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Characterizing the Micropores in Lacustrine Shales of the Late Cretaceous Qingshankou Formation of Southern Songliao Basin, NE China

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Abstract: Micropores of shale are significant to the gas content and production potential of shale, which has been verified in the research of marine shale gas; while, few studies have been conducted on lacustrine shales. This study collected 42 samples from three wells in the Late Cretaceous Qingshankou Formation of the southern Songliao Basin, NE China, and investigated these samples by the focused ion beam-scanning electron microscope (FIB–SEM) and nitrogen adsorption analysis techniques. Four types of micropores were identified in the samples, i.e., intergranular pore, intracellular pore, organic matter pore and microfracture. The pore structure type is characterized by open slit pores and "ink type" pores which are mainly 1.5–5 nm in diameter with mesopores as the main pores. The mesopores account for 74.01% of the pore volume and 54.68% of the pore surface area. Compared with the lacustrine shales from the Triassic Yanchang Formation in the Ordos Basin and Xujiahe Formation in the Sichuan Basin, the intergranular clay mineral interlayer pores are considered to be the main reservoir space for shale gas storage in the study area, followed by intraparticle pores, organic matter pores and microfractures. Maturity and micropore are the key controlling factors which affect the shale gas content of the Qingshankou Formation in southern Songliao Basin.

Key words: micropores, lacustrine shale, Cretaceous Qingshankou Formation, southern Songliao Basin

1 Introduction

In recent years, the theoretical and technological innovations of shale gas have led to the rapid development of shale gas exploration and development. Correspondingly, the research on the microscopic characteristics of shale reservoirs has received much attention among geologists. Shale gas mainly exists on the surface of organic matter and clay minerals in an adsorption state, and also exists in micro–nano pores of the organic matter shale in a free state. It is a typical source-storage integrated and continuous distribution of unconventional natural gas. Shale is developed with low porosity and low permeability, and the micropores provide main storage space for shale gas (Wei Mingqiang et al., 2011; Zou Caineng et al., 2016; Chen Xiaohong et al., 2018). Conventional thin-film observation, scanning electron microscopy and other testing methods can not satisfy the characterization of shale reservoir microstructure, and more precise techniques are needed to qualitatively or quantitatively analyze shale reservoir types, structures and morphology. Four methods are commonly used for studying the pores developed in unconventional reservoirs, i.e., imaging pore systems with SEM on ion–milled samples (Li et al., 2014), 3D reconstruction of pore system using focused ion beam (FIB–SEM), pore size measurements with BET analysis and pore size measurements with Hg–injection and nitrogen adsorption–desorption analysis (Chalmers et al., 2012; Chen Shengrong et al., 2015; Li et al., 2017; Chen et al., 2018). In this study, the microstructure of shale reservoirs was analyzed by the FIB–SEM technique. However, due to the small observation range, it is difficult to accurately describe the micropore distribution of shale. Therefore, we also used the nitrogen adsorption–desorption experimental method to compensate the limitations of the FIB–SEM on microscopic pore research.

There are significant differences worldwide in terms of the classification system of micropores in shale (Wang Weiji et al., 2017). The micropores in the Devonian

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Marcellus, Horn River, Barnett and Haynesville shales have been broadly divided into intergranular pores, intergranular interstices and organic pores (Milner et al., 2010). Scholars divided the micropores in the Barnett shale into two types which are micro-pores of diameter >0.75 nm and nano-pores of diameter <0.75 nm (Loucks et al., 2009; Liu Shugen et al., 2011; Xiao Xiaoming et al., 2015; Zhao Jianhua et al., 2016; Zhao Wenzhi et al., 2016; Wei Xiangfeng et al., 2017), and classified micropores in North American marine shale into intergranular pores, granular pores and organic matter pores (Huang Zhenkai et al., 2013). Some researchers classified the micropores in Paleozoic shales in the Sichuan Basin into three categories, i.e., organic matter nanopores, microcracks in organic matter and nanopores in granules (Zou Caineng et al., 2011; Passey et al., 2010; Javier et al., 2017).

Organic matter pores provide the main reservoir space for shale gas in the marine Devonian shales of North America (Amanda et al., 2009) and the Lower Silurian Longmaxi Formation of southern China (Wang Shejiao et al., 2012; Feng Dongjun et al., 2016). Important discoveries of lacustrine shale gas have also been made in Ordos Basin (Wang Xiangzeng et al., 2016) and Qaidam Basin (Yuan Yuan et al., 2016), and it was found that the microscopic pores have petroleum geological significance. However, for the Songliao Basin, a typical continental basin, there is little research on the micropores of shale. The Late Cretaceous Qingshankou Formation in the southern Songliao Basin with organic-rich shales and thin gray siltstone interbeds (Liu Bo et al., 2014) are important hydrocarbon source rock strata of conventional oil and gas, which are also favorable for shale gas reservation. A total of 133 wells have been found with gas logging abnormality showing an average total hydrocarbon content of 5.20%, and obvious logging response to shale gas. In addition, there has done some research on the micropore characteristics of shale in the Qingshankou Formation of the northern Songliao Basin, while the research in the southern basin is very few. Micropores were classified by studying the core observation and micropore characteristics. Compared the geological characteristics and gas content with the Triassic lacustrine shales from Yanchang Formation in Ordos Basin and the Xujiahe Formation in the western Sichuan Basin, the influence of micropores on gas content is discussed.

2 Geological Setting

The Songliao Basin, a Mesozoic–Cenozoic oil and gas-bearing continental basin, is a faulted-depression composite basin. The southern Songliao Basin can be divided into four primary tectonic units from west to east, including the western slopes, southwestern uplift, central depression and southeastern uplift (Fig. 1). The Songliao Basin has experienced four tectonic stages, i.e., extensional faulting, subsidence, tectonic inversion and Cenozoic faulting (Hu Wangshui et al., 2005; Li Shichao et al., 2017). The depression formations of the Upper Cretaceous from old to young are the Qingshankou, Yaojia, Nenjiang, Sifangtai and Mingshui Formations, respectively. Among them, the 1st, 2nd and 3rd members of Qingshankou Formation and the 1st member of Nenjiang Formation are the main source rocks in the depression period (Lu Shuangfang et al., 2017). The transgression event occurred in the early period of the Qingshankou Formation with the characteristics of overcompensated sedimentation of rapid subsidence and filling. The Qingshankou Formation is rich in organic matter that largely comprises semi-deep and deep lake sediments and includes thick black shales and interbedded oil shales (Guo Wei et al., 2004) (Fig. 2).

