Shale Reservoir Characteristics and Sweet Spot Identification of Lower the Cambrian Niutitang Formation in Northwestern Hunan Province, China

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ABSTRACT: The accumulation and productivity of shale gas are mainly controlled by the characteristics of shale reservoirs; study of these characteristics forms the basis for the shale gas exploitation of the Lower Cambrian Niutitang Formation (Fm), Southern China. In this study, core observation and lithology study were conducted along with X-ray diffraction (XRD) and electronic scanning microscopy (SEM) examinations and liquid nitrogen (N2) adsorption/desorption and CH4 isothermal adsorption experiments for several exploration wells in northwestern Hunan Province, China. The results show that one or two intervals with high-quality source rocks (TOC > 2 wt%) were deposited in the deep-shelf environments. The source rocks, which were mainly composed of carbonaceous shales and siliceous shales, had high quartz contents (> 40 wt%) and low clay mineral (<30 wt%, mainly illites) and carbonate mineral (<20 wt%) contents. The SEM observations and liquid nitrogen (N2) adsorption/desorption experiments showed that the shale is tight, and nanoscale pores and microscale fractures are well developed. The BJH volume (VBJH) of shale ranged from 2.144 × 103 cm3/g to 20.07 × 103 cm3/g, with an average of 11.752 × 103 cm3/g. Pores mainly consisted of opened and interconnected mesopores (2–50 nm in diameter) or macropores (>50 nm in diameter). The shale reservoir has strong adsorption capacity for CH4. The Langmuir volume (V0) varied from 1.63 cm3/g to 7.39 cm3/g, with an average of 3.95 cm3/g. The characteristics of shale reservoir are controlled by several factors: (1) A deep muddy continental shelf is the most favorable environment for the development of shale reservoirs, which is controlled by the development of basic materials. (2) The storage capacity of the shale reservoir is positively related to the TOC contents and plastic minerals and negatively related to cement minerals. (3) High maturity or overmaturity leads to the growth of organic pores and microfractures, thereby improving the reservoir storage capacity. It can be deduced that the high percentage of residual gas in Niutitang Fm results from the high reservoir storage capacity of adsorbed gas. Two layers of sweet spots with high storage capacity of free gas, and they are characterized by the relatively high TOC contents ranging from 4 wt% to 8 wt%.

Key words: Northwestern Hunan Province; Niutitang Formation; Reservoir characteristics; Control factors; Sweet spots identification

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1 Introduction

Shale gas has been recognized as an new independent resource type since 2012; it has become a hot spot in the exploration of unconventional resources in China and has brought both opportunities and challenges for oil and gas exploration (Guo et al., 2014; Wang, 2015; Bao et al., 2018; Chen et al., 2018; Zhai et al., 2018; Zhou et al., 2018). In 2017, shale gas productivity reached 10 billion cubic meters (100 × 108 m3) in China. Many studies have been conducted on shale gas exploration and development in Longmaxi Formation (Fm) in Sichuan Basin; these include analyses of facies, determination of gas accumulation conditions, study of reservoir characteristics, and determination of gas-bearing behaviors (Liang et al., 2016; Guo et al., 2014; Huang and Shen, 2015; Wan et al., 2017; Zeng et al., 2016; Zhang et al., 2015 c). With the shale gas exploration campaign in Southern China areas, gas accumulation mechanisms in different facies and gas preservation conditions were considered to explain the controlling factors that affect shale gas accumulation (Guo, 2013, 2016; Guo and Zhang, 2014; Wei et al., 2017; Peng et al., 2017; Xiong et al., 2018). Previous analysis showed that four factors

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control the accumulation and productivity of shale gas: the sedimentary facies, thermal evolution, pores, and fracture development. These factors are closely related to reservoir characteristics (Zou et al., 2015). Barnett shale (Newark East field) in Fort Worth basin of US, which is a typical model for commercial shale gas exploration, is characterized by abundant TOC content, strong gas adsorption capacity, low porosity, low permeability, well-developed organic pores, and high content of brittle minerals (Bowker, 2007; Hu et al., 2015; Jarvie et al., 2007; Tan et al., 2013). The Lower Cambrian Niutitang Fm is similar to the Barnett shale and is a key shale-gas drilling target in Southern China. It is expected to be a site that leads to a breakthrough in shale gas exploration in China (Hao et al., 2013; Hu et al., 2015; Tan et al., 2013). However, the exploration campaign has stalled; tests revealed that only a small number of wells were found to have industrial/commercial flows in Hubei Province owing to the overall low gas content, low percentage of desorbed gas, and uncertainty in sweet spot identification. Sweet spot identification and optimization are indispensable for the successful exploration and development of shale gas. It is extremely important to analyze the characteristics of shale reservoirs and their influences on shale gas occurrence states and to identify sweet spots as targets for exploration and development.

Further investigation on shale as a type of reservoir rock is necessary. Scanning electron microscopy (SEM) tests have shown that nanoscale pores and microscale fractures most likely formed an effective interconnected network and played an important role in migration pathways (Han et al., 2018; Chen et al., 2011; Curtis, 2002; Loucks et al., 2012; Slatt and O'Brien, 2011). Based on field investigation and laboratory analysis, the characteristics of the mineral composition, physical properties, and geochemistry of Niutitang Fm in northwestern Hunan Province were extensively studied (Zhang et al., 2015 a; Zhang et al., 2014; Zhou et al., 2015). The influence of material composition (organic matter and inorganic minerals) on the shale reservoir characteristics such as adsorption capacity, porosity, and permeability were also discussed (Liang et al., 2015; Liu et al., 2017; Loucks and Ruppel, 2007; Tan et al., 2014; Zhang et al., 2012; Lin et al., 2014; Lin et al., 2015; Lyu et al., 2018). There is no consensus among researchers on the reservoir characteristics of Niutitang Fm (Chen et al., 2011; Xue et al., 2015). In summary, there are three debated points: (1) Niutitang Fm is older than the intervals of Upper Devonian gas-producing in the US and lower Silurian in South China. The shale is in high maturity and overmatured stages. Because of organic matter carbonization, high crystallinity of clay minerals, and filling with pores of biologic material, shale reservoirs are generally tight and have poor storage capacity for free gas (Tian et al., 2015; Wang et al., 2016; Zhao et al., 2016). (2) The high TOC content results in a strong storage capacity. However, high maturity or overmaturity reduces the pore volume and adsorption capacity, further affecting the gas-bearing behaviors and occurrence states (Chalmers et al., 2012; Mastalerz et al., 2013; Huang et al., 2015; Tang et al., 2016). (3) There exist different viewpoints on the influence of inorganic minerals (clay minerals, quartz, pyrite, and carbonate minerals) on reservoir characteristics. Further quantitative research on the controlling factors of microscopic pore development and evolution is necessary (Chen et al., 2018 a; Zhang et al., 2014). Meanwhile, there is the need for more quantitative studies on the influence of the reservoir on shale gas occurrence states and identification of sweet spots (Zhang et al., 2015 b).

