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# Characterization of pore structure, gas adsorption, and spontaneous imbibition in shale gas reservoirs

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## ABSTRACT

The multiscale pore structure and its distribution as well as the occurrence state of shale gas and spontaneous imbibition in gas shale are studied experimentally. Most of the pores within organic matters are at nanoscale and are isolated in all directions. They have low porosity and connectivity, which account for the most of pore volume and determine the resource abundance of gas shale reservoirs. The contents of adsorption gas and free gas in the multiscale pore structure of shale rocks increase with the increase of gas pressure, of which the free gas increases faster than that of adsorption gas. The spontaneous imbibition shows the fast shift of  $T_2$  peaks to right at early stage of fluid transport through the interconnected network of pore structure in shale rocks. Our results may provide unified methods for clear understanding of the pore structure and fluid transport behavior in shale gas reservoirs and assist the future efficient exploitation of shale gas.

## 1. Introduction

Hydrocarbon production from shale gas reservoirs is playing an ever-increasing role in world energy supply today because of its enormous reserves and low greenhouse gas (GHG) emissions. Beyond, shale gas is not just good for the energy conservation and emission reduction—it is in line with the global momentum of clean energy (Tomain, 2012) and has the potential to replace petroleum as a new energy. Shale gas is an unconventional gas trapped in shale formations, where the local geological conditions lead to the great difficulty for gas recovery. Almost 70%–85% gas gets stuck to the shale matrix by adsorption (Das, 2012; Ross and Bustin, 2007; Zeng et al., 2014). In addition, the porosity and the permeability of shale beds are ultra-low, whose magnitudes are typically 1–6 orders below that of conventional gas reservoirs (Lee et al., 2016; Maurel et al., 2010). The shale gas reservoirs are usually buried in the deep underground thousands of meters below. It has been estimated that only 5%–15% content of the shale gas gets extracted by gas recovery (Bažant et al., 2014). If the enhanced gas recovery (EGR) increases by 8.2%, much cost can be cut down which is equivalent to drill a new gas well.

The shale gas reservoirs are featured by complex pore structure with different size, distribution, shape and curvature, which exert a great impact on gas storage. In previous studies, the simulation tools such as molecular dynamic (MD) provide a unique window to look into the

details of critical aspects in gas adsorption and desorption. Wu et al. (2015a) conducted MD simulations about the adsorption and displacement of gas in nanochannels, and found that the smaller slit pore stores more gas content under relatively low pressure. Zhu and Zhao (2014) proposed that there exists an optimal pore diameter for maximizing the adsorption capacity of methane in pore. They also established the equation of state for adsorption gas in nanopores by considering the disjoining pressure, and performed MD simulations to study the gas structure in nanopores under different external environments and fluid properties, which clarified the dependency of capillary condensation on pore size and curvature (Zhu and Zhao, 2014). Mosher et al. (2013) found that the adsorption amount of shale gas increases with pore size decreasing. Chen et al. (2017) found that the variation of pore-throat size may explain the adsorption hysteresis of shale gas in nanopores.

When the shale gas desorbs from the pore, it usually flows to the natural fracture then to the artificial fracture, and finally reaches to the gas well. Fig. 1 shows the fluid flow regimes in these multiscale channels depending on the Knudsen number  $Kn = \lambda/L$  ( $\lambda$  and  $L$  are the mean free path of gas molecule and the characteristic length of channel, respectively). When the pore-throat size is at nanoscale that comparable to the molecule ( $Kn > 10$ ), the molecule experiences a force field from the pore wall, resulting in extremely complex flow mechanisms. The dominated flow in this regime is free molecular flow, i.e., the Knudsen diffusion or surface diffusion, in which the classical Darcy law does not work

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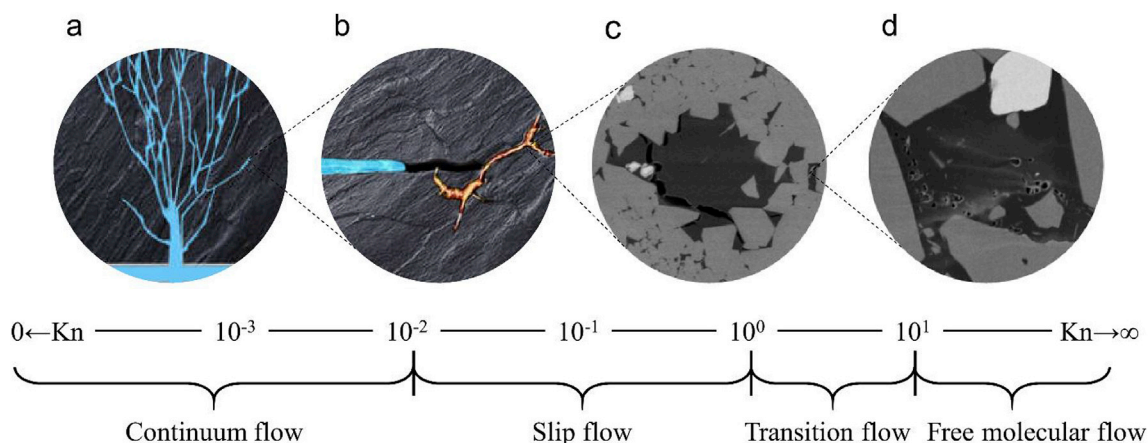