3 Samples and Methods

Several wells were drilled to evaluate the lacustrine shale gas reservoirs in the Changling and Wangfu depression of the southern Songliao Basin. Totally 42 shale core samples from three wells were collected from the Upper Cretaceous Qingshankou Formation (Fig. 1; Table 1). The samples were subjected to FIB-SEM, nitrogen adsorption-desorption, gas content test, isothermal adsorption and organic geochemistry analyses (Table 2). The FIB-SEM was conducted with Auriga Compactis model (Zeiss, Germany) by the Institute of Geology and Geophysics, Chinese Academy of Sciences. The FIB-SEM has a resolution of 5 nm and is equipped with Energy Dispersive Spectrometer (EDS) that can be used for elemental analysis and qualitative to semi-quantitative microporosity determinations (Mao Xiaoxiao et al., 2016). The micropore structure and pore size distribution were examined with a Micromeritics Instrument Corporation mesopore–micropore analyzer (model ASAP 2020) by Chengdu University of Technology for determination of single and multi–point BET specific surface area, Langmuir specific surface area, BJH mesopore, pore distribution, pore size, total pore volume and area (Guo Shaobin et al., 2013;Li et al., 2016), adsorption heat, average pore size analysis, aperture measurements from 0.35 to 500 nm, and for microporosity segment analysis with a resolution of 0.02 nm. The detection limit of pore volume is 0.0001 cm^3/g. The gas content and composition analysis was conducted by using YSQ-IV analyzer from Oil and Gas Survey, China Geological Survey.

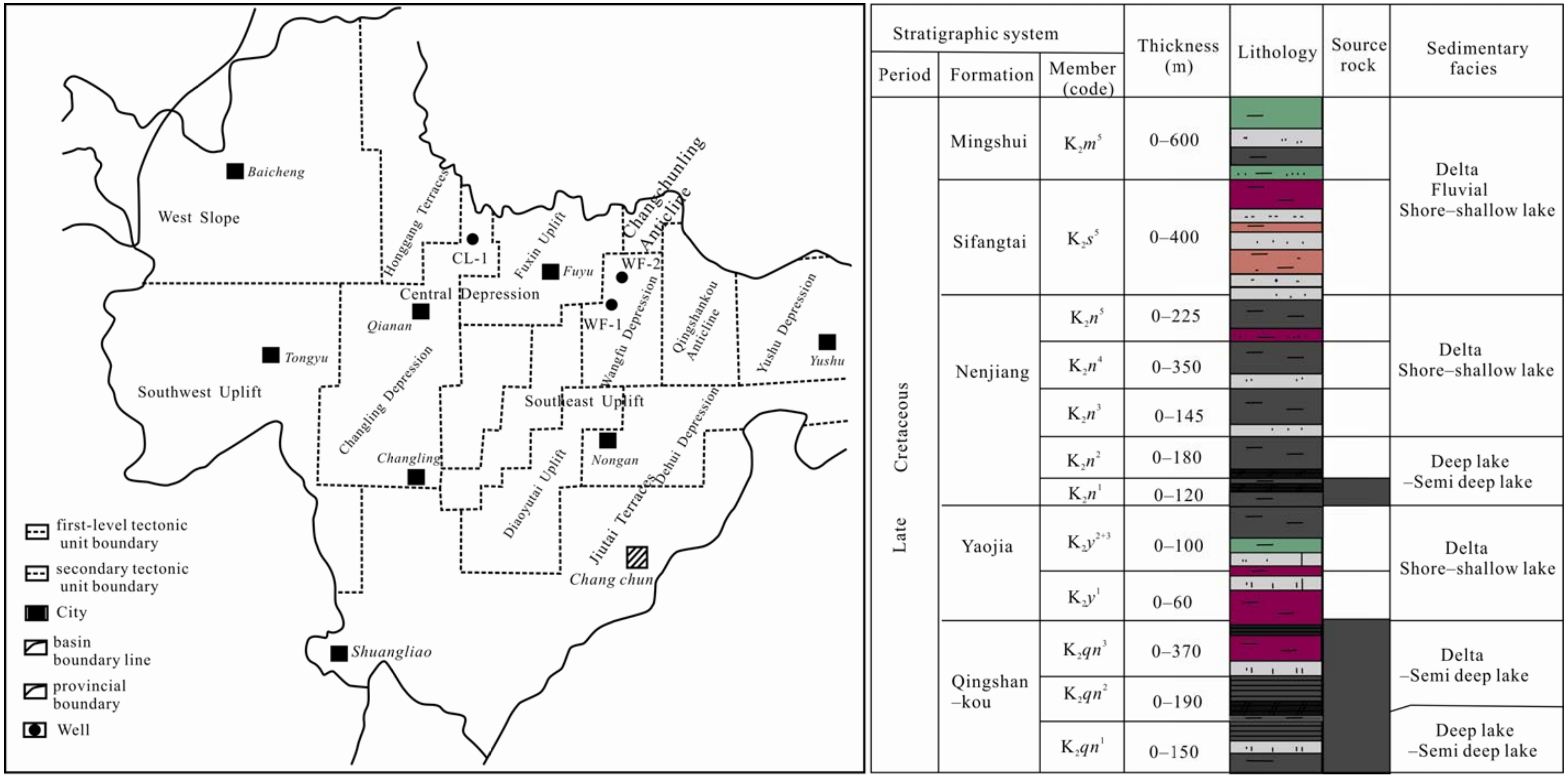

Fig. 1. Geographical location of the study area and stratigraphy.