In this study, the characteristics of shale reservoirs and their impact factors were studied quantitatively. The Niutitang shales were revealed in several wells drilled in northwestern Hunan Province. Liquid nitrogen (N₂) adsorption/desorption and CH₄ isothermal adsorption tests were performed to characterize the shale storage capacity for free gas and adsorbed gas, respectively. Sweet spots were identified by analyzing the influence of the reservoir property on shale gas occurrence states. The results will be of great significance for the shale gas exploration of Niutitang Fm.

2 Regional Geological Settings

Fig. 1 shows the locations of the target wells of this study. Both the northwestern Hunan Province and the Sichuan Basin belong to the Upper Yangtze Plate. The breakup of South China Pangaea Continent in the early Cambrian caused regional tectonic subsidence, and the relative sea level rose rapidly, creating the maximum flooding surface. Niutitang Fm was deposited in a low energetic and anoxic continental shelf environment (Liang et al., 2015). An extensively distributed black shale abundant in carbonaceous and siliceous material was deposited. As shown in Fig. 2, the black shale developed with horizontal bedding and coarsened upward. The upper part is mainly gray silty shale. Owing to the influence of submarine hot water, the shale is rich in elements such as Zn, Cr, As, S, Ba, V, and U, and minerals such as pyrite, barringerite, and barite. The study area is bounded by Baojing–Cili Fault, as shown in Fig. 1. The northwestern side of the area is the folded zone of the Yangtze Plate, and the southeastern side is the Jiangnan–Xueleng Mountain uplift zone. Multiple compression-shearing phases resulted in structural deformation and reconstruction from the Wuling period to Yanshan period in the study area. Accordingly, the NNE or NE-oriented fold and fault system formed, and the local shale gas concentration and preservation were significantly affected by the folding and faulting movements.
Fig. 1 Location map of study area.
(a) Marine shale distribution of the Lower Cambrian Niutitang Fm in South China; (b) the structural map of northwestern Hunan Province. China basemap after China National Bureau of Surveying and Mapping Geographical Information.
3 Samples and Methods

As outcrops cannot represent the parameters of a reservoir, core samples from Niutitang Fm are used in this study (Fig. 2a). For microscopy observation, organic geochemical samples were collected systematically at 2-m intervals, while petrology and reservoir samples were collected every 3 m. In addition, the sampling density was appropriately intensified in good-quality source rock intervals.

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First, the desorbed gas contents of Ciye 1 Well and Yongye 2 Well were obtained by the conventional gas content test method (referring to “Measurement method of shale gas content” SY/T 6940-2013). The residual gas content was obtained from a crushed core in the laboratory. The lost gas content was obtained by using the direct calculation method of the United States Bureau of Mines.

Second, shale samples were subjected to argon ion beam milling in a 691-type transmission electron microscope preparation system, and then, the pores characteristics were observed by using a FEI Quanta 200F field emission scanning electron microscope (resolution: <1.2 nm).

Third, low-temperature liquid N₂ adsorption/desorption experiments were conducted for parallel samples to analyze shale reservoir characteristics. It was measured by a NOVA-2000e specific surface area machine. Pore structures were determined based on the shapes of hysteresis loops. The pore volume and the specific surface area were calculated based on the Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) models. The analysis range of specific surface areas was higher than 0.001 m²/g, and the tested pore diameters varied from 0.35 nm to 500 nm.

Then, parallel samples were conducted for the gas adsorption capacity test with a constant temperature of 30°C. Experimental results were fitted with the Langmuir equation to obtain the isothermal adsorption curve, and the saturated adsorption capacity was calculated (Tan et al., 2013).

4 Results

4.1 Lithology characteristics

One or two layers of high-quality source rocks (TOC > 2 wt%) were defined as deep continental muddy shelf microfacies rocks, as seen in the case of Huaye 1 Well and Ciye 1 Well (Fig. 3). The main lithology of these rocks consists of black or gray-black shale and siliceous shale, which exhibit considerable lamellation. The pyrite presents radial, massive, layered or strip-like shapes. Vermiculite or macle shape barites can be seen at the bottom of the Niutitang Fm. Stone coal, (sapropel coal; calorific values exceed 800 kcal/kg), which developed at the bottom of the Niutitang Fm in Ciye 1 Well, is black scaly, semibright with glass luster. Clear slickensides can be seen in the surface between beddings with plenty of carbon. High dip-angle fractures in the core are filled or semi-filled with calcites veins. Optical microscopy observation shows that the carbon is speckled and wispy. Hydromica, clay minerals, and carbon are directionally arranged. Microfractures are further filled with calcites.

XRD results of 17 organic-rich shale samples from Ciye 1 Well show that the minerals predominantly comprise quartz and clay minerals, and then, carbonate, feldspar, and pyrite (Table 1). Clay minerals are mainly illite (90.1% on average). The mineral composition of Huaye 1 Well is similar to that of Changye 1 Well. Quartz + feldspar + pyrite, carbonate, and clay minerals are selected as the three-terminal elements in Fig. 4. The lithology of the investigated shales is mainly siliceous shale. Similar to Barnett shale, the Niutitang Fm shale has a high content of silica/quartz; as a result, complex matrix fractures are easily generated in the shale during hydraulic stimulation.

