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# The application of sorption hysteresis in nano-petrophysics using multiscale multiphysics network models

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#### ARTICLE INFO

#### ABSTRACT

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*Keywords:* Unconventional petrophysics Pore network modeling Sorption hysteresis Multiscale gas flow Scanning electron microscopy (SEM) images of organic-rich mudrock (shale) samples show a wide distribution of pore sizes (commonly between 1 nm and 1 µm) and complex pore spatial configurations (Loucks et al., 2012). Pore size and pore connectivity are important parameters in that they have first order impact on macroscopic flow properties of a porous medium. However, given the significant difficulty in capturing multiscale pores within a single three-dimensional image, and the possible uncertainties in the existence or absence of original throats in an acquired image, it is imperative to explore indirect methods to quantify the pore structure. In this paper, we simulate sorption in heterogeneous pore network models and study sorption and permeability hysteresis analyses as indirect methods for rock characterization.

Three network types are introduced to represent the multiscale pore topology of shale rocks; specifically: regular (type 1), series (type 2) and parallel (type 3). We conclude that, in appropriate size ranges, sorption hysteresis can distinguish the three types whereas permeability hysteresis can only separate parallel from series and regular. Furthermore, the simulations show that sorption hysteresis is sensitive to compaction/cementation (closing of throats) in all network types whereas permeability hysteresis is sensitive to the diagenesis in parallel networks only.

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#### 1. Introduction

Given the vast supply of energy offered by shale (organic rich mudrock) formations (U.S. Energy Information Administration, 2013), a proper petrophysical understanding of these reserves is of great importance. Petrophysical properties (such as permeability-porosity or capillary pressure-saturation relationships) are typical inputs into large scale reservoir simulators that provide insight into the flow and multiphase displacements on field scale. Such insight ultimately helps in the design of production and recovery strategies of these reserves. In order to obtain the needed properties, one can perform experimental measurements, theoretical derivations (typically available only for simplified media (Bear, 1988; Dullien, 1991)) or pore scale numerical modeling. Experimental measurements for tight media, such as shale, are highly time consuming and alternative methods, such as the pressure pulse decay technique, are being developed (Cui et al., 2009; Darabi et al., 2012). However, it is only a correct forward model of the rock behavior, which depends on the pore scale features, that can make any inverse interpretation possible.

Pore scale modeling requires knowledge of the microstructure details of the porous medium, but having such details within a representative elementary volume, REV, and being able to perform computationally

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intensive calculations on the REV, remains a challenge in upscaling complex media. Although three dimensional images, such as X-ray tomography, have provided petrophysicists with the opportunity to view the pore structure directly (Wildenschild and Sheppard, 2013), it is yet not feasible to include pores of multiple scales into one single image. In addition, due to the common presence of very small pore sizes (1 nm up to 1000 nm), non-destructive imaging techniques, such as X-ray, are incapable of resolving the interconnecting throats completely. Destructive imaging methods such as Focused Ion Beam Electron Microscopy (FIBSEM) are currently the only source of 3D pore structure information on submicron scale, but FIBSEM may result in incomplete or faulty information by either destroying existent throats or by potentially creating artifacts (Loucks et al., 2012; Heath et al., 2012). Furthermore, with increased resolution of an image, the field of view becomes smaller, therefore making it harder to infer how nanopores connect to micropores in a representative elementary volume.

Indirect non-destructive methods can complement the information gathered from FIBSEM or any other imaging technique. We refer to indirect methods as physical measurements that are sensitive enough to detect pore structure signature behaviors without directly mapping the pore structure itself. In this work, hysteresis due to nitrogen sorption is explored to characterize shale rock samples.

The idea that sorption loops are sensitive to pore structure in porous media has already been a focus of study in applied chemistry. The International Union of Pure and Applied Chemistry (IUPAC) has classified the sorption cycles into four main types as shown in Fig. 1. This

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Fig. 1. The IUPAC classification of sorption hysteresis loops.

classification, however, mostly focuses on pore shape and size distribution rather than pore connectivity.

We approach the application of sorption hysteresis from two angles. First, the amount of nitrogen adsorbed during adsorption and desorption is studied (we call this "sorption hysteresis"). Second, the relative permeability of a nonreactive, nonadsorbing gas, such as helium, for each nitrogen sorption cycle is investigated (we call this "permeability hysteresis"). We hypothesize that sorption hysteresis is mostly affected by pore configuration whereas permeability hysteresis is mostly affected by the spatial distribution of throats.