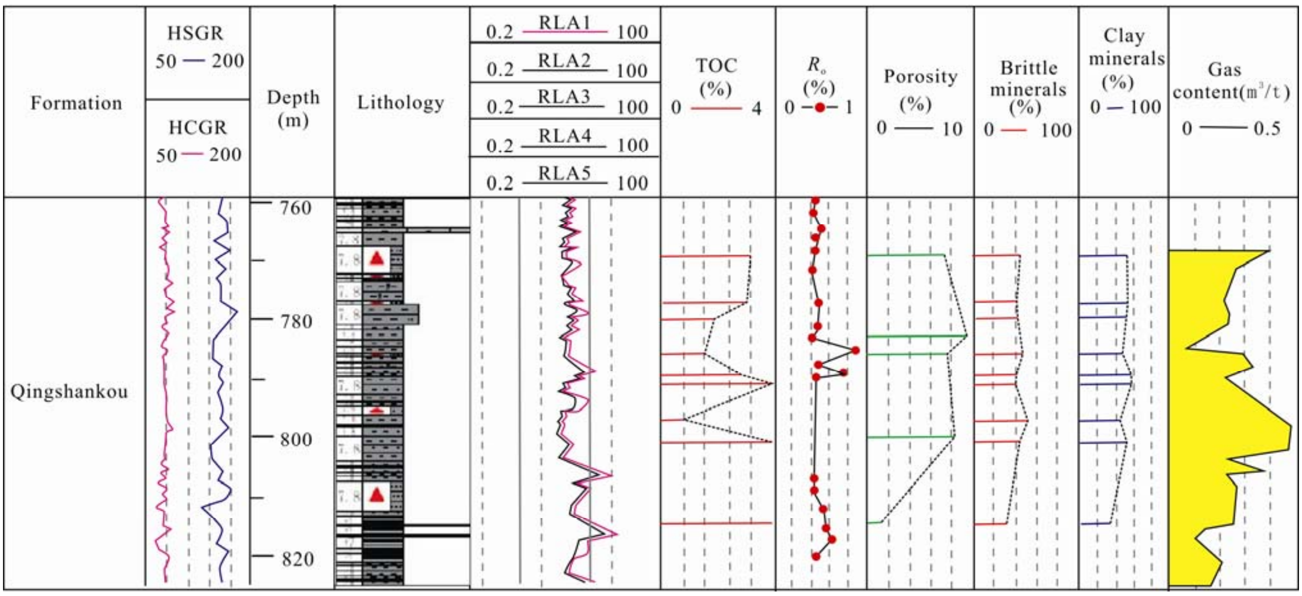

Fig. 2. Comprehensive geological profile of the Qingshankou Formation, WF-1.

Table 1 Samples information from Qingshankou Formation of the southern Songliao Basin

Well	Formation	Depth (m)	Lithology	TOC (%)	R_o (%)	Surface area (m^2/g)	Maximum adsorption volume @STP(cc/g)	Pore volume (cm^3/g)	Pore radius Dv(r) (nm)	Usothermal adsorption	Gas content
WF–1	K_1q_1	675–825	Gray black shale	2.50	0.4–0.86	3.4–21.2	8.3–21.2	0.01–0.03	3.1–7.5	2.1–3.4	0.46–0.74
WF–2	K_1q_1	679–710	Gray black shale	2.13	0.45–0.62	0.8–13.8	1.5–21.3	0.002–0.03	5.2–5.5	1.7–2.6	0.19–0.37
CL–1	K_1q_{2+3}	1987–1992	Black shale	2.32	0.58–1.5	9.6–20.9	14.1–28.4	0.02–0.04	4.2–4.6	1.9–2.1	0.59–0.84

Table 2 Samples experiment information of the Qingshankou Formation of the southern Songliao Basin

Well	Number of samples	FIB-SEM	Nitrogen adsorption-desorption	Gas content test	Isothermal adsorption	Organic geochemistry	Tectonic location
WF–1	25	2	2	25	3	25	Wangfu depression
WF–2	12	2	2	10	3	12	Wangfu depression
CL–1	5	2	2	5	4	5	Changling depression

Note: The unit is the number of sample tests.

4 Experimental Results

The organic matter in the Qingshankou Formation is mainly Type I and II. The total organic carbon (TOC) contents of 42 shale core samples are 0.5%–14% with an average value of 2.15%. The thermal evolution is low and the R_o values range from 0.4% to 1.5% with an average value of 0.76%. The shales are mainly composed of quartz, feldspar, carbonate, pyrite and clay minerals. The brittle mineral content is 30.7%–63%. Clay minerals include mixed illite-smectite (30%–79%), illite (15%–58%), and amounts of chlorite and kaolinite. The shale gas content is 0.19–0.84 m^3/t and the content of methane in the shale is 41.59%–87.76%. The results of isothermal adsorption show that the adsorption capacity is 1.71–3.61 m^3/t, with an average value of 2.97 m^3/t.

4.1 Pore structure types

Based on petrographic and FIB-SEM observations, the micropores of the Upper Cretaceous shale in the southern Songliao Basin can be divided into four types, i.e., intergranular, intragranular, organic matter and microfracture pores. These pores can be further divided into nine subcategories, i.e., interlayer pores in clay mineral, pores between pyrite crystal and clay mineral skeleton, pores in organic mineral particles, pores in clay mineral matrix, pores in clay mineral skeleton, pores in pyrite crystals, primary pores, hydrocarbon generating residual organic pores and edge cracks. We conducted EDS elemental analysis to obtain accurate classification of the micropores.

