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Journal of Petroleum Science and Engineering

journal homepage: www.elsevier.com/locate/petrol

Evaluation of pore size spectrum of gas shale reservoirs using low pressure nitrogen adsorption, gas expansion and mercury porosimetry: A case study from the Perth and Canning Basins, Western Australia



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ARTICLE INFO

Article history:

Received 5 November 2012

Accepted 9 November 2013

Available online 20 November 2013

Keywords:

gas shale

pore structure parameters

low pressure nitrogen adsorption

mercury porosimetry

ABSTRACT

Gas shales have a complex pore structure. Mechanisms of gas storage in the gas shale pore system are in two ways, free gas and sorbed gas. The nanometer scaled pore systems of gas shale reservoirs have a prominent contribution for gas storage, especially for adsorbing gas onto their surfaces. In this study three different methods of low pressure nitrogen adsorption, mercury porosimetry and gas expansion were used for pore structure characterization of gas shales. Mercury porosimetry and gas expansion methods have been used for a long time in characterization of conventional reservoirs but low pressure nitrogen adsorption has been considered recently as a tool for gas shale evaluation. The studied gas shale samples are coming from the Perth and Canning Basins, Western Australia. Analyzing the results of case study shows that the Canning shale samples have the specific surface area and micro/mesopore volume around $13 \text{ m}^2/\text{g}$ and $1.4 \text{ cc}/100 \text{ g}$, respectively, which are relatively higher than the same values for the Perth shale samples.

Quantitative analysis of the obtained results clarifies the shape, size and pore volume of the studied gas shale samples. However analyzing the results shows that there is not any consistency between similar parameters like effective porosity or pore size distribution (PSD) extracted from these techniques; several explanations have been proposed for justification of this inconsistency. As well as the results of this study make it clear that each of the usual techniques applied for characterization of gas shale pore systems has some deficiencies and cannot be used alone for this purpose. Whereas, by combining the results of these methodologies pore size spectrum of gas shales can be determined in a more accurate way.

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1. Introduction

Pore system characterization is an important step for evaluation of gas shale reservoirs. Therefore it is necessary to use new and more effective techniques to understand the pore structure, gas storage mechanisms and the relationship between pore size and gas storage capacity. The gas is stored in the gas shale reservoirs in the form of free gas and adsorbed gas. The adsorbed gas refers to the gas which can be attached to the surface of the clay minerals or organic materials. To have a better understanding about the adsorbed gas capacity of the gas shale reservoirs it is necessary to measure gas adsorption in both high pressure adsorption and low pressure adsorption analyses. The high pressure adsorption measurement is required to determine the

adsorbed gas capacity at reservoir pressure and temperature using the Langmuir isotherm curve (Lu et al., 1995; Ross and Bustin, 2007a). The low pressure adsorption measurement is very important for characterization of the gas shale pore system, pore size distribution (PSD) and studying the parameters which control the adsorbed gas capacity such as surface area and microporosity. PSD is required for fluid flow modeling because gas flow in the shale matrix is expected to be a combination of diffusive transport regime in nanopores and conventional Darcy flow models in larger pores (Javadpour et al., 2007). Low pressure adsorption measurement has been used extensively in surface chemistry analysis for characterization of porous materials but it has been hired for characterization of the nanopores in the shale samples recently (Ross and Bustin, 2009; Kuila and Prasad, 2011; Chalmers et al., 2012). In addition to the low pressure adsorption measurement technique there are other techniques which can be used for pore system characterization like helium pycnometry and mercury porosimetry (Giesche, 2006; Bustin et al., 2008; Ross and Bustin, 2009; Chalmers et al., 2012). The previous studies have mainly

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focused on determination of pore structure parameters of the shale samples. The present study hires low pressure nitrogen adsorption, gas expansion and mercury porosimetry to clarify the shape, size and pore volume of the studied gas shale samples. As well as it will provide new insights about the inconsistency between similar pore structure parameters derived from different techniques and deficiencies in the available techniques for evaluation of gas shale reservoirs.

Generally, in describing the pore size in shales the pores are all considered to fall within the nanopore range (Javadpour et al., 2007; Javadpour, 2009; Loucks et al., 2009) without any further classifications. Recently Loucks et al. (2012) defined a new pore size classification for mudrocks, however, in this study it has been preferred to use the pore size terminology of the International Union of Pure and Applied Chemistry (IUPAC), which was developed by Rouquerol et al. (1994). According to this pore classification pores are subdivided into three categories: micropores which include pores less than 2 nm diameter, mesopores which comprise pores with diameters between 2 and 50 nm, and macropores which include pores with diameters larger than 50 nm.