![Fig. 3 Lithology characteristics of the studied shales.](image-url)

(a) 2270 m, Ciye 1 Well, slicksides in stone coal, rich in carbon; (b) 2722 m, Ciye 1 Well, vertical fracture; (c) 2597 m, Huaye 1 Well, fracture filled
with calcite veins; (d) 2729 m, Ciye 1 Well, bedding pyrite; (e) 2724 m, Ciye 1 Well, vermicular barite; (f) 2552 m, Huaye 1 Well, black shale, bedding barite; (g) 2653 m, Ciye 1 Well, silt, biological debris (−); (h) 2712 m, Ciye 1 Well, silt, hydromica lineation(−); (i) 2596 m, Huaye 1 Well, slicksides in stone coal, rich in carbon; (j) 2599 m, Huaye 1 Well, carbonaceous silt stone with a microfracture (−); (k, l) 2564.24 m, Huaye 1 Well, energy spectrum analysis of rich barium mineral.

Table 1 Organic geochemistry and mineral content characteristics of the Niutitang Fm with rich organic shale.

<table>
<thead>
<tr>
<th>Well</th>
<th>Depth/m</th>
<th>TOC/wt %</th>
<th>R/o %</th>
<th>Quartz/wt %</th>
<th>Feldspar/wt %</th>
<th>Pyrite/wt %</th>
<th>Carbonate/wt %</th>
<th>Clay Minerals/wt %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Changye 1</td>
<td>1103–1224</td>
<td>0.03–17.6</td>
<td>2.02–3.13</td>
<td>26–94</td>
<td>0–12</td>
<td>1–12</td>
<td>0–85</td>
<td>3–48</td>
</tr>
<tr>
<td></td>
<td>1288–1344</td>
<td>0.75–9.89</td>
<td>2.6</td>
<td>52.3</td>
<td>5.8</td>
<td>4.2</td>
<td>8.7</td>
<td>27.9</td>
</tr>
<tr>
<td>Huaye 1</td>
<td>2483–2508</td>
<td>1.27–3.94</td>
<td>2.0–3.6</td>
<td>21.4–70.4</td>
<td>2.9–16.9</td>
<td>0–17</td>
<td>6.8–58.8</td>
<td>8.9–45.3</td>
</tr>
<tr>
<td></td>
<td>2526–2595</td>
<td>1.16–13.3</td>
<td>2.8</td>
<td>43</td>
<td>8.9</td>
<td>5.7</td>
<td>20.4</td>
<td>27.7</td>
</tr>
<tr>
<td>Ciye 1</td>
<td>2620–2653</td>
<td>1.18–6.44</td>
<td>1.52–2.04</td>
<td>27.6–59.1</td>
<td>6.5–13.9</td>
<td>1.2–22.8</td>
<td>3.8–37.9</td>
<td>16.9–42.9</td>
</tr>
<tr>
<td></td>
<td>2690–2732</td>
<td>3.93</td>
<td>1.76</td>
<td>41.6</td>
<td>9.6</td>
<td>6.3</td>
<td>15.3</td>
<td>27.2</td>
</tr>
<tr>
<td>Yidi 2</td>
<td>1728</td>
<td>0.52–5.96</td>
<td>2.18–2.30</td>
<td>5.1–55.7</td>
<td>25.57</td>
<td>2.7–87.9</td>
<td>3.69</td>
<td>27.6–61.3</td>
</tr>
<tr>
<td></td>
<td>3055</td>
<td>4.98–5.48</td>
<td>2.7</td>
<td>30.2–50.2</td>
<td>39.3</td>
<td>10.9–29.2</td>
<td>17.7</td>
<td>9.5–25.8</td>
</tr>
<tr>
<td>E’yangye 1</td>
<td>2652–2704</td>
<td>2.0–3.6</td>
<td>3.2–3.6</td>
<td>29–72.5</td>
<td>38.5</td>
<td>2.1–15.6</td>
<td>8.5</td>
<td>15.5</td>
</tr>
<tr>
<td>Barnett</td>
<td>1597</td>
<td>3.2</td>
<td>2.25</td>
<td>46.7</td>
<td>3.8</td>
<td>3.0</td>
<td>7.8</td>
<td>36.3</td>
</tr>
</tbody>
</table>

Note: The data of Changye 1 Well and Weiyi 201 Well are derived from references (Lin et al., 2014; Lin et al., 2015; Zhao et al., 2016). R, data of Huaye 1 Well is derived from references (Zhou et al., 2015). The data of Yidi 2 Well and Eyangye1 Well are derived from references (Chen et al., 2018 b). Barnett shale data are from reference (Chalmers et al., 2012) min-max (average)

Fig. 4 Mineral composition of Niutitang Fm.

4.2 Pore type

Based on the mineral composition and experienced geologic processes, shale pores are usually classified into three types: organic pores, inorganic pores, and microfractures (Chalmers et al., 2012; Loucks et al., 2012; Slatt and O’Brien, 2011; Xue et al., 2015). Although the Niutitang Fm shale is tight, various types of pores and microfractures are seen in SEM observations (Fig. 5). Detailed features of the pores and microfracture are
described below.

Fig. 5 SEM observation showing the development of pores and fractures. (a) organic pores, about 5–100 nm; (b) organic pores, about 5–100 nm; (c) pores between clay platelets, about 200–500 nm; (d) intercrystalline pores with pyrite framboïds, about 500 nm; (e) intercrystalline pores, about 100–300 nm; (f) microfracture, 20–30 μm in width (a, c, d, and f from Ciye 1 Well; b and e from Huaye 1 Well). (1) Organic pores are the most common type in the shale reservoir. Organic matters in the SEM image contain hundreds of small organic pores, which are approximately globular and bubble-like with elliptical cross sections. The diameters of organic pores range from 5 nm to 100 nm. The pore diameter near the center of the organic matter body is much larger than that on the edge (Curtis et al., 2012). In addition, large organic pores often contain many sub-scale pores.