4.1.1 Intergranular pore

Intergranular pores in study area is commonly found between inorganic mineral particles, clay minerals or two micropores, which are elongate or polygonal and have an irregular distribution. The intergranular pores of the shale are mainly clay mineral interlayer pores (Fig. 3a), or occur in banded pyrite crystals and clay minerals (Fig. 3b), and the inorganic mineral intergranular pores (Fig. 3c). The elongate nature of the pores is related to the structure and origin of the clay minerals, and the polygonal pores are

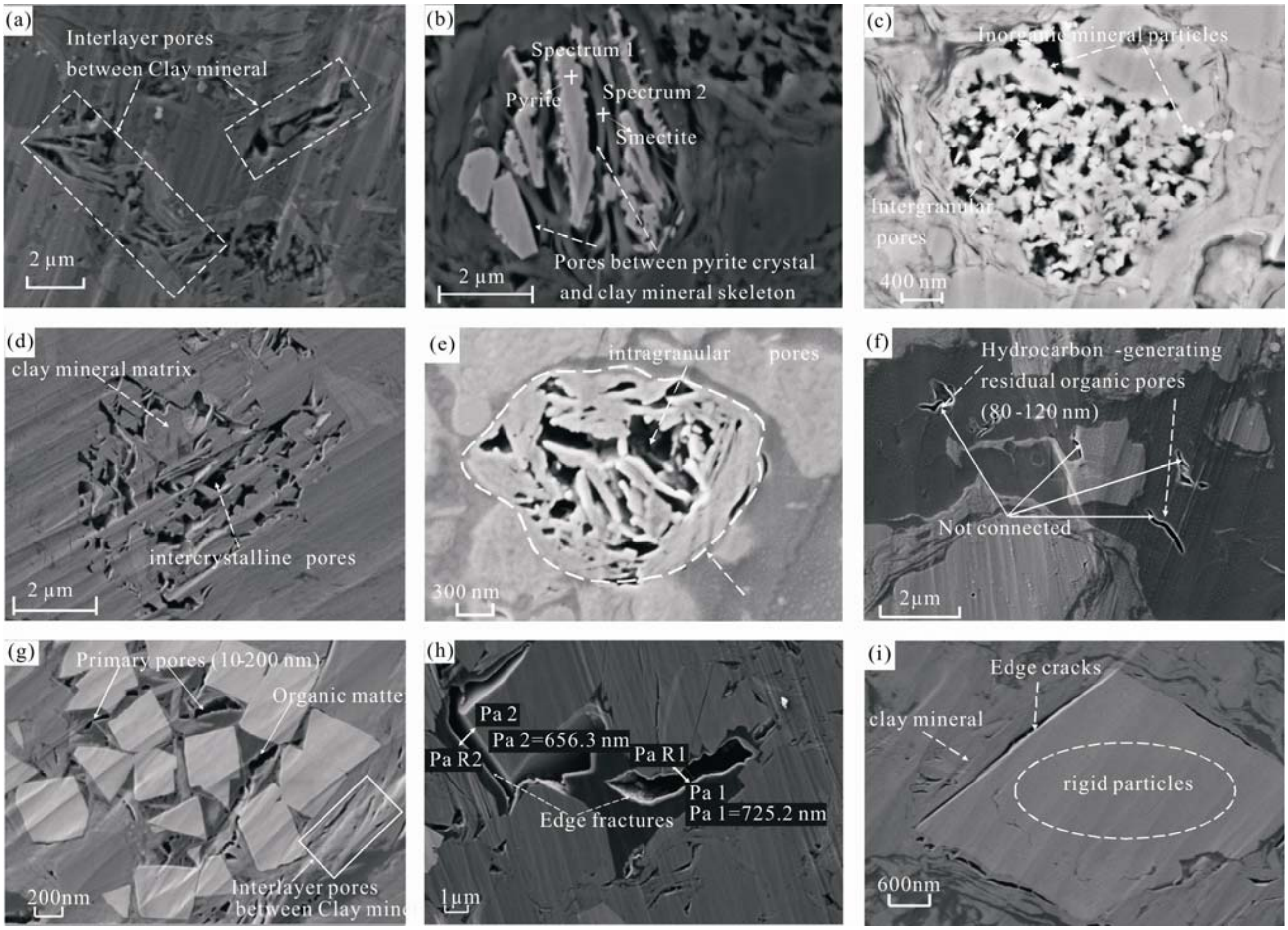

Fig. 3. Types of micropores in the Upper Cretaceous shale of the southern Songliao Basin.
Note: Pa1 and Pa R1 represent the first and last symbol of the length of the first crack; Pa2, Pa R2 represent the first and last symbols of the length of the second crack.

mostly due to the compaction of inorganic mineral particles (Xue Bing et al., 2015).

4.1.2 Intragranular pore

Intragranular pores are mainly formed in late diagenesis stage and are most common in clay minerals and pyrite crystals (Guan Quanzhong et al., 2016). These pores in study area are irregular or nearly spherical and occur as intercrystalline pores within clay minerals in the matrix (Fig. 3d), skeletal intragranular pores in clay minerals (Fig. 3e).

4.1.3 Organic matter pore

According to the origin, the organic matter pores in shale can be divided into two types which are hydrocarbon -generating residual pores and primary pores inside organic matter (Huang Zhenkai et al., 2013). According to the contact medium, these can be divided into three types, i.e., pure pores inside organic matter, composite pores between organic matter and clay minerals, intergranular pores between organic matter and matrix. We used the origin classification method as our classification system.

Hydrocarbon-generating residual pores are characteristic of a relatively high degree of thermal evolution and formed during the generation of organic matter. The pores size ranges from 80 nm to 1.5 μm. The shale samples in the CL-1 well are in the low maturation–mature phase and organic matter having started to be converted to hydrocarbons with pores range in size from 80nm to 300 nm. Such pores also exist in the WF-1 well (Fig. 3f).

Primary pores inside organic matter are usually well developed in the case of a low degree of thermal evolution, and the pores are retained inside the organic mass with sizes of 10–1.5 μm (Huang Zhenkai et al., 2013). The shale samples in the WF-1 well are immature to low–mature and this type of pores is rich with pore sizes range from 10nm to 200 nm (Fig. 3g).

4.1.4 Microfracture

Microfracture is an important storage space for free gas and is also a favorable factor for gas desorption. Edge microfractures are rich in the Qingshankou Formation and generally found between brittle mineral particles (quartz or feldspar), organic and clay minerals. Microfracture width is usually 50–800nm and length less than 5μm (Figs. 3h–i). In Fig. 4 (EDS experiment), the sample of Fig. 3b shows that the white mineral (Spectrum 1) is rich in Fe-S and lacks of Al-Si-O, the gray mineral (Spectrum 2) is rich in Al-Si-O with a small amount of Mg-S. We suggest that the white minerals are pyrite and the gray minerals are smectite (the C present in the spectrum is due to the carbon coating) according to EDS inferring mineral composition.

