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Investigation of moisture effect on methane adsorption capacity of shale samples

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ABSTRACT

The existing moisture in shale samples makes the evaluation for shale gas reservoirs more difficult due to its impact on the methane adsorption capacity and pore structure measurements. This paper compares the pore structure characteristics and methane adsorption capacity between dry and wet shale samples from Perth Basin, Western Australia. Pores with size between 0.4 nm and 100 nm were quantified by low-pressure N_2 and CO_2 adsorption. The comparative results demonstrate that moisture could alter the pore size distribution for big pores (> 16 nm) and small pores (0.4–16 nm) in different ways.

For each sample, the moisture effect on methane adsorption in shales changes with pressure: moisture effect on methane adsorption is more pronounced at lower pressure than higher pressure. For all samples, the effect of moisture on methane adsorption is related to the total organic carbon (TOC) content. Moisture could reduce methane adsorption by blocking clay- hosted small pores directly and organic matter-hosted small pores indirectly in high TOC samples. This phenomenon can effectively lead to a reduced Langmuir volume (V_L) and increased Langmuir pressure (P_L) when moisture exists.

1. Introduction

Unlike conventional gas, one of the critical parameters in evaluating shale gas is the methane adsorption capacity. The methane adsorption capacity is controlled by shale compositions (specifically organic matter richness) and external parameters (e.g., pressure, temperature, and moisture) [1–6]. The total organic carbon content (TOC) is positively related to methane adsorption capacity in shale-gas systems [1,3,5,7,8]. As the thermal maturity increases, the methane adsorption capacity increases due to the pore generation or the increasing aromaticity [1,9], and then follows a decreasing tendency at very high maturities (> 2.5% VR_r) [3]. In addition, kerogen type can also affect the methane adsorption capacity in the decreasing order of: type III > type II > type I [9]. Apart from the organic matter, the inorganic matter, mostly clay minerals play an essential role in methane adsorption for clay-rich shales [10,11]. A study on pure clay samples has shown that methane adsorption capacity of clay minerals follows the order of: smectite > mixture of illite and smectite > kaolinite > chlorite > illite [10].

For shales, pore structure can control the methane adsorption capacity [1,12–15]. Pores in shale can be divided into three categories based on the IUPAC: micropores (< 2nm), mesopores (2–50 nm) and macropores (> 50 nm) [16]. Micropores and fine mesopores can present in both organic matter and clays [17]. Even though organic matter hosted-porosity is more efficient than clay-hosted porosity in methane adsorption, the determination of clay hosted-porosity is critical for modelling methane adsorption [14], especially with moisture under an actual condition in shale gas reservoirs.

Moisture can reduce the methane adsorption capacity greatly by occupying sorption sites in shale gas reservoirs [9,18]. Studies related to the effect of moisture on methane adsorption have been conducted on coal, clay and shale samples using both experiment and simulation methods [4,11,18–24]. A Monte Carlo simulation study on clay minerals has claimed that water molecules cover the clay surfaces through forming water film [23]. A combined experiment and simulation study on shale has shown that moisture in actual condition could exist as capillary water in pores and water film bounded on pore surface. The capillary water blocks the available adsorption sites and the water film changes the interaction between methane and clay [11]. Another experiment study on low TOC shales demonstrated that moisture influences the macropores most and micropores least, and methane adsorption capacity reduces significantly. However, micropores are believed to be more important than macrospores in methane adsorption

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in shales [1,4,17]. Besides, methane adsorption capacity of shale decreases with increasing moisture content until a critical moisture content reached. By contrast, another study showed that no correlation exists between moisture content and methane adsorption capacity [18].

Although the effect of moisture on methane adsorption in shale gas reservoirs is well documented by experiment and simulation studies, the question about how moisture reduces the methane adsorption capacity is still not well-understood. Especially, how moisture changes the pore structure characteristics of micropores and its related impact on methane adsorption are unclear. In this study, methane adsorption and pore structure characteristics were measured for both wet and dry shale samples using high pressure methane adsorption and low-pressure adsorption (N_2 and CO_2), respectively. As it is not easy to distinguish clay or organic matter from shale in terms of wettability, and the shale sample in this study have low TOC, so we assume that water is more likely to adsorb on clay than organic matter. The results from this study give insights into the mechanisms of methane adsorption reduction with moisture which could contribute to the accurate evaluation of gas adsorption potential in shale gas reservoirs in real situations.

2. Methodology

2.1. Samples

A total of 5 shale samples, from Carynginia Formation, Perth Basin, Western Australia, were used in this study. Tables 1 and 2 show the mineralogical composition and the geochemical analysis of the samples studied, respectively. The total organic carbon (TOC) ranges from 0.23 to 3.03% and T_{max} from rock-eval ranges between 458 and 465 °C, which suggests that all the samples have very close maturity. The clay content of all the samples ranges from 28 to 56%. Herein, low TOC samples were chosen to explore the moisture effect on clay and organic matter in methane adsorption.

