Marine and Petroleum Geology 57 (2014) 509–520

Contents lists available at ScienceDirect

Marine and Petroleum Geology

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

Research paper

The composition and its impact on the methane sorption of lacustrine shales from the Upper Triassic Yanchang Formation, Ordos Basin, China

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ARTICLE INFO

Article history:
Received 18 April 2014
Accepted 21 May 2014
Available online 2 June 2014

Keywords:
Yanchang shales
Methane sorption
Residual bitumen
Clay minerals
Ordos Basin

ABSTRACT

The organic geochemistry, mineralogy and methane sorption of lacustrine shales of the Upper Triassic Yanchang Formation, collected from the south-eastern Ordos Basin, were investigated to characterize them and clarify the effects of shale composition on their sorbed gas capacity. These Yanchang shales have recently been selected as a target area for shale gas exploration in typical terrestrial strata in China. The two main sections of these shales containing type II organic matters, Chang 7 and Chang 9, have relatively high total organic carbon content (TOC) of 2–10%. The two shales also have similar mineralogies, mainly comprising quartz, clay minerals and feldspars. Both the Chang 7 and Chang 9 shales are generally in the oil window; Chang 9 is slightly more mature than Chang 7. Higher methane sorption capacity was observed for Chang 9 than for Chang 7 shales, determined on a dried basis at 50 °C. Methane sorption measurements were further performed on three samples from which the residual bitumen had been extracted, and their corresponding kerogen fractions, to gain insight into the effects of shale composition on methane sorption. This was significantly higher in solvent-extracted samples than in raw samples, indicating that residual bitumen largely restricts methane sorption on such shales. A positive correlation between the amount of clay minerals and methane sorption capacity of bulk rocks was evident, suggesting that clay mineral content is relevant to methane sorption. This result was also supported by the much higher methane sorption capacity of solvent-extracted shales compared to the extracted kerogen from those shales, when measured sorption data was normalized to TOC values. The effects of both residual bitumen and clay mineral on the methane sorption of bulk rocks have complicated the evaluation of methane sorption on organic matter in these mature shales.
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1. Introduction

Successful exploration and production of shale gas in USA has greatly encouraged investigations into the composition (both organic and inorganic) and the gas potential of shales from North America and Europe in the past ten years (Bowker, 2007; Chalmers and Bustin, 2007a, 2008a,b; Curtis, 2002; Gasparik et al., 2014; Hammes et al., 2011; Jarvie et al., 2007; Montgomery et al., 2005; Rexer et al., 2013, 2014; Ross and Bustin, 2007, 2008, 2009; Selley, 2012; Strąpoć et al., 2010). In the past few years, Chinese geologists have shown increasing interest in the composition and gas potential of shales (Chen et al., 2011; Han et al., 2013; Wang et al., 2013), and shale gas was licensed as a new type of mineral resource by the Chinese Government in 2011 to accelerate the exploration of shale gas. The reported results from China have exclusively focused on the very old marine shales from South China, e.g. very high-maturity Cambrian and Silurian strata (mostly VRo > 2.0%). Permian, Triassic, Jurassic, and Cretaceous to Paleocene lacustrine shales containing very abundant organic matter are widespread in northern China. These have contributed greatly to

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http://dx.doi.org/10.1016/j.marpetgeo.2014.05.010
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petroleum production in China (Duan et al., 2008; Gong et al., 2010; Tang et al., 2010; Zou et al., 2013). They are generally less mature than the marine shales.

Shale gas is stored differently depending on three types of geological environment: free gas in pores and/or fractures, gas sorbed onto kerogen and clay minerals and, to a lesser extent, gas dissolved in residual bitumen (Curtis, 2002). Isotherm sorption of methane onto shales under high-pressure conditions, and the relationship between shale composition and sorbed methane capacity, have been extensively studied to determine the gas potential of shales. The percentage of sorbed gas in the total gas varies greatly (Chalmers and Bustin, 2007a; Lu et al., 1995; Montgomery et al., 2005; Ross and Bustin, 2008). Positive correlations between the amount of organic matter and the sorbed methane capacity have frequently been reported (Chalmers and Bustin, 2007a, 2008a; Chen et al., 2011; Gasparik et al., 2014; Han et al., 2013; Lu et al., 1995; Ross and Bustin, 2007, 2009; Zhang et al., 2012).

These have suggested that organic matter exerts primary control over sorbed gas, but also indicated the significant effects of organic matter on the pore structure of shales (especially pores at the nanoscale) (Curtis et al., 2012; Loucks et al., 2009; Rexer et al., 2014; Ross and Bustin, 2007, 2009; Slatt and O'Brien, 2011; Valenza et al., 2013). The type of organic matter also affects the sorbed methane capacity. It is higher for type II/III kerogen, when normalized to the organic matter content, than for type I/II kerogen (Chalmers and Bustin, 2008a). Zhang et al. (2012) further suggested that kerogens with mainly aromatic structures adsorbs methane more readily than with mainly aliphatic structures.