4.2 Pore structure characterization

The parameters characterizing pore structure of shale are total pore volume, specific surface area and average pore size (Wang Anmin et al., 2017). The specific surface area determines the adsorption capacity of the shale, mainly affecting the content of adsorbed gas. The total pore volume directly represents the shale reservoir space and is proportional to the specific surface area, and the pore size determines the state of shale gas storage. Shale gas mainly exists in free gas in larger pores and adsorbed states in smaller pores (Xue Bing et al., 2015; Chen Lei et al., 2015). The Qingshankou Formation has a wide range of pore structure parameter distribution (Table 1). The experimental results show that the specific surface area of the Qingshankou Formation is 0.832–21.221 m^2/g with an average value of 11.637 m^2/g. When relative pressure (p/p_0) is near 0.98, the adsorption capacity reaches the maximum, and the total pore volume distributes between 0.0023 and 0.0441 cm^3/g (0.0249 cm^3/g on average). The pore size ranges from 3.0911 nm to 7.4855 nm, with an average value of 4.9926 nm.

The results of low-temperature N_2 adsorption–desorption experiments of six shale samples from the Qingshankou Formation are shown in Fig. 5. According to whether the hysteresis loop is closed, it can be divided into closed types (Figs. 5a, 5b, 5c, 5e, 5f) and open types (Fig. 5d) (Chen Lei et al., 2017; Wang Xiukun et al., 2017). According to the hysteresis loop shape, it can be divided into type A and type B. Figures 5a–e illustrate type A examples that have an approximately inverted triangular shape and is reversible when the relative pressure is low (Clarkson et al., 2012). Due to capillary agglomeration, when p/p_0 is around 0.40, the adsorption and desorption branch lines in the isotherm become separate and the desorption curve appears at the first inflection point. When p/p_0 is nearly 0.53, a second inflection point appears. It can be classified to type H2 of the IUPAC classification (a hysteresis loop type), representing "ink type" pores. Figure 5f shows a type B with the adsorption and desorption branch lines in the isotherm are slightly separated and the inflection point of the desorption curve is not obvious (Ambrose et al., 2010; Curtis et al., 2012; Mosher et al., 2013). This is classified to type H3 of the IUPAC classification, representing slit-type structural hole (Sing et al., 1985). Therefore, the shale from the Qingshankou Formation in the southern Songliao Basin mainly has a closed porosity, composed of ink– and slit–type pore structures.

According to BET surface area analysis of six shale samples, the specific surface area of the samples is 0.83–21.22 m^2/g, with an average value of 11.64 m^2/g (Table 1). The pore volume and size distribution obtained using the BJH model are shown in Fig. 6. The pore volume is 0.0028–0.049 cm^3/g, with an average value of 0.028 cm^3/g. Previous studies have shown that the desorption branch provides a good characterization of pore size distribution (Groen et al., 2003). The pore size distribution is of unimodal (Figs. 6a, 6b, 6c, 6e) or bimodal (Figs. 6d, 6f) with a size range of 0.72–65 nm, peaking at 1.5–5 nm. The BJH test results can be used to study the contribution of pore size to surface area and pore volume (Liu Xiandong et al., 2005). According to the hypothesis, pores in materials are rigid with regular shapes and without considering micropores (Rouquérol et al., 1999), the experiment only performs statistical analysis of mesopores

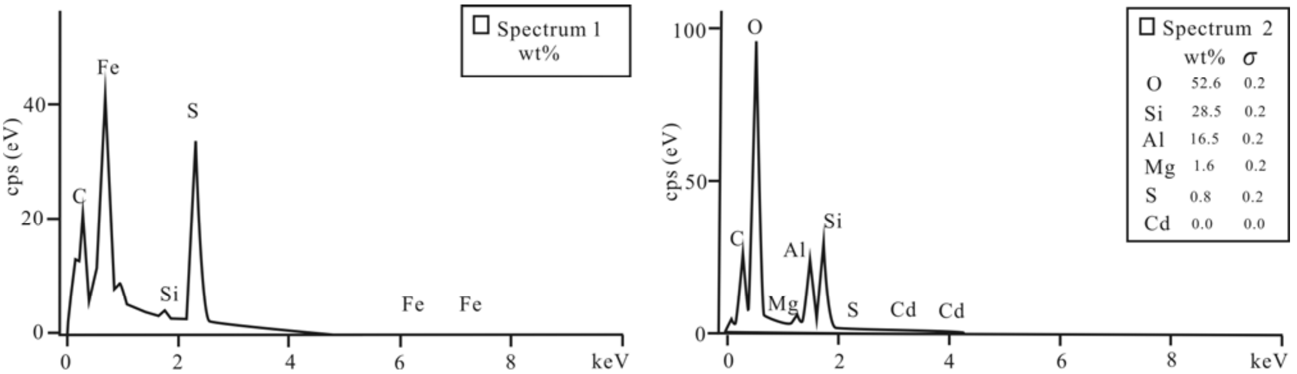

Fig. 4. X-ray EDS qualitative–semi-quantitative element analysis.