2.2. Sample preparation

All shale samples were crushed into powder and passed through a 60 mesh sieve (< 250 μm). The powders with size less 250 μm will be used in both high pressure and low pressure adsorption measurements. After crushing, all the samples were dried under vacuum at 110 °C for 8 h to remove residual moisture. Around 10 g of each dried sample was moisturized in an atmosphere of the saturated solution (KCl) at 25 °C for 72 h, which provides a consistent relative humidity (RH) of 84% [25]. Even though only one moisturization level was conducted, it still can offer informative comparisons between dry and wet samples on adsorption measurements. Besides, it has been observed that methane adsorption in shale decreases with increasing RH and no change exists between 75 and 97% RH [3]. Thus, the 84% RH we chose in this work can influence the methane adsorption in shales strikingly. The moisture content in the samples was determined by subtracting sample weight before and after moisturizing (Table 3), showing a range of 0.015–0.022.

Before loading the sample into the sample cell of the low-pressure/high-pressure adsorption measurements, all the samples were degassed to remove volatile compounds. From different degassing temperatures used and discussed for adsorption measurements on shales [26–28], 250 °C has been recommended to be the standard degassing procedure to maximize the results of low pressure adsorption [26], but clay minerals have been reported to be sensitive to high temperature [29]. Herein, the typical degassing temperature of 110 °C was used for dry samples to remove moisture during sample loading or weighing, which is consistent with the drying temperature. But for moisturized samples, 40 °C is used as the degassing temperature to remove free and condensed water but not high enough for clay-bound water under near vacuum condition. It is inevitable that water desorbs during degassing. Thus, we compared the weight of samples before and after 8 h degassing. The percentage of reduced weight after degassing for both dry and moisturized samples ranges from 4 to 6%, which indicates that the loss of bound water during degassing is very limited for wet samples. Therefore, the moisture content studied in this work is the adsorbed water excluding the condensed water and free water.

Bedsides, even though the degassing temperature is 110 °C for dry samples and 40 °C for wet samples, the different adsorption results between the dry and wet samples cannot attribute to the different degassing temperature as all samples were initially dried at 110 °C.

2.3. Low-pressure N_2 and CO_2 adsorption

Low-pressure N_2 and CO_2 adsorption for dry and wet shale samples were measured using a Micromeritics® Tristar II plus apparatus at 77 K and 273 K, respectively. 77 K and 273 K are provided by liquid nitrogen and mixture of ice and water, respectively. The relative equilibrium adsorption pressure (P/P_0) ranges from 0.01 to 0.99, where P is the gas vapor pressure in the system and P_0 is the saturation pressure.

The N_2-based adsorption isotherms were interpreted by Brunauer-Emmett-Teller (BET) method for surface area and N_2-based density functional theory for pores size distribution. The BET surface area was calculated in the P/P_0 range of 0.1–0.3 [30]. The CO_2-based adsorption isotherms were interpreted using Dubinin-Astakhov (D-A) and non-local density functional theory for micropores volume and pore size distribution, respectively. The Dubinin-Astakhov (D-A) was calculated

Table 2
Geochemical analysis from Rock-eval results for shale samples [6].

Sample	TOC (wt%)	S_1 (mg/g)	S_2 (mg/g)	S_3 (mg/g)	HI	OI	PI	T_{max} °C
AC2-1	3.03	0.54	1.95	0.16	64	5	0.22	459
AC2-2	0.64	0.13	0.25	0.17	39	27	0.34	458
AC2-3	1.82	0.33	1.15	0.03	63	2	0.22	460
AC2-4	1.08	0.19	0.52	0.09	48	8	0.27	465
AC2-5	0.23	0.04	0.10	0.19	43	83	0.29	N/A

Note: T_{max} of sample AC2-5 is not available as the TOC is too low to measure.

Table 3
Moisture content of shale samples.

Sample		AC2-1	AC2-2	AC2-3	AC2-4	AC2-5
Moisture content	$(m_w\text{-}m_{dry})/m_{dry}$ (g/g)	0.022	0.019	0.021	0.02	0.015

Table 1
Mineralogical composition from XRD results for shale samples [6].

Sample	Quartz (%)	Smectite (%)	Mixed illite/smectite (20%S)	Illite + mica (%)	Kaolinite (%)	Chlorite (%)	Total Clay (%)	Carbonates (%)
AC2-1	24.5	1.7	19.8	24.9	2.6	7.1	56	4.8
AC2-2	53.0	1.6	11.7	13.9	0.8	3.1	31.1	2.1
AC2-3	41.3	0.9	14.9	18.9	0.8	5.2	40.7	2.8
AC2-4	53.8	1.7	10.4	12.4	0.5	2.9	27.9	4.6
AC2-5	44.5	1.9	9.7	16	1.2	4.3	33.1	6.5

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