(2) The intergranular pore has diameters between 200 nm and 500 nm. These pores formed during transformation from smectites or other unstable minerals to stable illites after burial (Klaver et al., 2012).

(3) Intercrystalline pores with pyrites framboïds are observed. These pores are about 500 nm in diameter. Intercrystalline pores also developed within quartz, feldspar, calcite, and other minerals, though they cannot be created easily.

(4) In addition, shale contains some microfractures, which are about 20–30 μm in width and can extend to more than 200 μm.

4.3 Pore structure

It can be seen from Fig. 6 that low-temperature liquid N₂ adsorption/desorption curves are of the anti-S type, which is similar to the typical multi-layer adsorption curve. According to the differences in shapes, adsorption/desorption curves of the Niutitang Fm are classified into three types (Liang et al., 2015); see Table 2. Considering the adsorption/desorption curves along with the SEM observation shows that shale pores are predominantly open; examples of these pores are nanoscale cylindrical pores (organic pores), parallel-plate pores (pores between clay layers), and conical plane pores (intercrystalline pores or intergranular pores) (Xue et al., 2015). V_{Bü} ranges from $2.144 \times 10^{-3}$ cm³/g to $20.07 \times 10^{-3}$ cm³/g, and the average value is $11.752 \times 10^{-3}$ cm³/g. The detail analysis results are presented in Table 2.

(1) Type A: Adsorption/desorption curves differ greatly when the value of P/P₀ is higher than 0.6 and almost coincide when the value is smaller than 0.4. The inflection point can be seen clearly when the value is between 0.4 and 0.6. The pore structure is mainly of the types of organic pores and pores between clay layers. The TOC content for the sample ranges from 2 wt% to 8 wt%, and very good pores are developed, with $V_{Bü}$ ranging from $2 \times 10^{-3}$ cm³/g to $20 \times 10^{-3}$ cm³/g.

(2) Type B: Adsorption/desorption curves stay separate. The pore structure mainly belongs to the types organic pores, pores between clay layers, intercrystalline pores, and intergranular pores. The TOC content is higher than 8 wt%, and pore development is the best, with $V_{Bü}$ larger than $20 \times 10^{-3}$ cm³/g.

(3) Type C: The inflection of curves is not obvious. Adsorption/desorption curves are nearly parallel with a small hysteresis loop. The pore structure mainly belongs to the type of intercrystalline pores and intergranular pores. The TOC content is less than 2 wt%, and pores are poorly developed, with $V_{Bü}$ less than $2 \times 10^{-3}$ cm³/g.
4.4 Adsorption characteristics

CH₄ isothermal adsorption experiments were conducted on 15 samples from Huaye 1 Well. The values of Vᵢ are between 1.63 cm³/g and 7.39 cm³/g, and the average value is 3.95 cm³/g. The Langmuir pressure (Pᵢ) ranges from 1.39 MPa to 8.35 MPa, with an average of 5.13 MPa. The correlation between Vᵢ and Pᵢ is negative; this behavior differs from the trend between Vᵢ and TOC as seen in the results of Zhang’s experiments (Zhang et al., 2014). It can be deduced that more CH₄ can be adsorbed at a low pressure in samples with high TOC contents. Excluding one abnormal value, the Vᵢ values of 14 samples are higher than 2.0 cm³/g, which means that Niutitang Fm has a strong adsorption capacity for CH₄, higher than that of Longmaxi Fm (1.16 cm³/g) (Tang et al., 2016).

5 Discussions

5.1 Influence factors on gas storage capacity

Shale gas belongs to the typical “in situ” accumulation model. It is mainly stored as free gas in natural fractures and intergranular pores or as adsorbed gas in kerogen and clay-particle surfaces (Curtis, 2002). Reservoirs of Niutitang Fm are characterized by low porosity and strong adsorption capacity. Vᵢ and Vₑ represent the reservoir capacity for free gas and adsorbed gas, respectively. Based on previous studies, the characteristics of shale storage capacity are significantly affected by the combination of sedimentary facies, material composition (such as TOC and inorganic minerals), and thermal maturity (Curtis et al., 2012; Qin et al., 2017).

5.1.1 Facies

Pores mainly rely on solid supporting frameworks. Effective source rocks only develop under deep muddy continental shelf microfacies, with abundant organic matters (TOC > 2 wt%), clay minerals, and pyrites, which constitute the basic materials of the shale pores. For example, the development of organic pores, intergranular pores between clay minerals, and intercrystalline pores with pyrite framboids not only results in more pore space, but also increases the adsorption capacity for CH₄. The data in Table 3 show that Vᵢ is higher than 10.0 × 10⁻³ cm³/g, and Vₑ is higher than 4.0 cm³/g in deep muddy continental shelf microfacies. However, the shale from shallow continental shelf facies is not the source rock. It has a low TOC content (<2 wt%), a low content of clay minerals, and a high content of quartz; these results are in contrast to the results of the source rock (Vᵢ < 3.0 × 10⁻³ cm³/g and Vₑ < 2.0 cm³/g).

5.1.2 Material composition

The composition and content of materials affect the mechanical properties of rocks, which further influence the development of pores and fractures. Based on the influence on reservoirs, materials of Niutitang Fm are predominantly classified as organic matter, brittle minerals (quartz, pyrite), plastic minerals (clay), and cement minerals (carbonate), as listed in Table 4.

The residual carbon after primary oil migration is the main factor that controls the development of organic pores, which play a critical role in shale reservoirs (Xue et al., 2016; Cardott et al., 2015; Slatt and O’Brien, 2011; Tian et al., 2015; Zhang et al., 2012). If a shale reservoir has 7 wt% of TOC, and 35% of it is converted into oil and gas, 4.9% pore space will be created (Jarvie et al., 2007). Clay-rich sediments have a unimodal pore

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Table 2 Characteristics of liquid N₂ adsorption/desorption of Niutitang Fm.