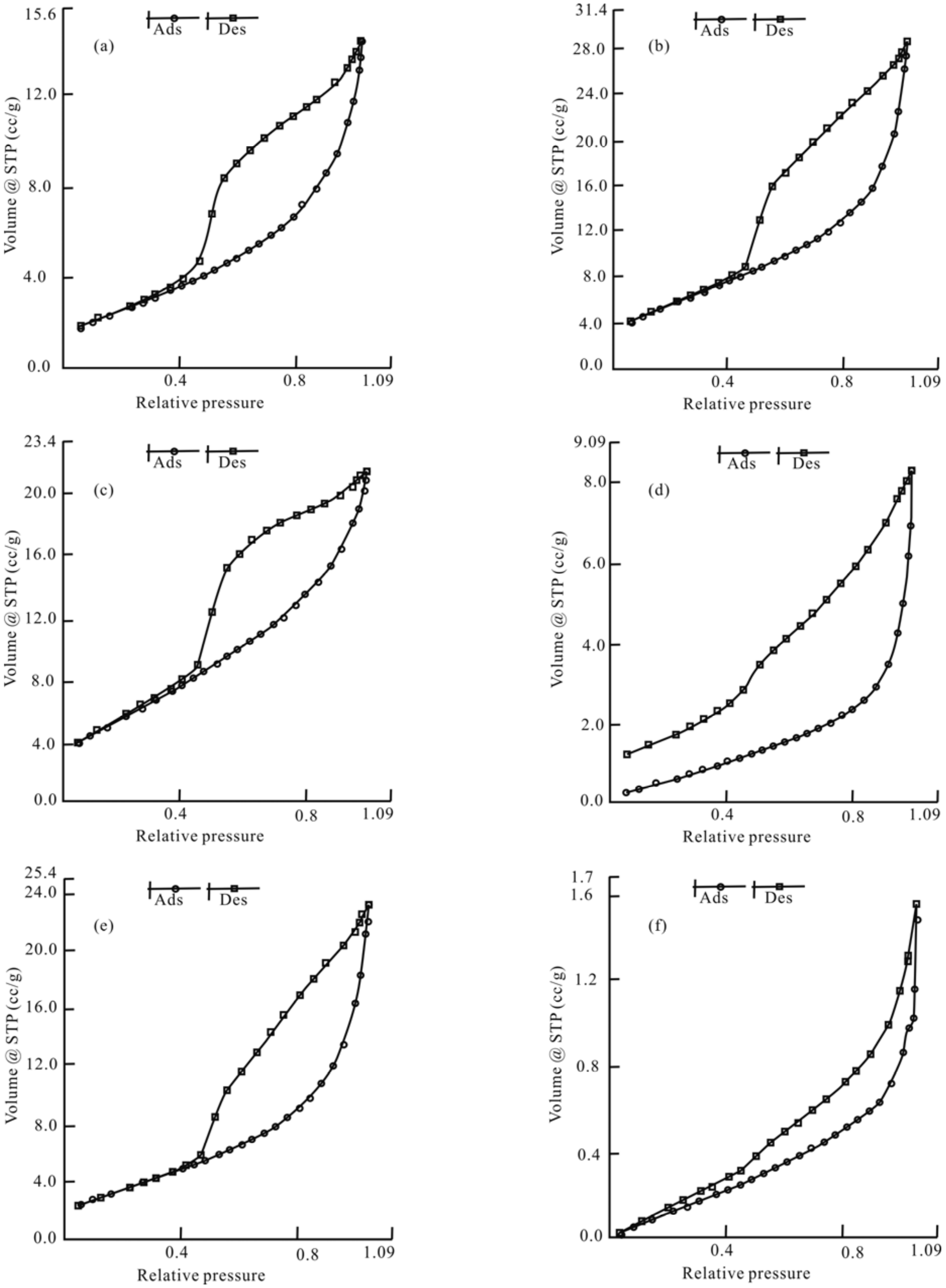

Fig. 5. Low-pressure nitrogen adsorption-desorption experiments on shale samples from the Qingshankou Formation in the southern Songliao Basin.

(a), (b) from CL-1; (c), (d) from WF-1; (e), (f) fromWF-2.

