

Characteristics of microscopic pore heterogeneity and development model of Wufeng–Longmaxi Shales in the Pengshui area of south-east Chongqing

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Abstract Normal-pressure shale gas reservoirs are widely distributed in south-eastern Chongqing and show good potential for resource exploration. This paper reports the organic matter (OM), physical, and pore characteristics, mineral composition, and gas content of representative shale samples from the Upper Ordovician Wufeng Formation and Member 1 of the Lower Silurian Longmaxi Formation (Long 1 Member). Microscopic pores within different shale layers of the Long 1 Member were classified, quantitatively evaluated, and their development mechanisms were systematically studied. We found that OM characteristics, mineral composition, and pore type were the main factors affecting the enrichment and preservation of shale gas. The characteristics of the Long 1 Member are mainly controlled by changes in the sedimentary environment. There are evident differences in total organic carbon content and mineral composition vertically, leading to a variable distribution of pores across different layers. Organic matter abundance controls the degree of OM pore development, while clay minerals abundance control the development of clay mineral-related pores. Total organic carbon content generally controls the porosity of the Long 1 Member, but clay minerals also play a role in OM-poor layers. Pore connectivity and permeability are influenced by the development of pores associated with brittle minerals. We propose a microscopic pore development model for the different layers. Combining geochemical data and this pore development model, layers 1–4 are considered to be excellent shale gas preservation and enrichment reservoirs. Poor preservation conditions in layers 5–7 result in high levels of shale gas escape. Layers 8–9 possess a better sealing condition compared with layers 5–7 and are conducive to the

enrichment and preservation of shale gas, and can thus be used as future potential target strata. This research provides a theoretical basis for exploring and evaluating shale gas potential in the studied region or other complex normal-pressure shale blocks.

Keywords shale gas, pore characteristics, Longmaxi Formation, reservoir model

1 Introduction

In recent years, normal-pressure shale gas has become a research hotspot in China owing to its complex formation mechanism and tectonic features (Fang and He, 2016; Ma et al., 2018; Guo, 2021). During the “Thirteenth Five-Year Plan” period (2016–2020), China has intensified exploration and development in marginal areas of the Sichuan Basin and outside this basin. Normal-pressure shale gas reservoirs, such as Pingqiao South, Dongsheng, Baima, Wulong, Daozhen, and Sangtuoping, have recently been discovered (He, 2021a; He et al., 2021b; Cai et al., 2021). Moreover, industrial gas flows have been obtained in normal-pressure shale gas reservoirs in Nanchuan, Wulong, and Pengshui. The first normal-pressure shale gas field in China has been commercially developed in the Dongsheng area and southern part of the Pingqiao area, with proven reserves reaching $1446.58 \times 10^8 \text{ m}^3$ (Guo et al., 2020; Cai et al., 2021).

Previous studies have shown that preservation conditions are the key factor affecting shale gas enrichment (Wei et al., 2017; Ma et al., 2018). The preservation of shales at the macro-scale geological level is mainly influenced by tectonic movements, duration of strata uplift, depth of burial, shale distribution area, and top and bottom conditions, among other factors (Mei et al., 2010;

Wei et al., 2017; Guo et al., 2020; Tang et al., 2021; Zeng et al., 2022). The key to the preservation of shale gas at the microscopic level mainly relates to the oil and gas self-sealing ability of microscopic pores within the shale formation system (Guo et al., 2022). Microscopic pores comprise the main reservoir space for shale gas, and the heterogeneous nature of such pores in Wufeng–Longmaxi Formation shales has been reported by previous authors (Xiao et al., 2019; Zhao, 2020; Li et al., 2022; Yang et al., 2022). Studies have shown that different types of pores contribute in different ways to shale gas accumulation. Differences in pore type affect the gas storage state, adsorption performance, gas enrichment mechanism, and connectivity of storage space. Cao et al. (2022, 2023) showed that the dominant pore types in shales of the Wufeng–Longmaxi Formation are organic matter (OM) pores in solid bitumen and sedimentary OM. Different tectonic conditions, diagenetic evolution, and evolution of fracture systems are the main reasons behind the different mechanisms of shale gas preservation (Chen et al., 2015; Yang et al., 2016; Zhang et al., 2018; He et al., 2019), while difference in pore type is one of the main reasons for differences in the preservation of normal-pressure shale gas in different layers and regions within complex tectonic zones.