<table>
<thead>
<tr>
<th>Type</th>
<th>Pores structure</th>
<th>SEM observation</th>
<th>TOC/ wt%</th>
<th>BET/Surface Area/(m²·g⁻¹)</th>
<th>Vᵢ/N₁ (10⁻³ cm³·g⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>cylindrical pores with two sides open, narrow-parallel-plate pores with four sides open, narrow parallel-plate pores, conical plane pores with four sides open, conical plane pores with two sides open, conical plane pores with four sides open</td>
<td>organic pores, intergranular pores, pores between layers of clay minerals</td>
<td>2–8</td>
<td>4–15</td>
<td>6–20</td>
</tr>
<tr>
<td>B</td>
<td>conical plane pores with two sides open, conical tube pores with two sides open</td>
<td>organic pores, intergranular pores, intercrystalline pores; pores between layers of clay minerals</td>
<td>&gt;8</td>
<td>&gt;15</td>
<td>&gt;20</td>
</tr>
<tr>
<td>C</td>
<td>intergranular pores, intercrystalline pores</td>
<td>&lt;2</td>
<td>&lt;4</td>
<td>&lt;6</td>
<td></td>
</tr>
</tbody>
</table>
size distribution (<10 nm) and an average total porosity of 5.6%. Quartz-rich shale reservoirs exhibit few micropores or mesopores, and the average total porosity is 1% (Ross and Bustin, 2009). For Longmaxi Fm shale in the southern Sichuan Basin, open and connected organic pores offer about 78% (55% adsorbed gas and 23% free gas) of the original gas-in-place, while inorganic pores account for only 22% (mainly free gas) of the original gas-in-place (Yang et al., 2016). The correlations between material composition and reservoir storage capacity (V_L and V_{BJH}) are shown in Fig. 7 and Fig. 8.

(1) TOC

The higher the volume of organic matter or TOC, the greater is the number and diameter of the organic pores. Both V_{BJH} and V_L show positive linear correlations with TOC, as shown in Fig. 7a and Fig. 8a. V_{BJH} and V_L increase with TOC, until TOC reaches 8 wt%. When the TOC exceeds 8%, V_{BJH} continuously increases from 16 × 10^{-8} \text{cm}^3/g to 20 × 10^{-8} \text{cm}^3/g, while V_L only increases from 7.13 \text{cm}^3/g to 7.39 \text{cm}^3/g (Table 4). This is because as the number of macropores increases with TOC, they contribute more to the increase in V_{BJH} (Fig. 9).

When TOC exceeds 8 wt%, macro pores contribute to more than 50% of V_{BJH}. However, the increase in the percentage of macro pores limits the specific surface area, and the adsorption capacity for CH_4 does not increase accordingly.

Table 3 Reservoir characteristics of Niutitang Fm in Well Huayu 1.

<table>
<thead>
<tr>
<th>Sedimentary facies</th>
<th>Pores type</th>
<th>TOC/ wt%</th>
<th>Muddy/ wt%</th>
<th>Siliceous/ wt%</th>
<th>Calcareous/ wt%</th>
<th>Pyrite/ wt%</th>
<th>BET surface area/(m^2/g)</th>
<th>V_{BJH}/(10^7 cm^3/g)</th>
<th>V_L/(cm^3/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shallow continental shelf</td>
<td>intergranular pores</td>
<td>&lt;1</td>
<td>&lt;30</td>
<td>&gt;40</td>
<td>&lt;20</td>
<td>&lt;3</td>
<td>2</td>
<td>3</td>
<td>&lt;2</td>
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<tr>
<td></td>
<td>intergranular pores,</td>
<td>1−2</td>
<td>&lt;25</td>
<td>&lt;30</td>
<td>&gt;40</td>
<td>4</td>
<td>2−7</td>
<td>3−7</td>
<td>Deep shelf</td>
</tr>
<tr>
<td></td>
<td>intercrystalline pores,</td>
<td></td>
<td></td>
<td></td>
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<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>intercrystalline pores,</td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
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<td>dissolved pores</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>organic pores</td>
<td>1−2</td>
<td>25−30</td>
<td>30−45</td>
<td>20−40</td>
<td>4−8</td>
<td>7−10</td>
<td>7−10</td>
<td></td>
</tr>
<tr>
<td></td>
<td>intergranular pores,</td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td>intercrystalline pores,</td>
<td></td>
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</tr>
<tr>
<td></td>
<td>intercrystalline pores,</td>
<td></td>
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<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>organic pores</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>intergranular pores,</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>intercrystalline pores</td>
<td>&gt;2</td>
<td>&gt;30</td>
<td>&gt;45</td>
<td>0−20</td>
<td>8−17</td>
<td>&gt;10</td>
<td>&gt;10</td>
<td></td>
</tr>
</tbody>
</table>

Table 4 Niutitang Fm reservoir characteristics of Huayu 1 well.

<table>
<thead>
<tr>
<th>Depth (m)</th>
<th>TOC (wt%)</th>
<th>Clay Minerals/ wt%</th>
<th>Quartz/ wt%</th>
<th>Pyrite/ wt%</th>
<th>Carbonate/ wt%</th>
<th>V_L (cm^3/g)</th>
<th>P_t (MPa)</th>
<th>BET surface area/(m^2/g)</th>
<th>V_{BJH}/(10^7 cm^3/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2457</td>
<td>1.04</td>
<td>39.9</td>
<td>44</td>
<td>5.3</td>
<td>0</td>
<td>2.08</td>
<td>4.41</td>
<td>0.617</td>
<td>2.173</td>
</tr>
<tr>
<td>2463</td>
<td>1.52</td>
<td>40.8</td>
<td>42.9</td>
<td>5.4</td>
<td>0</td>
<td>4.75</td>
<td>7.21</td>
<td>7.488</td>
<td>9.385</td>
</tr>
<tr>
<td>2500</td>
<td>2.67</td>
<td>29.6</td>
<td>43.8</td>
<td>4.4</td>
<td>10</td>
<td>5.49</td>
<td>5.79</td>
<td>10.56</td>
<td>9.894</td>
</tr>
<tr>
<td>2504</td>
<td>3</td>
<td>23.5</td>
<td>42.5</td>
<td>8.5</td>
<td>12.3</td>
<td>2.99</td>
<td>2.8</td>
<td>11.302</td>
<td>10.44</td>
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<td>2548</td>
<td>13.3</td>
<td>18.9</td>
<td>39.9</td>
<td>17</td>
<td>7</td>
<td>7.39</td>
<td>1.8</td>
<td>27.543</td>
<td>20.07</td>
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<tr>
<td>2555</td>
<td>8.81</td>
<td>27.1</td>
<td>41.9</td>
<td>8.8</td>
<td>5.5</td>
<td>7.13</td>
<td>2.55</td>
<td>24.369</td>
<td>17.62</td>
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<tr>
<td>2560</td>
<td>7.77</td>
<td>16.9</td>
<td>49.8</td>
<td>8.6</td>
<td>6.1</td>
<td>4.27</td>
<td>1.39</td>
<td>19.641</td>
<td>16.73</td>
</tr>
<tr>
<td>2563</td>
<td>6.78</td>
<td>24.8</td>
<td>42.2</td>
<td>9.9</td>
<td>4.6</td>
<td>4.43</td>
<td>1.62</td>
<td>21.258</td>
<td>17.31</td>
</tr>
<tr>
<td>2596</td>
<td>1.24</td>
<td>8.9</td>
<td>42.6</td>
<td>2</td>
<td>23.3</td>
<td>2.64</td>
<td>6.63</td>
<td>1.981</td>
<td>2.144</td>
</tr>
</tbody>
</table>

