sREV in FIB-SEM images of rocks such as chalk [Yoon and Dewers (2013)] and shale samples [Chen et al. (2013); (Gelb et al., 2011)]. Yoon and Dewers (2013) analyzed a ~12^3 μm^3 FIB-SEM image with a resolution of about 15 nm and found the sREV of their chalk sample to be between 5–10 μ m (1D). In another study, (Chen et al., 2013) investigated intrakerogen pores by FIB-SEM imaging on a shale sample ($3.4 \times 1.4 \times 1.2$ μm^3) with the resolution of 12 nm. They proposed that sREV for their sample is less than 1 μm (1D). X-ray nanotomography was used by (Gelb et al., 2011) to image a shale rock sample (~65^3 μm^3) at 50 nm resolution. The main image was divided into smaller cubes and the porosity of each cube was compared to that of the original sample for the standard deviation. Based on the standard deviation of each cube set, they established a ~30 μm REV for the sample (1-D). Unlike all the previous studies, (Keller et al., 2013) used a geostatistical analysis on multiple FIB-SEM images of Opalinus clay 6^3 –16^3 μm^3 in size at 2–20 nm resolutions. They proposed an REV of a few hundred microns for their clay samples by extrapolating their analysis beyond the actual size of the images. As mentioned earlier, high-resolution imaging tools suffer from a limited field of view. Thus, in all of the studies mentioned above, it is unknown whether the size of the original sample was sufficient for REV analysis. It is, therefore, likely that REV or sREV proposed by these studies is an underestimation of the true REV of their samples. Consequently, the pertinence of the mass transport simulation results typically performed on small three-dimensional images (<100^3 μm^3) to fluid transport at the reservoir scale is uncertain. One way to estimate the representative

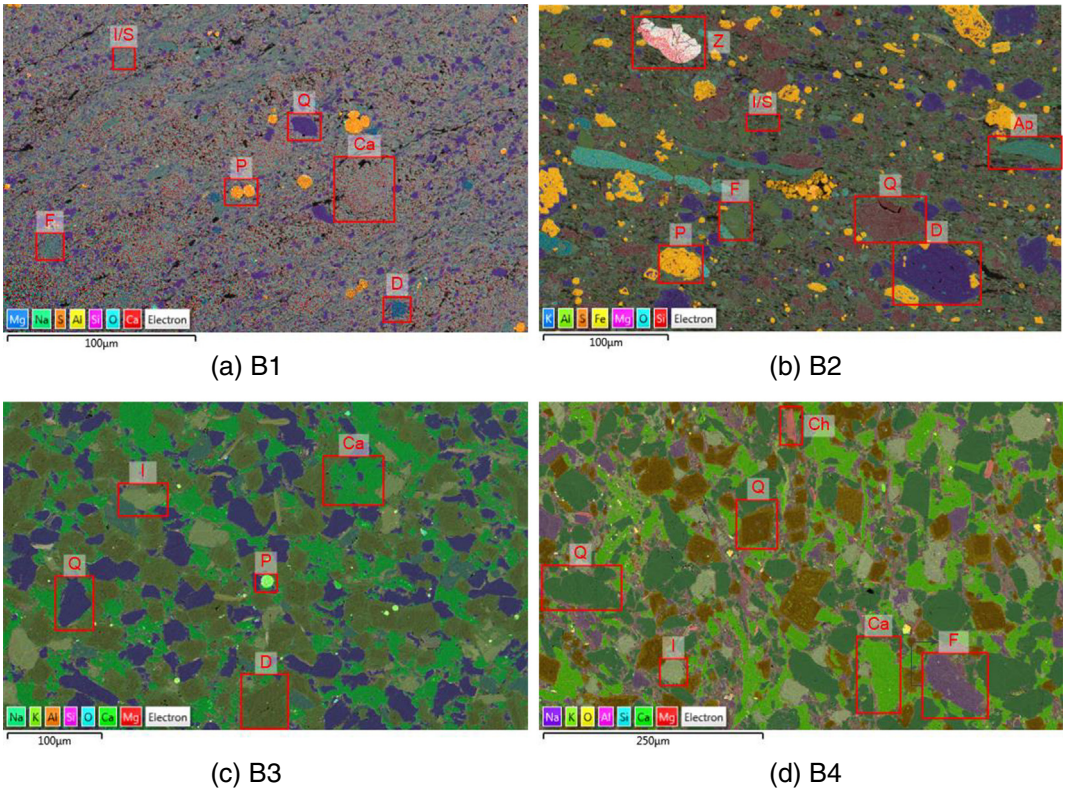

Fig. 2. Elemental maps of rock samples from EDS analysis [10 kV, 3200 nA, and 150 nm image resolution] (Q:Quartz, Ca: Calcite, P: Pyrite, D: Dolomite, I/S: Illite/Smectite, F: Feldspar, Ap: Apatite, Z: Zercon, I: Illite, Ch: Chlorite).