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Research paper

Pore characteristic analysis of a lacustrine shale: A case study in the Ordos Basin, NW China

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ABSTRACT

Organic shales deposited in a continental environment are well developed in the Ordos Basin, NW China, which is rich in hydrocarbons. However, previous research concerning shales has predominantly focused on marine shales and barely on continental shales. In this study, geochemical and mineralogical analyses, high-pressure mercury intrusion and low-pressure adsorption were performed on 18 continental shale samples obtained from a currently active shale gas play, the Chang 7 member of Yanchang Formation in the Ordos Basin. A comparison of all these techniques is provided for characterizing the complex pore structure of continental shales.

Geochemical analysis reveals total organic carbon (TOC) values ranging from 0.47% to 11.44%, indicating that there is abundant organic matter (OM) in the study area. Kerogen analysis shows vitrinite reflectance (Ro) of 0.68%–1.02%, indicating that kerogen is at a mature oil generation stage. X-ray diffraction mineralogy (XRD) analysis indicates that the dominant mineral constituents of shale samples are clay minerals (which mainly consist of illite, chlorite, kaolinite, and negligible amounts of montmorillonite), quartz and feldspar, followed by low carbonate content. All-scale pore size analysis indicates that the pore size distribution (PSD) of shale pores is mainly from 0.3 to 60 nm. Note that accuracy of all-scale PSD analysis decreases for pores less than 0.3 nm and more than 10 μm. Experimental analysis indicates that mesopores (2–50 nm) are dominant in continental shales, followed by micropores (<2 nm) and macropores (50 nm–10 μm). Mesopores have the largest contribution to pore volume (PV) and specific surface area (SSA). In addition, plate- and sheet-shaped pores are dominant with poor connectivity, followed by hybrid pores. Results of research on factors controlling pore structure development show that it is principally controlled by clay mineral contents and Ro, and this is different from marine systems. This study has important significance in gaining a comprehensive understanding of continental shale pore structure and the shale gas storage–seepage mechanism.

1. Introduction

Conventional resource opportunities are diminishing, therefore, unconventional resources like shale gas are being exploited. Advanced techniques such as horizontal drilling and multi-stage hydraulic fracturing have significantly promoted the commercial development of shale gas in North America. To date, the geological reserves of shale gas reservoirs are estimated to be approximately 456.24×10^{12} m^3 globally (Perry and Lee, 2007), with shale gas currently contributing approximately 17% of the total domestic gas production in the USA (Bai et al., 2013). The success of shale gas production in the USA has encouraged other countries such as Canada, Poland, Australia, India, Argentina, and China to explore their potential. Especially in China (Li et al., 2012), shale gas, as one kind of unconventional resource, is an important energy supplement. The geological reserves of shale gas reservoirs are estimated to be approximately 134×10^{12} m^3 in China (MLR, 2012). In April 2011, the Liuping-177 well located in the Ordos Basin was successfully drilled by Shanxi Yanchang Petroleum, representing the

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first continental shale gas well in China. Production was successful (initial gas production was 2350 m^3/d), thereby demonstrating the existence of continental shale gas reservoirs in China (Liu et al., 2013; Jiang et al., 2014). In addition, previous studies have shown the potential of shale gas resource to be considerable in the Ordos Basin (the geological reserves of shale gas is 1.51×10^{12} m^3) (Ding et al., 2013; Liu et al., 2013; Jiang et al., 2014).

Shales are fine-grained sedimentary deposits that were deposited in marine, marine–continental, and continental environments. Globally, shale gas is almost entirely produced from marine shales (e.g., Barnett Shale in the Fort Worth Basin, USA; Horn River Shale in the Western Canadian Sedimentary Basin of Canada; and Longmaxi Shale from the Sichuan Basin of China), with only a small proportion being produced from non-marine shales. For example, the shale gas reservoir from the Upper Triassic Yanchang Formation in the Zhiluo-Xiasiwan area in the Ordos Basin of central China consists of continental shales, with large shale gas resource potential (the volume of shale gas is 626.4×10^8 m^3 to 6037.2×10^8 m^3) (Liu et al., 2013; Jiang et al., 2014; Wang et al., 2012).

Shale is a heterogeneous porous media with a complex pore system, and it is possible that shales in these reservoirs contain pores of various sizes (Bustin et al., 2008; Clarkson et al., 2012). According to pore size terminology from the International Union of Pure and Applied Chemistry (IUPAC) (Rouquerol et al., 1994), pores can be classified into three groups: micropores (pore diameters less than 2 nm), mesopores (pore diameters between 2 and 50 nm), and macropores (pore diameters more than 50 nm). This pore size classification has been widely utilized to describe gas shale pore systems (Clarkson et al., 2012; Ross and Bustin, 2009; Labani et al., 2013). Although the knowledge of pore structure characteristics of porous systems at different scales is very important for characterizing the storage, transport, and production of shale gas, such characteristics are unfortunately not yet well understood.