(2) Brittle minerals

Under deep muddy continental shelf microfacies, the quartz content is 45–60 wt%. However, V_{BJH} is not correlated with quartz, as shown in Fig. 7b and Fig. 8b. This is mainly because during diagenetic processes, the nanopores between rigid quartz particles are further compacted and filled with plastic clay minerals, organic matter, and cement, resulting in serious loss of pore volume (Wang et al., 2017) as proved by the SEM observation. As the average size of quartz particles is about 30 μm, the adsorption capacity for CH_4 is small; hence, there is no apparent correlation between V_L and quartz. However, the brittleness of shale increases with increasing quartz content. Pores and microfractures can be easily generated when stress is applied, thereby improving the pore space and permeability of the shale.

Table 4 shows that pyrites are abundant, with a maximum content of 17 wt% in Huayu 1 Well. The SEM results show that pyrites particles contain a large number of intercrystalline pores. V_{BJH} and V_L are positively correlated with pyrites (Fig. 7c and Fig. 8c). However, upon eliminating the influence of TOC, we find that V_{BJH} and V_L are negatively correlated with pyrites (Fig. 7d and Fig. 8d), implying that the increase in V_{BJH} and V_L is mainly caused by TOC rather than pyrite.

(3) Plastic minerals

The clay minerals (predominantly illites) are large, forming nano–micron scale intergranular pores and interlayer pores. V_{BJH} and V_L show negative linear correlations with the illite content. However, when the influence of TOC is eliminated, V_{BJH} and V_L show positive correlations with illites (Fig. 7e and Fig. 8e). The

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corresponding correlation coefficient is lower than that for TOC. This phenomenon indicates that CH₄ mainly occurs in organic pores rather than in pores between clay minerals. When the content of clay minerals increases, the plasticity of the rock increases. Unstable clay minerals can block throats easily during the hydraulic stimulation.

(4) Cement minerals
Carbonate minerals have a strong ability of chemical cementation and inhibit the development of nano–micron pores and microfractures (Huang and Shen, 2015). Although dissolution can lead to the formation of dissolved pores, the low content of carbonate minerals will make it hard to create many pores. Besides, carbonate minerals have poor adsorption capacity for CH₄. Therefore, \( V_{\text{BJH}} \) and \( V_L \) show negative linear correlations with the carbonate mineral content (Fig. 7f and Fig. 8f).

![Fig. 7 Analysis of the influence on BJH volume of TOC and inorganic minerals in deep muddy shelf microfacies.](image)

![Fig. 8 Analysis of the influence on Langmuir volume of TOC and inorganic minerals in deep muddy shelf microfacies.](image)

5.1.3 Thermal maturity
The influence of the thermal maturity of organic matter on the reservoir pores is complex. When \( R_o \) is smaller than 0.9%, organic pores are rarely developed (Hu et al., 2015). The porosity rises with increasing thermal maturity, until \( R_o \) reaches 3.5% (Chen and Xiao, 2014; Cheng and Xiao, 2013). High thermal maturity leads to more pores and seepage space and improves the adsorption capacity for CH₄. When \( R_o \) > 3.5%, micropore development exhibits a downward trend, while mesopores continue to increase slowly (Ambrose et al., 2010; Cheng and Xiao, 2013; Zhang et al., 2012). High or over maturity may reduce the adsorption capacity, while the pore volume does not decrease significantly.

In this study, when Niutitang Fm enters the late diagenetic stage, the organic matter is in the high maturity or overmature stage (\( R_o = 2\%–3\% \)). During the thermal evolution, the organic matter underwent degradation, hydrocarbon generation, and expulsion, and then, nanoscale pores formed (Hu et al., 2015). Because the values of \( R_o \) and TOC of Niutitang Fm are extremely high, the mesopores and macropores of Niutitang Fm are well...
developed, resulting in a significant increase in $V_{BH}$ and $V_L$. Moreover, better development of microfractures also improved the storage ability for CH$_4$.

$$y=2.5671x+30.648$$

$R^2=0.7307$

Fig. 9 Correlation between contribution percentages to BJH volume from macropores and TOC.

5.2 Shale gas occurrence states and sweet spots identification

5.2.1 Gas-bearing characteristic

Gas content and gas-bearing characteristics are the key indexes to evaluate whether gas-bearing shale has industrial development value (Lin et al., 2014). In general, free gas is primarily responsible for gas-in-place of shale gas, whereas the adsorbed gas partially determines the longevity of shale-gas-producing wells. For example, the gas content of Longmaxi Fm in Jiaoye 1 Well in Sichuan Basin ranges from 0.44 m$^3$/t to 5.19 m$^3$/t, with an average of 1.97 m$^3$/t. The main percentages of shale gas are desorbed gas and lost gas, while the percentage of residual gas is low. The gas content of Niutitang Fm in southern Sichuan is less than that of Longmaxi Fm; this is probably because of the declining of matrix pores from the overmature stage and the poor preservation condition during the diagenetic processes (Wang, 2015 b).