Fig. 1. Flow regimes in multiscale channels based on the Knudsen number. Fluid transport in (a) Gas well, (b) Artificial fracture, (c) Natural fracture (slit pores) and (d) nanopores.

anymore (Monteiro et al., 2012; Swami et al., 2012; Wang and Marongiu-Porcu, 2015). As the pore-throat size increases, the transition flow and the slip flow take over, in which the interaction frequency of gas-gas molecules exceeds that of the gas-pore wall. When the channel size is much larger than that of gas molecule, the continuum flow (Hagen-Poiseuille flow) dominates. Javadpour (2009) and Darabi et al. (2012) proposed an apparent permeability with Knudsen diffusion and convection taken into account, which simplified the flow formula into the form of Darcy law by replacing the permeability. However, the surface diffusion was not considered in the model, yet it may be the dominated flow in shale gas recovery (Sheng et al., 2015; Wu et al., 2015b). Moreover, fluid transport behavior during the spontaneous imbibition of shale rocks is of significant for shale gas recovery (Li and Horne, 2004), while there is lack of efficient experimental method to investigate the water flowing process.

The macroscopic breakthroughs of shale gas recovery show an increasingly dependency on the microscopic achievements, which raise the broader interest and requirements of microscopic studies. Although there have been some sound explorations (Liu et al., 2016; Sander et al., 2017; Ma et al., 2016; Ambrose et al., 2010; Jiang et al., 2016; Yuan et al., 2015), yet it is still insufficient to uncover the complex mechanism of fluid transport in multiscale pore structure of shale gas reservoirs. It is due to the lack of field data and knowledge of pore structure, occurrence state of shale gas and fluid transport behavior in shale gas reservoirs that restrict the efficient exploitation of shale gas. Further, the experiments for investigating the fluid transport behavior during the spontaneous imbibition shale rocks are seriously deficient for the gas recovery (Roychaudhuri et al., 2013).

In this paper, we first carry out experiments using unified methods including nuclear magnetic resonance (NMR), scanning electron microscopy (SEM) and X-ray nano computed tomography (nano-CT) in combination to experimentally study the multiscale pore structure and its distribution, size, shape and maturity of organic matters which relate to the resource abundance and the value for exploitation and development. Then, occurrence state of shale gas under different pressures and spontaneous imbibition in the interconnected pore structure in shale rocks are studied and discussed.

## 2. Materials and experiments

In this study, the shale samples were drilled from a 3 km underground shale gas well in Ordos Basin, China. The shale samples were all first soaked in distilled water with a vacuum pressure (15 MPa) impregnation treatment for 24 h. The samples prepared for the gas adsorption and fluid transport experiments were then drying at 90 °C for 4 h.

In this study, the NMR experiments were carried out to investigate the porosity, the multiscale pore structure and its distribution of shale rocks

using the low-field NMR (LF-NMR) system (Niumag, MacroMR12-150H-I) with a resonant frequency of 12.8 MHz and magnet strength of 0.28 T at 32 °C. The LF-NMR is a powerful technique that can probe the motion status of molecules inside the pores, pore structures of rocks, the course of reaction and their relations, etc. The LF-NMR system was also used to perform gas adsorption and fluid transport experiments. The Carr-Purcell-Meiboom-Gill (CPMG) sequence was used in the experiments. The parameters for the NMR experiments are listed in Table 1. Fig. 2a shows a schematic diagram of the system showing how the experiments are conducted. Fig. 2b is the designed core holder for providing confining pressure and environmental temperature.

The coil diameters were 25 mm, 60 mm and 70 mm for the porosity and pore structure tests, gas adsorption and fluid transport experiments, respectively. The gas employed for the gas adsorption experiments was pure methane under gas pressures of 0.66 MPa, 1.48 MPa, 2.45 MPa, 4.45 MPa, 5.79 MPa, 7.76 MPa at room temperature (25 °C). The fluid transport experiments were conducted on the evacuated shale samples by water (H<sub>2</sub>O) injection with an injection pressure of 5 MPa and a confining pressure of 7 MPa at 25 °C. The magnetic resonance signals of water filled cores were recorded every hour, and finally ended at 7.3 h.

The pore structure of shale samples was observed and analyzed using a super-resolved field-emission SEM (FE-SEM, Hitachi SU8220), which revealed the pore structure such as pore size, shape, and its distribution. In addition, when combined with statistical methods, the porosity, the pore size, and its distribution, shape and curvature can also be obtained. At the same time the elemental components of shale samples were analyzed by the backscattered electron signals. The samples for the SEM observations were sliced into small blocks (15 × 10 × 5 mm<sup>3</sup>). Considering that the sample surfaces were uncoated with conductive coating and the abundant organic matters in the samples, the accelerating voltage was set relatively low with a close working distance. Since it is often hard to distinguish the nanopore from the anomalous morphology induced by mechanical polishing, we adopted ion beam milling technique (Hitachi IM4000) after polishing. The sample surface was bombarded by the accelerated argon ions and finally achieved a real profile beneath the surface with a “mirror surface” effect.

The characteristics of non-destructive analysis and three-dimensional

Table 1  
The parameters of CPMG sequences for the NMR experiments.

	Porosity and pore structure tests	Gas adsorption experiments	Fluid transport experiments
Wait time (ms)	2000	3 000	1 000
Echo spacing (ms)	0.10	0.20	0.25
Number of trains	16	16	64
Number of echoes	3 000	16 000	15 000

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