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Research paper

Pore characteristics of lacustrine shale within the oil window in the Upper Triassic Yanchang Formation, southeastern Ordos Basin, China

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A R T I C L E I N F O

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A B S T R A C T

Organic matter (OM)-rich shale in the Yanchang Formation is recognized as a promising hybrid shale oil/shale gas system in lacustrine strata in China. Being mainly in the oil window, both the type and distribution of the pores in the shale have been affected by several factors, including petroleum expulsion and retention, mineral and organic composition, and compaction. To obtain a better understanding of the factors controlling OM pore development, 10 core samples were selected with various OM content from the Chang 7 and Chang 9 members of the Yanchang Formation for pore characterization. The investigation combined microscopic observation, both of thin sections and ion milled surfaces of samples with low-pressure carbon dioxide and nitrogen adsorption and bulk porosity measurements, with the main emphasis being on OM-associated pores.

The selected samples generally have relatively low total porosity, with the pores being poorly connected. Most pore types found in marine shales were present. The size of porous OM is usually small, mostly measuring several hundreds of nanometers to a few micrometers, which suggest a general compaction effect on pore development. The pore development is related to different petroleum expulsion processes. Shale with very porous OM was a very important feature. The OM coexisting with fluorescent lipitinites was present in a cross-linked nanofiber structure, possibly related to altered extracellular polymeric substances (EPS) by compaction and maturation. Various OM types that varied in both size and shape did not contain any visible pores (e.g. woody relics, migrated solid bitumen and OM laminae in close association with clay minerals). Unusually low meso- and macropore volume in the sample with the highest total organic carbon (TOC) content was confirmed by the predominance of OM laminae without visible pores; however, abundant micropores were indicated by CO_2 adsorption analysis. Meso- and macropores in this sample had probably not developed due to a significant compaction effect. Due to the small numbers of studied samples, these OM pore characteristics need to be viewed with caution.

1. Introduction

Pores in shale not only provide the space for petroleum storage, but also form the flow-path network for liquids and gases. Therefore, the porosity and distribution characteristics of pores (e.g., their size, type and connectivity) are important for resource potential evaluation and development of unconventional shale reservoirs (Bustin et al., 2008; Loucks et al., 2009; Ross and Bustin, 2009; Schieber, 2010; Sondergeld et al., 2010; Wang and Reed, 2009). Most of the investigated shale units, ranging in age from the Late Proterozoic to the Paleogene, were deposited in marine environments (Chalmers et al., 2012; Fishman et al., 2012; Klaver et al., 2015; Löhr et al., 2015; Loucks et al., 2009, 2012; Lu et al., 2015; Mastalerz et al., 2013; Milliken et al., 2013; Pommer and Milliken, 2015; Ross and Bustin, 2007, 2009; Slatt and O'Brien, 2011; Zeng et al., 2016). However, lacustrine shales have contributed a large proportion of conventional petroleum production in several parts of the world, including China, Africa, Brazil, etc. (Katz, 1990; Katz and Lin, 2014). They may, therefore, have unconventional resource potential. Lacustrine shales differ largely from marine shales due to their limited geographical distributions, their sensitivity to very frequent changes in climate, and the large number of interbedded sand and mud layers (Fang et al., 2016; Katz and Lin, 2014; Ko et al., 2017;

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Lei et al., 2015; Wang et al., 2016). Organic matter (OM)-rich shales in the lacustrine Yanchang Formation are the major sources of oil in the Ordos Basin with proved reserves of about 30.75×10^8 t until 2012 (Yang et al., 2013), which is one of the most productive petroliferous basins in China. The Chang 7 Member of the Yanchang Formation (abbreviated to Chang 7 shale here) has recently been demonstrated as one of the most promising hybrid shale oil/shale gas systems in lacustrine strata in China (Loucks et al., 2017; Wang et al., 2014, 2016; Yang et al., 2013, 2016; Zhang et al., 2015a,b). The total porosity in this member has mainly been controlled by its clay mineral content (Fu et al., 2015; Jiang et al., 2016; Wang et al., 2015), which is consistent with the predominance of intraparticle pores in clay platelets and a relatively high clay mineral content (Loucks et al., 2017; Wang et al., 2016). The Chang 7 shale probably lacked early cementation during diagenesis, and thus the lithification was less developed, but the compaction effect was more extensive (Ko et al., 2017). In general, the average size of OM pores was found to be much smaller in the Chang 7 shale than in Eagle Ford marine shale (Ko et al., 2017); in addition, the sizes and types of interparticle pores show significant differences between the silty laminae and clayey laminae of the Chang 7 shale (Lei et al., 2015), which is related both to silt grain-size and sorting degree (Ko et al., 2017). Besides the Chang 7 Member of the Yanchang Formation, a few samples have also been investigated for the Chang 9 Member (Ko et al., 2017; Loucks et al., 2017), abbreviated to Chang 9 shale here, which is also rich in OM (Guo et al., 2014a; Zhang et al., 2007).