Although work based on percolation theory (Seaton, 1991; Liu et al., 1992; Liu and Seaton, 1994) estimated the mean coordination number by fitting the desorption curve for industrial porous materials, shale rocks can have unique network topologies. Therefore, merely prescribing a mean coordination number, as is common in conventional rocks (Bernabe et al., 2010) may not honor the rock's heterogeneous pore structure. H1, H2 and H3 hystereses have been modeled with a regular structured network and tuning the size (N), coordination number (Z), macropore ( $r_p > 100$  nm) surface area ( $\frac{A_{\text{ext}}}{A}$ ), and micropore ( $r_p < 2$  nm) volume  $V_m$  (Efremov and Felenov, 1991). However, the two key parameters ( $\frac{A_{\text{ext}}}{A}$ ) and  $V_m$  were treated as tuning parameters and did not explicitly exist in the network model.

In our previous work (Mehmani et al., 2011; Mehmani and Prodanović, 2014; Prodanovic et al., in press), we hypothesized that a multiscale network model that explicitly honors the spatial configuration of small scale pores (i.e. microporosity relative to intergranular or intragranular porosity) is capable of capturing unique petrophysical features in tight gas sandstone and carbonates. In other work (Mehmani et al., 2013), we introduced multiscale multiphysics network models to simulate gas flow from first principles in mudrocks. In this paper, we extend multiscale, multiphysics networks to model sorption and permeability hysteresis. In contrast to the traditional IUPAC nomenclature, our definition of nano/macro pores are literal in that nanopores are pores in the nanometer scale (1  $\mu$ m <  $r_p$  < 1  $\mu$ m) and micropores are in micrometer scale (1  $\mu$ m <  $r_p$  < 1 mm).

Pore network models are cost efficient representations of porous media. They can capture the geometry and topology of the medium and include small scale physics (capillary and viscous forces). For a detailed review on pore network models and their history refer to references (Blunt, 2001; Valvatne and Blunt, 2003; Joekar-Niasar and Hassanizadeh, 2012). Network models have even shown promise in fibrous materials (Thompson, 2004). It is thus expected that the authors' developed model will be extensible to organic rich shale media (Loucks et al., 2009). Presently microfractures — which are common in mudrocks — are not included in our networks.



Fig. 2. Schematic of the application of nitrogen sorption hysteresis in nano-petrophysics.

We aim at investigating the interplay of pore type classification (inter/intra particle and organic matter pores) (Loucks et al., 2012) via three network types. Adsorbed and permeability hystereses for various size distributions are explored. Fig. 2 depicts the role of nitrogen sorption cycles in the scope of this work. The hysteresis phenomena we investigate here assist in (both forward and inverse) evaluation of the pore structure and fluid physics and ultimately contributes to building multiscale multiphysics networks where direct information on them is difficult to obtain. The networks which honor the multiscale pore structure and fluid physics can improve the calculation of important petrophysical properties such as permeability (absolute/relative), capillary pressure curves, electric logs and NMR response interpretations (such as 2D NMR maps).

#### 2. Methodology

#### 2.1. Simplified classification and network generation

All of the network models constructed in this work are unstructured (that is, they are not on a regular grid such as cubic) and threedimensional. A description of the pore network types is as follows.

Type 1 (regular): A single scale network in which the average coordination number is four (Fig. 3a). This type is based on the Delaunay tessellation of the Finney pack of spheres (Finney, 1970; Mason and Mellor, 1995; Bryant et al., 2004). After the extraction of the network, we conceptualize each pore as a sphere with a radius equivalent to the inscribed radius of the four neighboring tetrahedra. While we use the network of a granular medium as a base in this modeling, we do not claim that shale pore spaces resemble granular media. Three-dimensional images and associated shale pore networks are still not easily available: multiple authors show 3D FIBSEM images (Heath et al., 2012, 2011; Chen et al., 2012), but technical difficulties have thus far prevented the extraction of a pore throat (ball and stick) network. A recent paper has also used 2D SEM images for statistical reconstruction of 3D pore space (Gerke and van Genuchten, 2011). We proceed with this standard model with the understanding that it is a conceptual model and could otherwise be replaced by any single length scale network.

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