Preservation factors, such as roof and floor conditions, over-maturation of organic matter, structural style, fracture condition, and detachment layer affect the gas-bearing property of the Niutitang Fm in South China; as a result the gas-bearing property of this formation is poorer than that of the Longmaxi Fm. The core of Niutitang Fm shale is generally characterized by low content of CH$_4$ and high percentage of N$_2$. The gas content in Ciye 1 Well is the highest in the study area. The total gas content (predominantly CH$_4$) ranges from 0.3282 m$^3$/t to 0.9452 m$^3$/t, with an average of 0.68 m$^3$/t. The percentages of residual gas in Ciye 1 Well are 44.8–83.5% (69.7% on average), while the percentages of desorbed gas only account for 8.3–22.3% (14.9% on average). It is different from the Longmaxi Fm in Yongye 2 Well in that the total content of CH$_4$ ranges from 1.0–3.5 m$^3$/t; desorbed gas accounts for 33.8–79.0% (55.4% on average); and residual gas accounts for 4.0–40.5% (20.4% on average). This clear difference is shown in Fig. 10, and it will be further revealed from a deeper analysis of reservoir characteristics.

5.2.2 Influences of reservoir characteristics on gas occurrence states

According to the Langmuir theory, adsorbed CH$_4$ only occupies about 1 molecular thickness of the pore...
volume (the typical adsorbed layer is about 0.7-nm thick on average). For a pore 100 nm in diameter, the adsorbed CH₄ only occupies about 4% of the whole pore volume. The proportion occupied by adsorbed CH₄ to pore volume increases gradually with the decrease in pore diameters (Chen et al., 2017; Ambrose et al., 2010). SEM observations show that the pores of Niutitang Fm are small (diameters generally less than 100 nm). It can be deduced that the adsorbed CH₄ in the original reservoir accounts for a significant proportion of the pore volume.

Assuming that a reservoir is buried at a depth of 2500 m, the in situ reservoir pressure is about 25 MPa, and the in situ temperature is about 368.15 K (considering the ground temperature as 293.15 K and the ground pressure as 0.101 MPa and the geothermal gradient as 3°C/100 m). Based on the CH₄ deviation coefficient graph, the CH₄ deviation coefficient is about 0.96. Referring to equation (1), the original natural gas volume coefficient can be calculated as ~0.00487 (Zhang and Zhang, 2004).

\[
B_{gi} = P_{sc} Z_i T/(P_i T_{sc}) \tag{1}
\]

where \(B_{gi}\) is the in situ gas volume coefficient; \(P_{sc}\) is the ground standard pressure (0.101 MPa); \(Z_i\) is the in situ CH₄ deviation coefficient; \(T\) is the reservoir temperature (K); \(P_i\) is the in situ reservoir pressure [MPa]; and \(T_{sc}\) is the ground standard temperature (293.15 K).

Under deep muddy continental shelf microfacies, \(V_{BH}^i\) of Niutitang Fm is about \(11 \times 10^{-3}\) cm³/g on average, and the storage capacity for free gas is about 2.258 cm³/g after conversion. The storage capacity for adsorbed gas (\(V_i\)) is 4.57 cm³/g on average, and it accounts for 66.9% of the total reservoir storage capacity. In contrast, \(V_{BH}^i\) of Longmaxi Fm is about 0.016 cm³/g on average, and the storage capacity for free gas is about 3.285 cm³/g after conversion. The shale storage capacity for adsorbed gas (\(V_i\)) is 1.16 cm³/g on average, accounting for only 26.1% of the total reservoir capacity.

It can be inferred that in the in situ high-pressure reservoir, the shale gas of Niutitang Fm is mainly stored as adsorbed gas. When the core is pulled out to the ground, a small percentage of the adsorbed gas will be converted into free gas or lost gas because of the decrease in temperature and pressure. As the reservoir is tight, the storage capacity for free gas is poor, though the storage capacity for adsorbed gas is good. Most of the adsorbed gas is still stored in shale reservoir even after desorption experiment. Therefore, the percentage of residual gas in the crushed core is high (69.7% on average in Ciye 1 Well), which is in agreement with the result that the storage capacity for adsorbed gas accounts for 66.9% of the total reservoir storage capacity on average. However, in the case of Longmaxi Fm, similar agreement is seen with low residual gas percentages (Zhang et al., 2015 c).

### 5.2.3 Targeting Sweet spots Layer

It is believed that the tight reservoir and strong adsorption capacity result in low percentages of free gas and thus affect the gas development. The experimental results of residual gas proportion in Ciye 1 Well are in accordance with the theoretically calculated results of the adsorbed gas proportion in Huaye 1 Well, as shown in Fig. 11 and Table 5. The adsorbed gas percentage in Huaye 1 Well varies with TOC, and it will be the lowest when TOC is between 4 wt% and 8 wt%.

![Fig. 11 Comparisons between residual gas in Ciye 1 Well and adsorbed gas in Huaye 1 Well.](image)

<table>
<thead>
<tr>
<th>TOC%</th>
<th>1 - 2</th>
<th>2 - 4</th>
<th>6 - 10</th>
<th>&gt;10</th>
<th>average</th>
</tr>
</thead>
<tbody>
<tr>
<td>(V_{res}(\text{cm}^3/\text{g}))</td>
<td>0.002159</td>
<td>0.001067</td>
<td>0.01722</td>
<td>0.0207</td>
<td>0.011</td>
</tr>
<tr>
<td>Storage capacity for free gas (\text{cm}^3/\text{g}) (16.3%)</td>
<td>0.443326</td>
<td>2.08768</td>
<td>3.55934</td>
<td>4.250513</td>
<td>2.258727</td>
</tr>
<tr>
<td>Storage capacity for adsorbed gas (\text{cm}^3/\text{g}) (83.7%)</td>
<td>2.283</td>
<td>4.24</td>
<td>5.27</td>
<td>7.39</td>
<td>4.57</td>
</tr>
</tbody>
</table>

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In previous studies, sweet spots were not accurately identified from the whole sequences, resulting in a low shale gas flow even after successful hydraulic stimulation. Considering the high TOC and $R_o$ of Niutitang Fm, the potential of hydrocarbon generation is not a crucial problem. With a good preservation condition, more attention should be paid to the sweet spot intervals, which are critical to commercial development.