Relatively high-maturity shales have a higher capacity for sorbed methane than those of low maturity, for comparable organic matter and moisture contents (Gasparik et al., 2014; Jarvie et al., 2007; Ross and Bustin, 2007, 2009). This phenomenon might be related to the development of organic porosity in shales of elevated maturity (Curtis et al., 2012; Valenza et al., 2013). Some clay minerals, e.g. montmorillonite and illite, have abundant micropores and also a high methane sorption capacity (Ji et al., 2012; Liu et al., 2013; Lu et al., 1995; Ross and Bustin, 2009), and have been reported to contribute to the methane sorption on shales when measured on a dried basis (Chalmers and Bustin, 2008a; Gasparik et al., 2012; Rexer et al., 2014).

For shales of normal maturity, the residual bitumen filling the pores or pore throats (Jarvie et al., 2007; Ross and Bustin, 2009) complicates the evaluation of the effects of shale composition on methane sorption. After bitumen is extracted using an organic solvent, the surface area of mature shales increases (Valenza et al., 2013). A recent study (Guo et al., 2014) further suggested that removing the residual bitumen leads to a large increase in the sorption of N_2 and CO_2 on mature shales. As a result, the calculated surface areas and volumes both of the micropores and mesopores of bitumen-extracted shales were much higher than in shales where the residual bitumen has not been extracted. However, to date this effect has not previously been investigated for methane sorption on mature shales under high pressure.

Lacustrine shales from the Upper Triassic Yanchang Formation are some of the most important petroleum resources in China. Preliminary investigations have indicated a good gas potential (Wang et al., 2012), and detailed characterization is accordingly much needed. This study investigated the organic and mineral compositions of 41 core samples from the Yanchang Formation, collected from the south-eastern Yishan slope of the Ordos Basin (Fig. 1a). Ten bulk samples were selected for methane sorption analysis. A further three samples were chosen to demonstrate the effect of the composition of the shale on the methane sorption by mature shales, by comparing the methane sorptions of bulk rock and bitumen-extracted rock, and their corresponding kerogens.

2. Samples and experiments

2.1. Geological background and samples

The Ordos Basin in northern-central China (Fig. 1a) is the second largest sedimentary basin in China, with vast oil and gas reserves (Dai et al., 2005; Duan et al., 2008). It is an intracratonic depression basin covering an area of approximately 32×10^4 km^2 (Li, 1996) and is well known as one of the most tectonically stable basins in China (Dai et al., 2005). It comprises six major structural units (Duan et al., 2008): the Yimeng uplift in the north, the Weibei uplift in the south, the Tianhuan depression and western edge thrust belt in the west, the Jinxi fold belt in the east, and the central Yishan slope (Fig. 1a). The Ordos Basin is a large asymmetrical syncline with a broad, gently dipping eastern limb and a narrow, steeply dipping western limb. The Tianhuan Sag forms the axis of the syncline (Ding et al., 2013). The Yishan slope, which covers the largest area of the Ordos Basin, dips at less than 1° toward the west. It is the main oil and gas exploration and production area in the basin. One main source unit is Upper Paleozoic strata (Dai et al., 2005) marked by Carboniferous–Permian coal measures; another is the lacustrine shales in the Yanchang Formation in Upper Triassic strata (Duan et al., 2008). Generally, most of the large conventional gas fields occur in the northern Yishan slope, and have been genetically related to the Carboniferous–Permian coal measures (Dai et al., 2005). Most of the oil fields in the basin occur in the Southern Yishan slope, with oil mainly originating from the Yanchang shales (Dai et al., 2005; Duan, 2012; Duan et al., 2008; Wang et al., 1995). The area has recently been targeted for shale gas exploration in terrestrial strata, and the Yan'an region (Fig. 1a) was selected in 2012 as a national demonstration zone for shale gas in China. The first vertical well and the first horizontal well in this area, specifically for gas exploration from terrestrial shales, were drilled in 2011 and 2012, respectively, and hydraulic fracturing was also carried out.

The Yanchang shales, which are divided into 10 sections (Fig. 1b), result from the evolution of lake deposits in the Late Triassic (Yao et al., 2009). Sections Chang 9 and Chang 10 were deposited during the genetic and expanding stage of the lake basin, which later reached its peak stage. Chang 7 developed rapidly once this peak stage had been reached. During the deposition period of the Chang 4, 5 and 6 sections, the area and depth of the lake basin significantly decreased. The lake basin subsequently evolved into its final stage, and Chang 1, 2 and 3 sections were mainly deposited in the resulting deltaic environment. Oil shale, black shale and carbonaceous shale are very common in semi-deep and deep-lake conditions in the Chang 7 section, and petroleum resources have been well correlated with the shales in this section, which contains abundant organic matters (Duan et al., 2008; Zhang et al., 2009). The Chang 9 shales also have a very high potential for oil generation (Zhang et al., 2007). In the present study, shale samples from the Chang 7 and 9 sections were collected from 20 wells drilled in the south-eastern Yishan slope (Fig. 1a, Table 1). These are denoted Chang 7 and Chang 9 shales for the convenience of discussion in this context.

2.2. Experimental analysis

2.2.1. Geochemical analysis

Shale cores were crushed and −200 mesh material samples were selected for geochemical analysis. About 100 mg was placed in a crucible with 5% HCl at 80 °C to remove carbonates. A Leco C230 carbon analyzer measured the total organic carbon content (TOC in Table 1). A Vinci Rock Eval 6 instrument determined the type, maturity and hydrocarbon generation potential of the organic

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