Effect of particle size on high-pressure methane adsorption of coal

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Received May 15, 2016; Accepted August 6, 2016

Abstract: Adsorbed gas cannot be neglected in the evaluation of coalbed methane and shale gas since a significant proportion of gas is stored in the form of adsorbed gas. Adsorbed methane of coal and shale has been widely studied by high-pressure methane adsorption experiment. In sample treatment of the experiment, the sample is crushed and sieved to a particular particle size range. However, how particle size influence high-pressure methane adsorption is still unclear. In this study, low-pressure nitrogen (N_2) and high-pressure methane adsorption have been measured on coal samples with different particle size. According to N_2 sorption analysis, pore volume and surface area increase with particle size reduction. Pore size distribution of small pores (<10nm) changes among varying particle size. Pore volume proportion of small pores (<10nm) increases and pore volume proportion of big pores (>10nm) decreases with decreasing particle size. Decreasing particle size by crushing sample introduces new connectivity for closed pores to the particle surface. The responses of isotherms of high-pressure methane adsorption are different with different particle size. Methane adsorption at initial pressure (145psi) increases with decreasing particle size. Adsorption increase rate at high pressure (435-870psi) decreases with particle size reduction. This can be explained that fine sample has more pore volume and higher pore volume proportion of small pores (<10nm). Sample with particle size of 150-250μm has the highest Langmuir volume.

Key words: *particle size, high-pressure methane adsorption, coal, shale gas*

1 Introduction

Natural gas production in shale gas and coalbed methane is perspective, especially when taking gas adsorption properties into consideration. Hydrocarbon gas (mostly methane) can be stored in pores and fractures, and as adsorbed gas on organic and inorganic matter. However, heterogeneous pore structure and complex chemical composition in shale and coal make the quantification of gas storage challenging (Labani et al., 2013). To evaluate methane adsorption capacity under reservoir condition, one reliable and popular research method is high-pressure methane adsorption experiment (Chalmers and Bustin, 2007a; Chalmers and Bustin, 2007b; Ross and Bustin, 2009; Zhang et al., 2012; Gasparik et al., 2014).

Prior to the high-pressure adsorption experiment, the sample is crushed and sieved to a particular particle size. Surprisingly, different particle size has been used in high-pressure methane adsorption related studies (Ross and Bustin, 2009; Zhang et al., 2012; Gasparik et al., 2014). Table 1 shows particle size used in high-pressure methane adsorption related studies. A study by Clarkson and Bustin (1999) has suggested that a particle size of 4mesh (<4.75mm) and 60mesh (<0.250mm) coal sample has a negligible effect on high-pressure methane adsorption (Clarkson and Bustin, 1999). Another study on pure clay samples has shown that methane adsorption capacity of clay minerals increases with decreasing particle size because of the enlarged internal surface area (Ji et al., 2012). Furthermore, the effect of particle size on gas adsorption porosimetry and high-pressure

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CO_2 adsorption has been investigated as well (Chen et al., 2015; Lutynski and González González, 2016). The gas adsorption porosimetry study on shale has shown that micropore volume generally increases with particle size reduction. The high-pressure CO_2 adsorption study on coal and shale has shown that coal samples have different composition among varying particle size, and shale samples get extended surface area with decreasing particle size. Although high-pressure methane adsorption has been widely used, the effect of particle size on the high-pressure methane adsorption is not clear.

In this study, we measured high-pressure methane adsorption on the same coal samples but with different particle size. Low-pressure N_2 sorption was also engaged to give an insight into the pore structure of the studied samples. This study attempted to find out if and how particle size of sample influence high-pressure methane adsorption.

Table 1 Particle sizes used in high-pressure methane

Literature	Particle size (μm)
Gasparik et al, 2013	500-1000, <100
Ross and Bustin, 2009	<250
Chalmers and Bustin, 2012	<250
Zhang et al., 2012	150-500

2 Materials and methods

2.1 Materials

For the purpose of this study, the particle size of the sample was the only experimental variable. Herein, commercial coal provided by BM Alliance coal operations Pty Ltd was used. Table 2 shows the sample information. The mean vitrinite reflectance of the sample is 1.43%.

For the purpose of this study the original coal was sieved to four particle size range: <325mesh, 200-325mesh, 60-100mesh, and 18-20mesh.

Table 2 Composition of the studied sample

Telovitrinite (%)	71.1
Detrovitrinite (%)	3.7
Fusinite (%)	3.3
Semi-Fusinite (%)	13
Macrinite (%)	0.3
Inertodetrinite (%)	4
Mineral matter (%)	4.5

2.2 Methods

2.2.1 High-pressure methane adsorption

High-pressure (up to 870psi) methane adsorption was measured at 30°C by a commercial volumetric apparatus (High pressure volumetric analyzer) (Fig 1). The experiment setup basically consists of a vacuum pump, an outgassing furnace with a temperature controller, a sample cell, a thermostat bath connected with sample cell for controlling the experimental temperature and a reference cell connected to two pressure transducers (high pressure transducer and low pressure transducer). All valves are controlled by a software on the connected computer. Pressure and temperature are precisely recorded. Prior to the experiment, helium expansion is used to measure the void volume, which is defined as the total volume of helium that can penetrate in the sample cell with the sample inside. After that, the system completely evacuated and methane dosed into the reference cell. As soon as the pressure equilibrium criteria are met (pressure variation less than 0.01 bar in one minute or waiting for 30 minutes after dosing the gas into the reference cell), methane is injected into the sample cell. Methane volume dosed in the system could be calculated based on pressure, temperature, sample cell volume and the gas compressibility factor. The gas compressibility factor is determined by Peng-Robinson equation of state. The amount of adsorbed methane in sample cell is calculated by the static volumetric method as follows:

$$n_{ads} = n_{dosed} - n_{void} \quad (1)$$

Where n_{ads} is the amount of moles adsorbed by the sample, n_{dosed} is the amount of moles dosed into the system, and n_{void} is the amount of moles occupying the void volume.

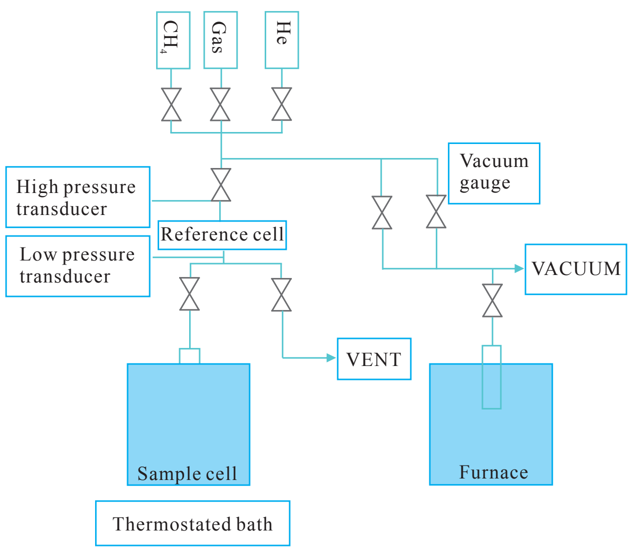

Fig. 1 Schematic diagram of high pressure volumetric analyzer

Gas adsorption is usually described by isotherms, the amount of adsorbed gas as a function of pressure at constant temperature. For methane adsorption on coal and shale, Langmuir equation is used to fit experiment result in many studies. According to Langmuir equation, the amount of adsorbed methane (V_{ads}) can be expressed as follows:

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