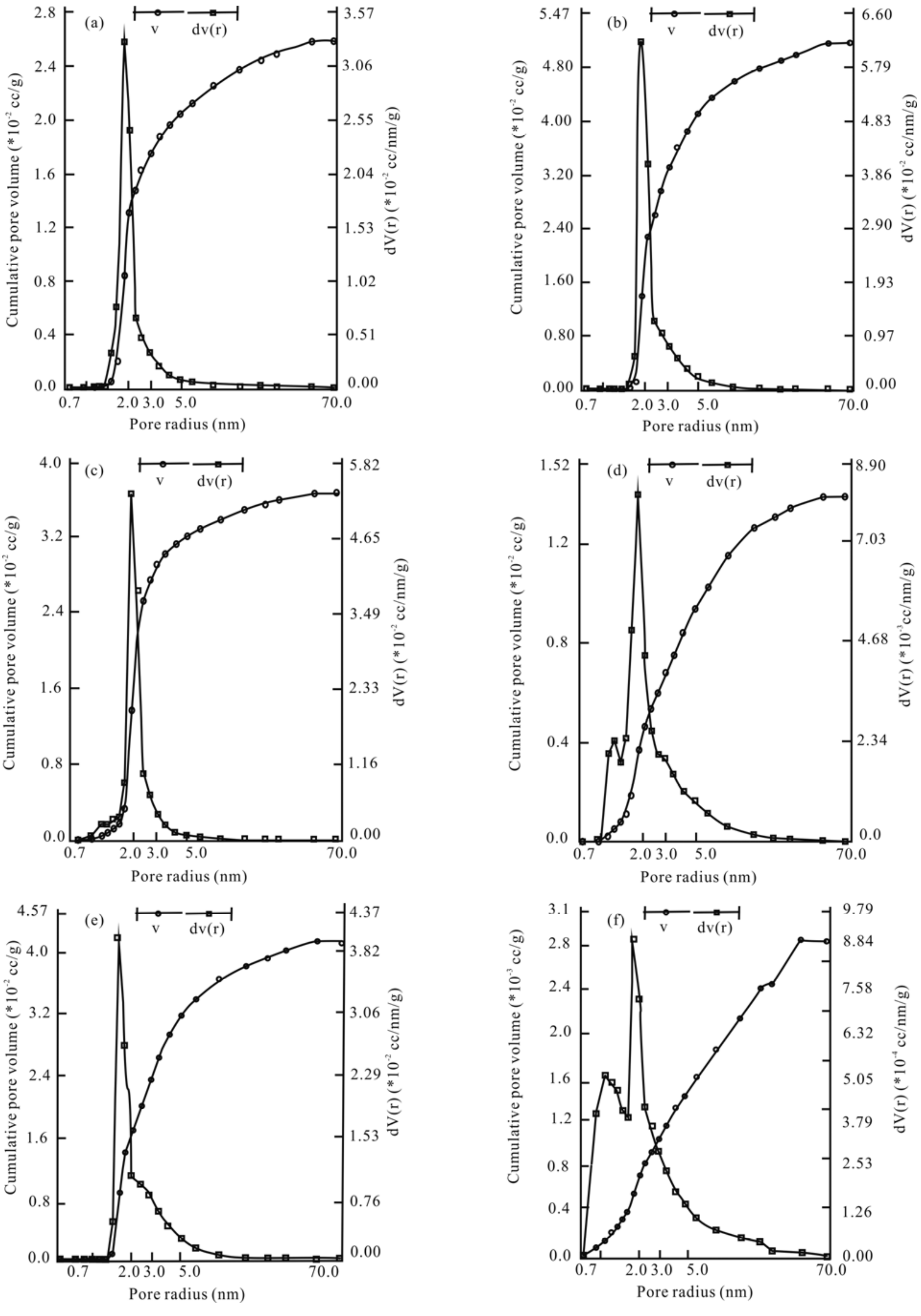

Fig. 6. Shale pore volume and size in the Qingshankou Formation from the southern Songliao Basin.
(a), (b) from CL-1; (c), (d) from WF-1; (e), (f) fromWF-2.

and macropores. The results show that the largest contributor to pore volume and specific surface area is mesopores, accounting for 67.3%–80.9% of the total pore volume and 34.4%–50.6% of surface area, and the macropores are negligible.

5 Discussions

5.1 Micropore types in lacustrine shale

5.1.1 Micropore types in the Qingshankou Formation

The type of micropores has an effect on the gas-bearing in shale (Feng Xiaolong et al., 2017). The intergranular pores in the lacustrine shale reservoirs of Qingshankou Formation are the most abundant, followed by the intragranular pores, organic matter pores and micro-cracks at least (Table 3). The interlayer pores inside clay mineral of the intergranular pores are rich and large in size with good connectivity that is the most effective shale gas reservoir space in the study area. In contrast, other microporosity types are detrimental to shale gas accumulation due to small scale and poor connectivity (Yang et al., 2017).

5.1.2 Other lacustrine shales

To further analyze the porosity of lacustrine shale and its effect on gas content, we compared the shale characteristics of study area with the Yanchang Formation in the Ordos Basin and Xujiahe Formation in the Sichuan Basin.

The Yanchang Formation in Ordos Basin is also a typical set of lacustrine organic-rich shale sediments in northern China (Wang Xiangzeng et al., 2016). The intragranular pores are the most abundant, the fractures and organic matter pores are the second and the intergranular pores are the poorest (Table 3). The mineral inter-pores within clay aggregates are rich, and the pore sizes are large and have good connectivity, which are the most favorable space for reservoir and seepage and have important oil and gas geological significance (Wang Xiangzeng et al., 2016; Cui Jingwei et al., 2017). The intergranular pores between pyrite crystals and the organic matter pores are associated with good connectivity, which are good for shale gas accumulation (Cao Qian et al., 2015).

The Xujiahe Formation in the Sichuan Basin is a typical lacustrine shale in southern China. The intergranular pores in the reservoirs are the most abundant, followed by micro-fractures and dissolved pores, and the organic pores and residual primary porosity are very few in some areas (Huang Jinliang et al., 2016). The interlayer pores between clay mineral in the intergranular pores and micro-cracks are the riches with good connectivity that is the most effective shale gas reservoir space. The dissolution pores are common in calcareous shale in the northwest of Sichuan Basin. Although the intergranular pores of pyrite are common, their contribution for shale gas accumulation is few due to poor connectivity.

By comparison, the microporosity of the three lacustrine shale formations has the following characteristics. Firstly, the shale micropores in the Qingshankou Formation and the Yanchang Formation are classified into four types. Furthermore, the shale of Xujiahe Formation also contains dissolved pores. This is because that the Xujiahe Formation shale in some areas has a high content of carbonate rocks and is formed by the acidic water-soluble corrosion caused by organic matter decarboxylation. Secondly, intergranular pores dominate in shale of the Qingshankou Formation and the Xujiahe Formation, and the intragranular pores dominate in shale of the Yanchang Formation. Thirdly, the organic matter pores are not the main reservoir space of lacustrine shale, which are quite different from marine shale (Wang Liang et al., 2014; Wang Min et al., 2016; Jiu Kai et al., 2016). Fourthly, the primary organic pores in shale of the Qingshankou Formation are the micropores type that lacking in the other two layers and also the unique reservoir space in the low-

Table 3 Comparison of pore types in Qingshankou, Yanchang and Xujiahe formations

<table>
<tr><th>Pores / Formation</th><th colspan="4">Intergranular Pores</th><th colspan="3">Intraparticle Pores</th><th colspan="2">Organic matter pores</th><th>Microfr–acture</th></tr>
<tr><td>Yanchang Formation</td><td>Intergranu–lar pores</td><td>Intergran–ular pores</td><td>Pores between clay mineral</td><td>Rigid particle edge pores</td><td>pores between pyrite crystals</td><td>Mineral inter–pore within clay aggregates</td><td>Pores within particles</td><td colspan="2">Pores within organic matter</td><td>Cutting fracture</td></tr>
<tr><td>Qingshankou Formation</td><td>Interlayer pores Between Clay mineral</td><td>Pores between pyrite crystal and clay mineral skeleton</td><td colspan="2">Pores between inorganic mineral particles</td><td>pores bewteen clay mineral matrix</td><td>Pores between clay mineral skeleton</td><td>Pores within pyrite crystals</td><td>Primary pores</td><td>Hydroca-rbon generate-ng residual organic pores</td><td>Edge fractures</td></tr>
<tr><td rowspan="2">Xujiahe Formation</td><td colspan="4">Intergranular Pores</td><td colspan="2">Dissolved pores</td><td>Residual primary porosity</td><td colspan="2">Organic matter pores</td><td>Microfr-acture</td></tr>
<tr><td colspan="2">Interlayer pores Between Clay mineral</td><td colspan="2">Pores within pyrite crystals</td><td colspan="2">Dissolved pores</td><td>Residual primary porosity</td><td colspan="2">Organic matter pores</td><td>Layer bounda-ry fractures</td></tr>
</table>

mature lacustrine shale.