A previous study showed that layers 1–3 in shales of the Wufeng–Longmaxi Formation can be characterized by high total organic carbon (TOC) content and high gas content, suggesting a sweet-spot area (Ma et al., 2018). Recently, wells deployed in the Nanchuan area targeting layers 8–9 of this formation have also achieved good production. However, there are few relevant studies on the quantitative evaluation of the vertical heterogeneity of microscopic pores in normal-pressure shale gas blocks and the factors influencing this heterogeneity.

Therefore, our aim here was to identify the pore development characteristics of different layers within Member 1 of the Lower Silurian Longmaxi Formation (Long 1 Member) in the normal-pressure shale gas block of south-eastern Chongqing, along with the mechanism of shale gas enrichment and preservation. The Long 1 Member of Well PY-A in the Sangtuoping syncline, Pengshui area, was selected as the research object. Experimental results, including OM characteristics, shale physical characteristics, shale mineral composition, reservoir architecture characteristics, and gas content, were obtained, and a quantitative study of the vertical heterogeneity of the reservoir was conducted. The main types of OM pores and inorganic shale pores in different layers, along with their changing patterns, were identified, and the effects of different types of organic and inorganic pores on shale gas enrichment and preservation were analyzed. This study provides a theoretical basis for resource exploration and development of shale gas in the Wufeng–Longmaxi Formation and other normal-pressure regions.

2 Regional geological background

The Pengshui area is located on the eastern fringe of the Sichuan Basin, in the Xiang–Exi fold belt (Shu, 2014; Hu, 2017). The tectonic evolution of the region was mainly controlled by the Yanshanian movement and Himalayan orogeny. The region was affected by tectonic compression from the south-eastern part of the Xuefeng Mountain, resulting in strata uplift and erosion, forming a trough-like tectonics with north–south-trending residual alternating anticlines and synclines. The study area of the Sangtuoping syncline is located in the transition zone of the rough-like tectonics zone east of the Pengshui–Jiansi fault. The two limbs of the syncline comprise Cambrian–Silurian strata, and this is a relict syncline (Fig. 1).

3 Sample collection and experimental analysis

In the Jiangnan oilfield, the shales of the Upper Ordovician Wufeng Formation and Long 1 Member are divided into nine layers. Well PY-A was drilled in the Pengshui area. The cumulative thickness of shales in the Long 1 Member in this area is approximately 112.5 m. Layers 1–5 contain approximately 35.5 m of black shale, and layers 6–9 are gray-black shale. The shale thickness of layers 5–9 reaches 77 m. In this study, 60 shale samples were collected from the Long 1 Member. Only 1 m of layer 2 of the Longmaxi Formation was found in Well PY-A, and samples could not be retrieved; therefore, this layer will not be discussed further. Representative samples were selected for organic carbon content analysis, whole-rock X-ray diffraction, nitrogen adsorption–desorption, shale physical properties determination, scanning electron microscopy (SEM), and micro-scale computerized tomography (micro-CT) scanning.

Total organic carbon content was measured using a CS-230 carbon and sulfur analyzer, standardized by the national standard GB/T 19145-2003. Whole rock mineral composition was determined using an Ultima IV X-ray diffractometer (Rigaku, Japan), standardized by SY/T 5163-2018. Shale reservoir characteristics were quantified using an ASAP2020 low-temperature gas adsorption instrument for pore analysis. The ASAP2020 can perform specific surface area analysis down to 0.0001 m²/g and pore-size analysis from 0.35 to 50000 nm, with a resolution of 0.02 nm for micropore sections and minimum detection of 0.0001 mL/g for pore volume. Shale samples were characterized using SEM after argon ion polishing. Shale porosity and permeability were measured using a Poro PDP-200 overburden porosity permeability tester, standardized by the national standard GB/T 34533-2017. Micro-CT scanning was performed using the German Universal Nano-X-ray Digital Core