Perth and Canning are two major sedimentary basins of Western Australia with high potential for gas shale reservoirs. The Perth Basin is a north-northwest trending, mostly onshore sedimentary basin extending about 1300 km along the southwest margin of Western Australia. It contains two main organic rich shale formations with gas development potential including the Permian Carynginia Formation and the Triassic Kockatea Shale. Canning is a super basin in northwest of Western Australia. Based on the initial evaluations, this basin has two primary gas shale targets including the organic rich Ordovician Goldwyer Formation and the Carboniferous Laurel Formation. According to the U.S. Energy Information Administration (EIA) (2011) report recoverable gas shale resources are estimated to be in excess of 59 trillion cubic feet (tcf) for the Perth Basin and around 230 tcf for the Canning Basin.

In the current study two sets of gas shale samples were studied, 17 samples from the Perth Basin (12 samples from the Carynginia Formation and 5 samples from basal member of the Kockatea Shale named Hovea member) and 6 samples from the Goldwyer Formation of the Canning Basin. Table 1 lists the available results of XRD and geochemical analyses for some studied samples from the Perth Basin. Total organic carbon (TOC) content for the available samples range between 0.23 and 3.03 wt%. T_{max} which could be tied to thermal maturity of the samples varies between 458 to 509 °C, however as it is clear the samples RB2-S1, RB2-S2 and RB2-S3 have the higher T_{max} values and therefore they are in the higher thermal maturity status compared to AS2 series samples. The XRD results show the large variability in the mineralogical composition. While RB2 series samples are rich in clay

content, most of the AS2 samples are rich in quartz content except AS2-S1.

2. Experimental methodologies

2.1. Gas expansion

Porosity measurement on the shale samples present several challenges. Gas expansion technique is an old fashioned procedure for measuring effective porosity of a rock sample. However to do this technique on the shale samples it needs some degree of modification. Due to the low porosity of the shale samples (usually less than 5% pu) the equilibration time between the sample cell and reference cell is extra-long. Therefore measuring porosity using core plug is not feasible. Luffel and Guidry (1992) recommended a new evaluation technique for porosity measurement of the shale samples. According to their procedure the shale samples should be crushed in order to increase the surface area and decrease the equilibration time. As a result, in the current study Luffel and Guidry (1992) procedure has been followed. The studied samples were crushed to yield particle sizes between 12 and 60 mesh sizes (1.40 mm and 250 μ m). The crushed samples should be heated to remove gas, free water and any other possible hydrocarbons. In order to achieve this, the samples were heated at 110 °C for 8 h. The main concern during heating the shale sample is preserving the organic materials and the clay bound water. Easley et al. (2007) identified and quantified the evaporated components of the Barnett shale samples during heating using thermogravimetric analysis (TGA) in conjunction with a gas chromatograph. Based on their study during heating the shale samples up to 400 °C, only water becomes mobilized. Their results showed that at higher temperatures kerogen, carbonate minerals and clay bound water were liberated from the samples. Considering this point the procedure used for heating the shale samples in the present study is not expected to have any effect on the matrix of the shale samples. It is worth mentioning that in this study helium was used for porosity measurement.

2.2. Low pressure nitrogen adsorption

Low pressure nitrogen adsorption (< 18.4 psia) can be used to obtain the following information in microporous materials (Gan et al., 1972):

- specific pore volume: total pore volume per mass of the sample expressed as cm^3/g ,
- shape of the pores,
- specific surface area: total surface area per mass of the sample expressed as m^2/g , and
- pore sizes and their distribution.

The nitrogen adsorption and desorption isotherms were collected at 77 K (–196 °C) using a Micromeritics® TriStar II 3020 apparatus. Samples were crushed to < 250 μ m to be used for low pressure isotherm analysis. Traces of gas and water molecules available in the sample compete with the nitrogen molecules for adsorption sites, therefore, it is required to remove moisture content and degas the samples prior to pore structure analysis (Bustin and Clarkson, 1998; Busch et al., 2007). For drying the shale samples, the samples were oven dried for 8 h at 110 °C similar to the preparation procedure for gas expansion method.

In the following section there is a brief explanation on the theory behind the extraction of pore volume, pore size, pore shape and surface area based on the results of low pressure adsorption measurement.

Table 1

Geochemical analyses and mineralogical composition of some samples from the Perth Basin.

Sample name	TOC content (wt%)	T_{max}^a (°C)	Quartz (wt%)	Clay (wt%)	Carbonate (wt%)
AS2-S1	3.03	459	25	56	5
AS2-S2	1.36	466	49	34	5
AS2-S7	0.64	458	53	31	2
AS2-S8	1.82	460	41	41	4
AS2-S9	1.08	465	54	28	4
AS2-S10	0.23	n/a	45	33	6
RB2-S1	2.99	484	18.2	49.6	–
RB2-S2	2.54	481.5	20.2	52.7	9.7
RB2-S3	1.43	509	42	48.1	–

^a T_{max} is one of the output parameters of the Rock-Eval pyrolysis and could be tied to thermal maturity of the rock sample.

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