



Experimental study of swelling of organic rich shale in methane



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ABSTRACT

Gas is stored in shale mainly in free and adsorbed phases. Since a significant amount of gas in the shale is adsorbed to the organic matter and/or clay minerals, it is possible that gas adsorption will induce shale swelling, which may then have an impact on the gas flow behavior in the shale thus its gas production. In this work, strain behavior was studied in two gases, helium and methane, at different pore pressures under constant hydrostatic pressure at 20 MPa on two shale samples. The results show that porosity and volumetric strain are functions of gas pressure and the strain is larger in methane than helium demonstrating gas adsorption induced swelling for the shale samples. The calculated methane adsorption induced swelling strain is at a magnitude of 0.1% volumetrically with pressure at 10 MPa for the shale samples studied. The adsorption induced shale swelling strain shows a Langmuir-like relationship with pressure and is proportional to the amount of methane adsorbed. The results also show slight anisotropic strain behavior between the two directions of parallel and perpendicular to the bedding and strain hysteresis with methane in and out of the shale. The gas adsorption induced swelling may influence gas flow in gas shale, thus more research in this topic is warranted.

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1. Introduction

With the successful exploration and development of shale gas reservoirs in the USA, shale gas has received increasing interests from many other countries as an alternative hydrocarbon resource (Hartwig and Schulz, 2010; Soroush et al., 2010; Tan et al., 2014). Despite the success of shale gas production in the North America, shale gas is difficult to produce due to its low reservoir permeability and gas storage behavior. Gas shale is rich in organic carbon and clay minerals, resulting in significant amount of methane being adsorbed, along with compressed free gas in other pores (e.g., Labani et al., 2013; Yu and Sepehrnoori, 2014). Adsorption is one of the key gas storage mechanisms as 20% to 85% of the gas in place is adsorbed in shale (Hill and Nelson, 2000) and it has a significant impact on gas production behavior (Pan and Connell, 2015).

Shale is composed with complex mineral pore and fracture network which is the main site for gas storage and pathway for gas transport. At reservoir conditions, as gas is produced and gas pressure decreases, the shale may experience strain change as a poroelastic material due to the effective stress change. This would affect the pore structures thus the gas flow behavior in the shale pore/fracture system. Moreover, since a significant amount of gas in the shale is adsorbed to the organic matter

and/or clay minerals, it may also cause adsorption-induced strain change, as adsorption/desorption changes surface potential energy of the adsorbent leading to its swelling/shrinkage (Pan and Connell, 2007). The sorption-induced shale swelling/shrinkage will also lead to the change of pore structure at reservoir conditions, further impacting on the gas flow behavior in the shale.

Gas adsorption in shale has been studied extensively especially recently. However, all these adsorption measurements are mainly focused on the adsorption capacity of the gas shale and the various factors affecting adsorption (Chalmers et al., 2012; Chareonsuppanimit et al., 2012; Gasparik et al., 2014; Guo, 2013; Lu et al., 1995; Ross and Marc Bustin, 2009; Weniger et al., 2010; Zhang et al., 2012). Nevertheless, there has been no laboratory measurement of swelling on shale associated to gas adsorption (Heller and Zoback, 2014). Heller and Zoback (2014) used pure mineral particles of carbon, illite and kaolinite instead of natural shale samples to indirectly investigate the importance of swelling in gas shale, due to the long equilibration time and small swelling strain of adsorption-induced shale swelling. Other shale swelling measurement has been using water or its vapor (e.g., Yuan et al., 2014a). However water–shale interaction is different as clay minerals have strong interaction with water and the mechanism of water induced swelling is different to that of the gas adsorption induced shale swelling. Thus it is important to study the gas adsorption induced shale swelling, which could provide insight to the gas and shale interaction and gas transport in shales.

In this work, laboratory measurements of adsorption capacity and corresponding strains, including axial strain and radial strain, on two

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gas shale samples were carried out under constant hydrostatic pressure. Helium was first used to study the strain behavior as helium is considered non-adsorbing to shale at reservoir temperature and methane was then used to study the adsorption and shale strain behavior. Then adsorption-induced swelling strain was analyzed based on the experimental strain data in helium and methane. Strain anisotropy and hysteresis were also investigated.

2. Experimental methods

2.1. Shale samples

Shale samples used in this study were recovered from the outcrop of the lower Cambrian Niutitang formation in Hunan province, Southern China. The lower Cambrian Niutitang formation is one of the main target formations for shale gas exploration in China (Tian et al., 2015). Sampling location at Taoyuan county is located in the northwest of Hunan province, which belongs to upper yangtze platform (Lin et al., 2014). The main geological structures and the sampling location are shown in Fig. 1 (Wang et al., 2014; Lin et al., 2014). There are three main formations in the Lower Cambrian in the northwest of Hunan province, the Niutitang, Palang and Qingxudong formations from bottom to up. The hydrocarbon-producing organic rich shales are mainly developed in the Niutitang formation, which was deposited in deep-water shelf environment during Paleozoic Marine sedimentation (Wang et al., 2014; Zhang et al., 2014). Shales in Niutitang formation have good gas production potential because of its large distribution area, large thickness, high organic carbon contents and high thermal maturity, and it is considered as the key breakthrough area of shale gas exploration in Southern China (He et al., 2015; Lin et al., 2014; Nie et al., 2011). Northwestern areas of Hunan province have gone through repeated tectonic movements, where drape and fault structures have developed. The Lower Cambrian organic rich shale formations developed in the northwest of Hunan province are controlled by Huahuan-Cili large abyssal fault. Thickness of the organic rich shales gradually decreases from northwest to southeast of Hunan province, and the maximum thickness is about 200 m (Xiao et al., 2012; Zhang et al., 2014). Shale gas reservoirs in northwestern areas of Hunan province have experienced large tectonic uplift and its geological conditions are similar to that of Eastern United States (Xiao et al., 2012).