Various experimental techniques have been employed to investigate the characteristics of the complex pore structure in shales, and these can be summarized into three types: microscopic observations, radiation detection, and fluid invasion (Wang et al., 2015a,b). Microscopic observations are used to observe pore type, pore distribution, pore geometry, and pore size. Such observations include the use of field emission scanning electron microscopy, transmission electron microscopy, computed tomography (CT) scan, and focused ion beam scanning electron microscopy (Bernard et al., 2012; Chalmers et al., 2012a, b; Loucks et al., 2009, 2012; Wei et al., 2013; Yang et al., 2013a). Recently, radiation detection including small angle and ultra-small angle neutron scattering techniques (SANS/USANS) have been successfully applied to describe the pore structure of shale reservoirs (Clarkson et al., 2012, 2013). Fluid invasion, which can reflect nanoscale to micron-size pores (Bustin et al., 2008; Wang et al., 2015a,b), is a promising and widely used technique for investigating qualitative pore shapes and statistical characteristics of pore-size distribution (PSD), where techniques such as low-pressure gas adsorption/desorption experiments, mercury intrusion, and helium pycnometry (Chalmers and Bustin, 2007; Ross and Bustin, 2007, 2009; Chalmers et al., 2012a, b; Clarkson et al., 2012b, 2013; Tian et al., 2013; Yang et al., 2013b, 2014) are used. These methods can also be used to obtain the total pore volume (PV) and specific surface area (SSA).

In this study, three laboratory methods, low-pressure N_2 adsorption, CO_2 adsorption, and high-pressure mercury intrusion, are used to characterize the pore structure of continental shales from the Chang 7 member of the Yanchang Formation in the Ordos Basin. Each method has a limitations because of the nature of the rock and application conditions involved with the technique. For example, mercury is a non-wetting-phase fluid and has difficulty accessing shale nanoscale pores. As high-pressure mercury invasion method can cause artificial fractures, the technique is mainly used to analyze macroporous range pores, and thus, mercury intrusion is proficient at calculating the PSD of pores that are between 30 nm and 200 μm (Bustin et al., 2008). Low-pressure N_2 physisorption experiments are conducted at a temperature of less than −196 °C (melting point of liquid nitrogen), and N_2 adsorption isotherms are collected at −196 °C to reflect mesopores (2–50 nm) (Bustin et al., 2008). Therefore, low-pressure N_2 physisorption is used to calculate the PSD of mesopores by Barrett–Joyner–Halenda (BJH) model, and the SSA is calculated by using low-pressure N_2 physisorption analysis with the multipoint Brunauer–Emmett–Teller (BET) equation and PV is calculated by BJH model (Gregg and Sing, 1982). CO_2 physisorption occurs at a temperature of 0 °C, providing the necessary molecular kinetic energy for CO_2 molecules to enter microporous pores approaching a size of 0.35 nm (Bustin et al., 2008). Therefore, CO_2 physisorption can detect micropores, and the micropore PSD can then be calculated using the Dubinin–Radushkevich (DR) model. Thus, using a combination of the three methods, shales can be characterized effectively and comprehensively.

2. Geological setting

The Ordos Basin, located on the northwestern North China plate, is a polycyclic superposition basin with geological reserves for shale gas reservoirs of approximately 19.9×10^{12} m^3 (MLR, 2012). The Ordos Basin can be divided into six structural units (Xiao et al., 2005): the Yimeng uplift zone in the north, the Weibei uplift zone in the south, the Jinxi flexural fold zone in the east, the Yishan slope in the midsection, the Xiyuan obduction zone, and the Tianhuan depression in the west (Fig. 1). Of these units, the Yishan slope has a gentle westward tilt and stable deformation structures and is currently a major area of petroleum production. The Archean Eonothem and Paleoproterozoic basement of the Ordos Basin has undergone five evolutionary stages (Wang et al., 2011; Jiang et al., 2013): Meso–Neoproterozoic aulacogen, Early Paleozoic shallow marine platform, Late Paleozoic strand plain, Mesozoic inland depression, and Cenozoic fault depression. Overall, the Ordos Basin has an eastern regional tilt to the west with a gentle slope and a lack of anticlines and faults. Structural activities spanning from the late Triassic to the Upper Cretaceous have profoundly affected hydrocarbon generation, migration, and accumulation (Yang, 2002). Late Triassic tectogenesis formed a large scale inland freshwater lacustrine environment in the internal part of the Ordos Basin, where the Yanchang Formation was deposited.

The study area is located on the southeast Yishan slope of the Ordos Basin (Fig. 1), an area generally lacking detailed information on pore structure characteristics (Liu et al., 2015; Sun et al., 2015; Ji et al., 2014). The focus is the Chang 7 member of the Yanchang Formation of the Upper Triassic (Fig. 2). The Chang 7 member deposits consist of deep and semi-deep lacustrine oil shale facies that are mostly black or blackish gray, with elevated organic carbon contents and act as one of the main source rocks in study area (Jiang et al., 2013; Sun et al., 2015). The thickness of the Yanchang Formation is 300–3000 m, with the Chang 7 member burial depth being 500–2000 m (Jiang et al., 2013).

3. Samples and experimental methods

3.1. Shale samples

Several wells were drilled to evaluate the continental shale gas reservoirs in the study area, but only 10 wells were cored (Fig. 1). In total 46 shale samples between 1000 m and 2000 m, were collected