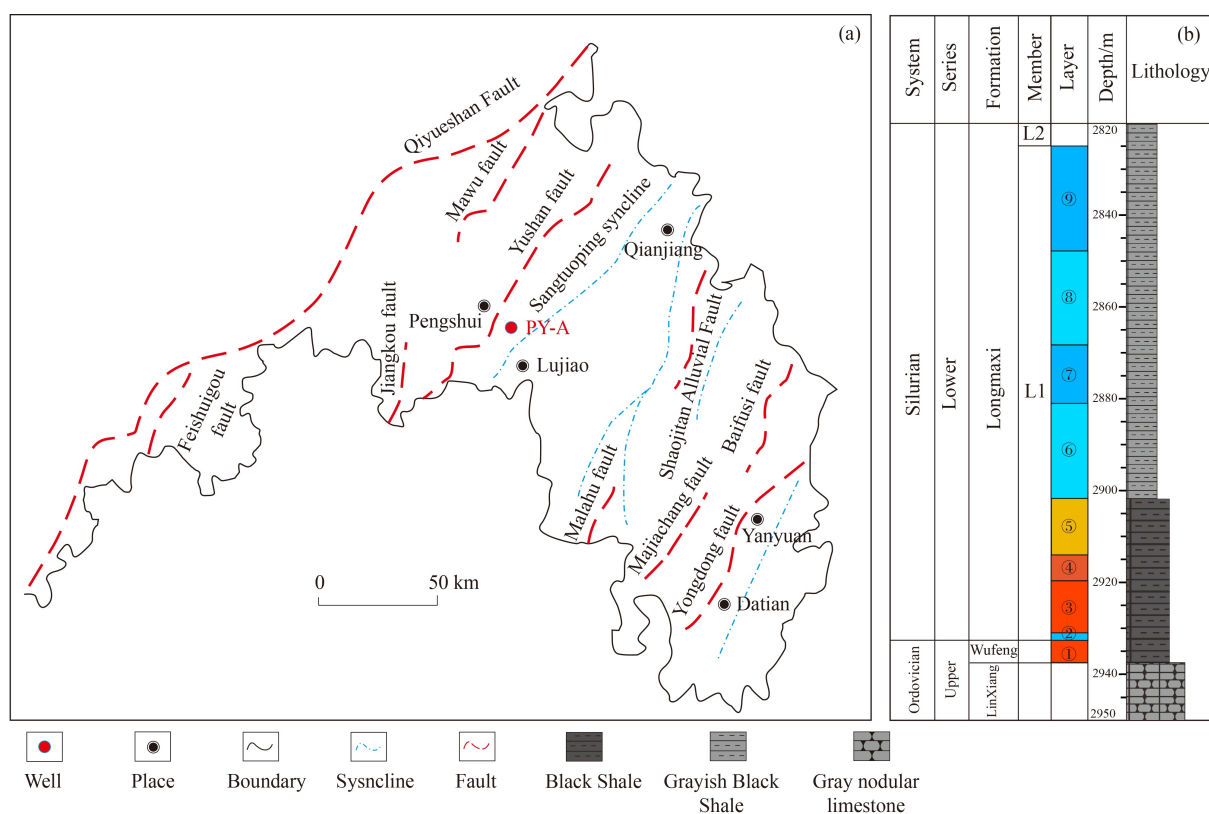


Fig. 1 Structural diagram of the study area (a) and histogram of the Wufeng–Longmaxi Formation (b).

Analysis System, according to the national standard GB/T 29034-2012.

4 Geological characteristics of the Wufeng–Longmaxi Formation shale gas

4.1 Organic matter characteristics

Organic matter abundance is the material basis for shale gas generation. A higher TOC content can provide a higher hydrocarbon generation capacity (Huang et al., 2020); meanwhile, OM can also provide a relatively large specific surface area for gas adsorption, offering important storage space for shale gas. Previous studies have shown that the vertical heterogeneity of reservoirs in the Wufeng–Longmaxi Formation in southern China is largely controlled by the abundance of OM (Jiang et al., 2021; Qiu et al., 2020). The organic carbon content of the Long 1 Member of Well PY-A varied in the vertical direction, showing a strong heterogeneity (Fig. 2). Layers 1–4 comprised organic-rich shales with TOC contents ranging from 0.19% to 4.44% (avg. 3.15%). Black shales occurred in layer 5 and gray-black shales occurred in layers 6–7, having TOC contents ranging between 0.57% and 1.72% (avg. 0.99%). Layers 8–9 mainly comprised gray-black shales, with TOC contents ranging between 0.69% and 1.97% (avg. 1.34%); these showed a three-

stage TOC distribution trend of highest at the bottom, lowest in the middle, and slightly higher at the top.