As show in Fig. 2, two sweet spot layers were identified; in these layers, the TOC ranges from 4 wt% to 8 wt%. Suitable thickness, high quartz content, and low content of clay minerals and carbonate minerals are in favor of hydraulic stimulation and development. Besides, strong storage capacity of free gas will be suitable for high productivity (Table 6).

**Conclusions**

A thorough study of the Niutitang Fm reservoir characteristics in the northwestern Hunan Province was conducted. This paper describes the characteristics of gas-bearing states and sweet spot identification. The results will provide knowledge for extensive shale gas exploration and development in South China in the future. Some findings are listed below:

1. Under deep muddy continental shelf microfacies, one or two layers of high-quality source rocks in Niutitang Fm are mainly composed of carbonaceous shale and siliceous shale. The mineral compositions are as follows: high quartz content, low content of clay minerals (predominantly illites), and the low content of carbonate minerals.

2. Though the reservoir of Niutitang Fm is tight, nanoscale organic pores, intergranular pores between clay minerals, intercrystalline pores with pyrites framboinds, and microscale fractures can be observed by SEM. These pores belong to the types open and interconnected mesopores and macropores. The shale reservoir has strong adsorption capacity for CH₄. $V_L$ ranges 1.63–7.39 cm³/g, and the average is 3.95 cm³/g. However, the shale reservoir storage capacity for free gas is poor. $V_{dR}$ ranges from $2.144 \times 10^{-3}$ cm³/g to $20.07 \times 10^{-3}$ cm³/g, and $11.752 \times 10^{-3}$ cm³/g on average.

3. The characteristic of sweet spots layers of Niutitang Fm.

<table>
<thead>
<tr>
<th>Facies</th>
<th>Lithology</th>
<th>Thickness/m</th>
<th>TOC/wt%</th>
<th>Quartz/wt%</th>
<th>Clay/wt%</th>
<th>Carbonate</th>
<th>$V_{dR}$/(cm³·g⁻¹)</th>
<th>$V_L$/(cm³·g⁻¹)</th>
<th>Percentage of free gas/%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deep muddy continental</td>
<td>carbonaceous</td>
<td>20–40</td>
<td>4–8</td>
<td>&gt;40</td>
<td>30–40</td>
<td>&lt;20</td>
<td>$(10 - 17) \times 10^{-3}$</td>
<td>4–6</td>
<td>&gt;50</td>
</tr>
<tr>
<td>shelf</td>
<td>siliceous</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>shelf</td>
<td>siliceous</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

4. In Ciyue 1 Well, the desorbed gas percentage of Niutitang Fm is low (14.9%), while the residual gas percentage is high (67.9%). This is probably because of the weak storage capacity for free gas (26.1%) and the strong storage capacity for adsorbed gas (66.9%). Sweet spot layers were identified; these layers are characterized by strong storage capacity for free gas and TOC ranging from 4 wt% to 8 wt%.

**Highlight**

- TOC is the most important factor for shale gas storage capacity.
- Plastic minerals have a positive influence on shale storage capacity for gas.
- Cement minerals have a negative influence on shale storage capacity for gas.
- Shale gas occurrence states are affected by the shale reservoir characteristics.
- Two layers of sweet spots are identified.

**Acknowledgements**

This work is granted by the Nation Natural Science Foundation(41603046) and the Natural Science Foundation of Hunan Province (2017JJ1034).

**References:**


| Total storage capacity/(cm³·g⁻¹) | 6.32768 | 8.805934 | 11.64051 | 6.828727 |

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中文摘要
湘西北下寒武统牛蹄塘组储层特征及甜点段识别
页岩储层特征控制了页岩气的聚集和生产。研究页岩储层特征，分析储层对页岩气赋存状态的影响，识别甜点段是深水陆棚南缘下寒武统牛蹄塘组页岩气勘探的基础。本研究开展了湖南省西北地区几口勘探井的岩心和岩屑观察，并进行了多种实验分析，如 XRD、SEM、低温液氮吸附实验及甲烷等温吸附实验等。结果表明，在深水陆棚环境中沉积了一层或两层高质量烃源岩（TOC> 2 wt%）。烃源岩主要含腐泥页岩和硅质页岩，并富含石英（> 40 wt%）。但粘土矿物（<30 wt%，主要是伊利石）和碳酸盐矿物（<20 wt%）含量相对较低。扫描电镜观察和液氮吸附/解吸实验表明，页岩储层致密，但纳米级孔隙和微米级裂缝发育良好。页岩的 BJH 体积（V_{BJH}）为 2.144×10^{3} 至 20.07×10^{3} cm^{3}/g，平均为 11.75×10^{3} cm^{3}/g。孔隙主要由开放型、互相联通的中孔（孔径 2-50nm）或大孔（孔径>50nm）组成。页岩储层对 CH_{4} 具有很强的吸附能力，Langmuir 体积（V_{L}）在从 1.63-7.39 cm^{3}/g 之间，平均为 3.95 cm^{3}/g。页岩储层的特征受以下几个因素控制：（1）深水泥质陆棚是页岩储层发育最有利的环境，控制着孔隙发育的物质基础。受页岩储层的储集能力随着 TOC 含量和塑性矿物的增加而增强，但随着胶结矿物的增加而减少。（2）有机质成熟度及成熟程度导致有机孔隙和微裂缝的发育，提高了储层的储集能力。牛蹄塘组残余气比例高是由于储层赋存能力强所导致，牛蹄塘组识别出两层甜点段，具有对游离气体的储集能力强。
关键词
湘西北、牛蹄塘组、储层特征、控制因素、甜点识别