5.2 Effect of microporosity on gas content

It can be seen from Table 4 that the Qingshankou Formation and the Yanchang Formation have strong similarities in sedimentary facies, shale thickness, organic carbon content and mineral composition (mainly clay minerals), which are quite different from the geological conditions of the Xujiahe Formation (Ye Jun et al., 2008). However, the gas content of Qingshankou Formation is lower than Yanchang Formation and Xujiahe Formation. The research shows that the gas content in Xujiahe Formation is only related to TOC, and the pore type is not the main controlling factor (Wu Yanyan et al., 2015; Xi Zhaodong et al., 2018). The results show that there are two main controlling factors of gas content in Qingshankou Formation which are micropores and maturity.

By the analysis of micropore types and structure, there are two reasons. At first, pores with large-scale and good connectivity are the key pore types that affect the gas-bearing of shale. Compared with the Qingshankou Formation, the Yanchang Formation has more organic matter pores, and the pore types are diverse; the scale is large, the pore size is about 5–1500 nm, the nano-micron organic matter pores are abundant (Wang Xiangzeng et al., 2016), and a large number of micro-pores can greatly increase the permeability of shale (Nie Haikuan et al., 2014; Nie Haikuan et al., 2016). In addition, the organic matter pores of the Yanchang Formation generally exist in the rigid particle skeleton, and the connectivity is greatly increased due to the connectivity of the rigid particles themselves. Secondly, the micro–cracks in the Qingshankou Formation are not as rich as the Yanchang Formation. The fractures of Qingshankou Formation are small in scale, especially the extensibility of cracks is poor, which affects the occurrence of free gas and desorption of adsorbed gas at the later stage. The connectivity of Qingshankou Formation shale is poor and difficult to form a scale, which is not conducive to shale gas accumulation. However, there are more micropore types in shale of the Qingshankou Formation, such as the primary organic pores and edge cracks which are lack of reservoir space for Yanchang Formation.

From the analysis of organic matter maturity, the Qingshankou Formation was lower in particular, the two wells (WF-1 and WF-2) in the Wangfu sag have shallow burial and some of R_o below 0.5%.The low maturity has two aspects on gas content, one is that the organic matter types of the Qingshankou Formation tend to be sapropelic. According to the relationship between R_o and instantaneous hydrocarbon generation rate of shale producing oil and gas in the United States, the shale of the Qingshankou Formation is in shale oil and gas symbiosis, mainly based on oil production, and limited in gas production capacity. The other reason is that the maturity affects the organic matter pores. Previous studies have shown that with the increase of R_o, the matrix porosity in shale decreases, while the change of organic matter pores is different. Some researchers believed that the higher the maturity of shale organic matter, the greater the amount of gas and the richer the organic matter (Jarvie et al., 2007). They also proposed a shale porosity evolution model for the Eagle Ford shale. It is believed that when the source rock depth reaches the hydrocarbon generation threshold, the organic matter porosity increases rapidly and then increases slowly (Cander et al., 2012). According to the analysis results of shale in the Longmaxi Formation and the Qiongzhusi Formation in the Changning and Weiyuan areas (Wang Feiyu et al., 2013), it is believed that the organic matter porosity increases with the increase of organic matter maturity when R_o is 1.3%–2.0%, but when it is >2.0%, the organic matter porosity generally decreases with increasing depth. When the maturity of organic matter reaches a high–over–mature stage, the number and pore size of organic matter pores are greatly increased. Some scholars believe that when the maturity of organic matter reaches a high–over–mature stage, the number and pore size of organic pores are greatly improved. The Qingshankou Formation shale is mainly in the low–mature to mature stage, and a small number of samples are in the mature to high maturity stage. The organic matter pores increase with the increase of maturity, but the lower depth and maturity affect the formation of hydrocarbon-generating residual organic pores which are the most important storage spaces for shale gas (Guo Shaobin et al., 2013). It affected the organic matter porosity and gas content of Qingshankou Formation.

6 Conclusions

(1) There are four main types of micropores in the Qingshankou Formation which are intergranular, intracellular, organic matter pores and microfractures. The pore structure type is dominated by slit– and ink–types,

Table 4 Comparison of basic geological parameters of Qingshankou Formation, Yanchang Formation and Xujiahe Formation

Parameter / Formation	Sedimentary facies	Depth (m)	Thickness (m)	Kerogen type	TOC (%)	R_o (%)	Quartz (%)	Clay mineral	Gas content (m^3/t)
Yanchang Formation	Deep lake – half deep lake	1500	Tens of meters	II_1	2	0.7–1.3	20–25	I/S	1.9–2.69
Qingshankou Formation	half deep lake	700–2000	Hundreds of meters	I, II_1	2.13–2.5	0.4–1.5	25–48	I/S, I	0.19–0.84
Xujiahe Formation	Delta front, shallow lake	2000–5000	30–80	III	2–12	0.6–2.05	12–53	I/S, I	0.84–1.45

and the connectivity is poor. The pores are mainly mesopores and the pore sizes lie in 1.5–5 nm. The contribution of micropores to pore volume and specific surface area is significant.

(2) The main types of storage space for lacustrine shale gas are different. The Qingshankou Formation is rich in intergranular pores, the Xujiahe Formation is rich in intergranular pores and microfractures and the Yanchang Formation is rich in intragranular pores. The organic matter pores are not the main storage space, which are quite different from marine shales.

(3) Micropores development and maturity are the main controlling factors affecting the gas content of Qingshankou Formation, while the intragranular pores and organic matter pores have important petroleum geological significance for the Yanchang Formation, and the Xujiahe Formation is only related to TOC. The shale formations with rich micropores and maturity stage (R_o>0.7%) are favorable areas for further shale gas exploration of the Qingshankou Formation in southern Songliao Basin.

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