4.2 Mineral composition characteristics

The mineral composition of the Long 1 Member shale was dominated by quartz and clay minerals (Fig. 2), followed by feldspar and carbonate minerals, with a small amount of pyrite. The base of layer 1 had a high pyrite content, indicating that the local environment was predominantly reductive during this depositional period. The proportion of quartz ranged from 4.3% to 64.0% (avg. 40.26%), showing a trend of significantly increasing quartz content through layer 1, and then a gradually decreasing trend from layers 3–9. The clay content of the shale ranged from 21.7% to 88.8% (avg. 38.8%). The TOC content was highest in layer 1 and gradually decreased upwards, while the clay content began to increase in layers 8–9. A relatively high clay content in layer 1 can be attributed to argillaceous sediment dominating the early sedimentation period, along with a lack of bioturbation. The high levels of clay minerals in the upper layers (8–9) have been attributed to the mixing of terrigenous debris and formation of secondary clay minerals via diagenesis (Zhao et al., 2016). The carbonate content of layers 3–7 is higher than in other layers, ranging from 3.9% to 17.4% (avg. 8%). Wang and Carr (2012) studied the Marcellus shale in the Appalachian

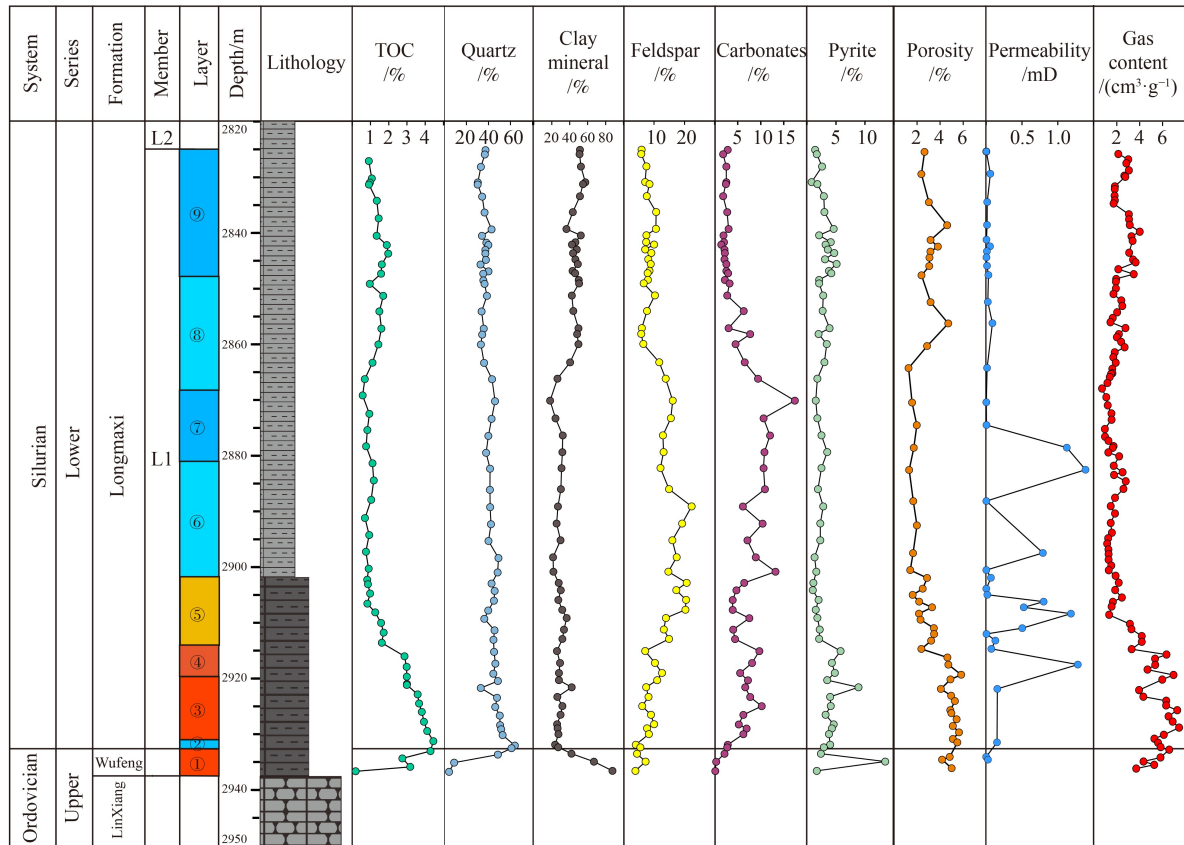


Fig. 2 Longitudinal distribution characteristics of geochemical parameters of Wufeng–Longmaxi Formation in Well PY–A.

Basin and suggested that rocks with high quartz and carbonate contents are more likely to form suitable oil and gas reservoirs under a given thermal evolution pathway. Meanwhile, carbonate minerals also produce dissolution pores and microfractures, which enhance percolation capacity in the shale and facilitate shale gas migration (Liang et al., 2021).

4.3 Physical property characteristics of the reservoir

Porosity and permeability are important physical indicators affecting shale gas reservoirs. Our experimental results showed that the porosity of the Long 1 Member ranged from 1.27% to 5.65% (avg. 3.43%) (Fig. 2), while permeability ranged from 0.0002 to 1.28 mD, (avg. 0.2443 mD), indicative of low porosity and low permeability reservoirs. Layers 1–4 had the highest porosity (avg. 4.78%), while layers 5–7 had the lowest (avg. 2.25%); the porosity of layers 8–9 (avg. 3.1%) was slightly higher than that of layers 5–7. An opposing trend was found for permeability, with the highest permeability occurring in layers 5–7 and lower permeability in layers 1–4 and 8–9.