The mineral compositions of the two samples were determined using X-ray diffraction (Table 1). Quartz, feldspar, and pyrite are the brittle mineral components, which combined about 68.08% of the total mineral content for Sample 1 and 68.32% for Sample 2. The average content of clay minerals is about 25.18% for Sample1 and 24.51% for Sample2. The dominant clay mineral type is illite, which has a high gas adsorption and storage capacity (Heller and Zoback, 2014; Lu et al., 1995). Clay minerals are often mixed with organic matter in gas shale. A high clay mineral content implies the shale contains a large number of pores which provide space for gas to occur and migrate (Hou et al., 2014).

The total organic carbon (TOC) and vitrinite reflectance (R_o) results of the two samples are also summarized in Table 1. The organic matter in the samples is type II kerogen. The TOC of the shale is the most important factor that controls methane adsorption capacity (Hou et al., 2014) and methane adsorption capacity is positively correlated with TOC (Lu et al., 1995; Zhang et al., 2012). The organic matter contains the main adsorption sites in which nanometer-sized pores exist (Loucks et al., 2009). When the shale maturity is greater than 0.60% R_o , a large number of nanometer-sized pores can be observed in organic matter (Loucks et al., 2009). The average TOC of the two samples is about 6.96%, which suggests that the samples may have high adsorption capacity. Furthermore, the samples have high maturity at about 2.45 $R_{o,max}$ % (Table 1), which also suggests that the shale may have high adsorption capacity. As gas shale with high maturity has pores with large specific surface areas, this makes the gas adsorption capacity correspondingly higher (Ross and Marc Bustin, 2009).

Samples were prepared to cylinders at diameter of 50 mm and length of 100 mm for adsorption and strain measurements. Considering that gas migrate mainly along the bedding direction, the samples were drilled parallel to the bedding. Using the samples drilled parallel to the bedding, gas can flow quickly into the microfractures and pores developed on the bedding plane and then migrate into the shale matrix, so that gas in the shale samples can reach saturation state faster. To avoid damaging the original pore structure of the shale, the core samples were dried in a vacuumed chamber at room temperature for a few days until their weight did not change. After drying, the weight of the two samples was 436.25 and 432.51 g.

2.2. Pore structure characterization

Fresh surfaces of the shale samples were polished by argon ion beam milling system at an accelerating voltage of 4 kV for six hours. Mineral grains, pores, and microfractures in the shale samples were observed by scanning electron microscope (Quanta 200 F) under the condition of back scattering. Mineral compositions were determined using energy spectrum analysis.

Micropore surface area, volume, and size distribution in the shale samples were obtained by N_2 adsorption at 77 K using NOVA1000e surface area and pore size analyzer. The off-cuts of the two samples were crushed into grains of about 1 mm in size. The prepared graininess samples were dried in an oven at 105 °C for 10 h before the tests and then degassed in the pore size analyzer for another 10 h. The relative pressure was applied from 0.04 to 0.98 during the testing process. Surface area and pore size distribution were calculated using Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) models.

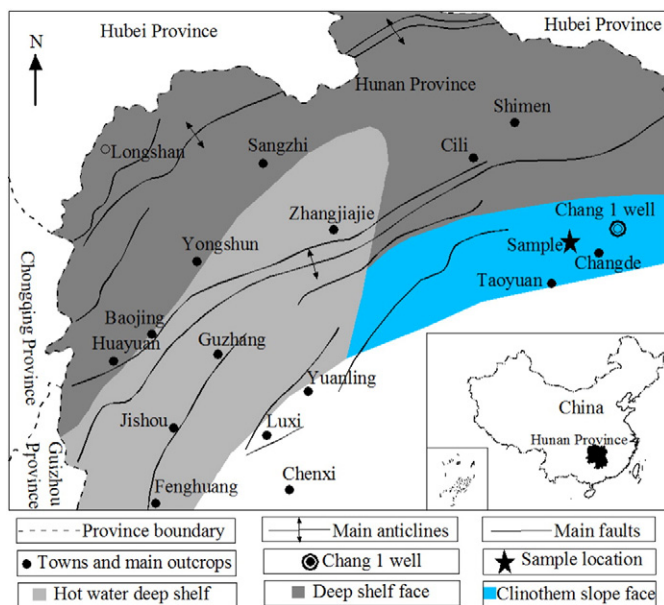


Fig. 1. The location of Hunan province in China, the main geological structures and the sample location in northwest of Hunan province.

Table 1

TOC, $R_{o,max}$ and mineral compositions of the shale samples.

Sample ID	Mineral compositions (%)				TOC (%)	$R_{o,max}$ (%)
	Quartz	Feldspar	Pyrite	Clay minerals		
Sample1	54.74	11.38	1.96	25.18	6.74	2.42
Sample2	56.44	10.12	1.76	24.51	7.17	2.47

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