Shale within the oil window was affected by compaction, cementation and self-source oil infilling, which has greatly lowered the size and number of primary interparticle pores (Ko et al., 2016; Loucks and Reed, 2014; Löhr et al., 2015; Mastalerz et al., 2013; Pommer and Milliken, 2015). However, secondary pores within OM begin to develop during petroleum generation and subsequent expulsion as a combined result of hydrocarbon loss, volume change of residual organic solids, and exsolution of gases (Guo et al., 2017; Jarvie et al., 2007; Loucks et al., 2009, 2012; Löhr et al., 2015; Valenza et al., 2013). Most of the Chang 7 and Chang 9 shales from the southeastern Ordos Basin are in the oil window, as suggested by the range of vitrinite reflectance (VRo) values between 0.7% and 1.2% (Fu et al., 2015; Guo et al., 2014a; Jiang et al., 2016; Tang et al., 2014). Pores in the OM in Chang 7 shale have been commonly observed to be less developed than the intraparticle pores within clay and mica aggregates (Loucks et al., 2017; Wang et al., 2016); however, OM pores dominated the pore systems of many of the samples investigated (Ko et al., 2017). The major factors influencing the development of OM pores in the Chang 7 shale have not so far been elucidated in detail. Residual OM in the Chang 7 shale from the southeastern Ordos Basin is mainly solid bitumen (about 95% by volume of the total OM) and minor vitrinite, inertinite, alginite and waxy terrestrial liptinite (Hackley et al., 2017). Palynofacies analysis of the Chang 7 and Chang 9 shales from the central, southern and southeastern parts of the Ordos Basin has provided evidence that the shales are rich in *Botryococcus* algae, acritarchs and amorphous organic matter (AOM), with minor amounts of vitrinite and inertinite also present (Ji et al., 2006, 2009, Ji and Xu, 2007; Zhang et al., 2015a). The relationship between OM maceral type and OM porosity is not clear. Scanning electron microscopy (SEM) observations have been combined with low-pressure gas adsorption measurements in recent studies to gain an insight into the pore characteristics of the Chang 7 shale (Ko et al., 2017; Loucks et al., 2017; Wang et al., 2015). Nonetheless, possible linkages are yet to be established between the results of FE-SEM observation and those of low pressure gas adsorption for the OM pores in the shale.

In this study, the characteristics of pores, especially those in OM, were mainly described using SEM images of ion-milled surfaces of shale samples from both the Chang 7 and Chang 9 members. To gain an insight into the distributions of pores smaller than the SEM detection limits, and to quantitatively evaluate their characteristics, low-pressure

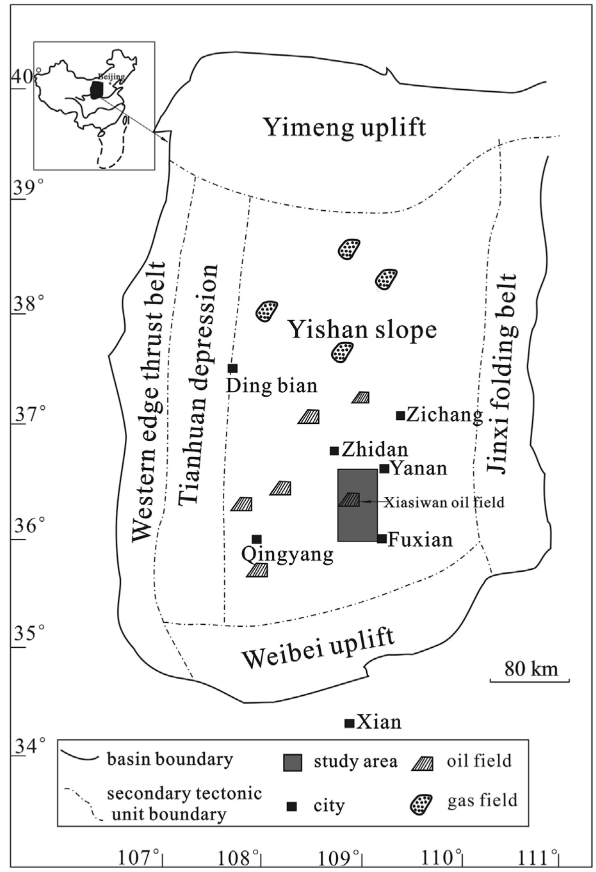

Fig. 1. Simplified map of the Ordos Basin showing major structural units and the distribution of oil and gas fields (modified after Guo et al., 2014a).

CO_2 and N_2 adsorption methods were used in pore-distribution analysis, along with mercury intrusion and helium pycnometry. Through the combined investigations, this study has tried to elucidate the relationships between OM maceral type and OM porosity and between FE-SEM observed pore characteristics and measured pore distributions by low-pressure gas adsorption, and finally to determine the main factors controlling OM pore development in the studied sample set.

2. Samples and experimental analysis

2.1. Geological backgrounds and samples

The geological backgrounds of a relatively large sample set from which the samples were selected in this study were introduced in Guo et al. (2014a). Briefly, the Ordos Basin is located in the western part of the North China Craton. The Ordos Basin is subdivided into six structural units: the Tianhuan Depression and Western Edge Thrust Belt in the west, the Jinxi Fold Belt in the east, the Yimeng Uplift in the north, the Weibei Uplift in the south and the Yishan Slope in the middle (Fig. 1). Continental deposition was initiated as a result of the Indosinian movement in the Late Triassic. The Yanchang Formation represents the whole lacustrine basin evolution and is separated into 10 members (Chang 10 to Chang 1 in ascending order, Fig. 2). The Chang 10 Member was developed early in lake formation, and is mainly composed of sandstone and sandy mudstone which were deposited in a fluvial environment. With continuous subsidence, the lake area became larger, and mudstones are present in the Chang 9 Member in the deep lake facies. The Chang 8 Member was also deposited during an expansion of the lacustrine basin. The Chang 7 Member was deposited during the peak stage of basin development when the water was deepest and the lake area was the largest. Thick black shales/mudstones