4.4 Gas-bearing characteristics

The gas content of marine shale shows a positive

correlation with OM abundance (Wen et al., 2015; Zhang et al., 2015); a higher TOC content normally indicates a higher gas content. The TOC content of layers 1–4 was highest, and was accompanied by the highest gas contents, ranging from 3.20 to 7.35 cm³/g (avg. 5.55 cm³/g) (Fig. 2). The average gas contents of layers 5–7 and 8–9 were 1.74 and 2.22 cm³/g, respectively, indicating that the upper part of the Long 1 Member shows slightly better gas potential than the middle part.

5 Vertical heterogeneity of microscopic pores and its influencing factors

5.1 Microscopic pore types

The pore characteristics of shale are essential indicators for evaluating the potential of shale gas resources. According to the International Union of Pure and Applied Chemistry (IUPAC) classification, shale pores can be divided into three categories based on pore size: micropore (< 2 nm), mesopore (2–50 nm), and macropore (> 50 nm). Our experimental results showed that the studied pores were mainly micro–mesopores < 50 nm in diameter (Figs. 3(b), 3(d), 3(f)).

Relative pressure changes in shale during low-temperature nitrogen adsorption and desorption can form

a “desorption loop”, and the type and size of microscopic pores can influence the type of desorption loop. Our experimental samples all showed desorption loops. The adsorption capacity of layers 1–4 was highest owing to the highest TOC content, giving a H_2 -type desorption loop morphology, with ink-bottle-like pore morphology (Fig. 3(a)). The TOC content of layers 5–7 was significantly lower than that of layers 1–4, accompanied by lower adsorption of nitrogen. The size of the hysteresis loop gradually decreased, and the desorption loop type transitioned from H_2 type to H_3 type (Fig. 3(c)),

indicating a gradual transitioning of pore morphology from ink-bottle-like to slit-like. Layers 8–9 had higher clay and OM contents compared with those of layers 5–7; hence, layers 8–9 had a better gas adsorption capacity. The hysteresis loop type returned to H_2 type (Fig. 3(e)) and the pore morphology was mainly ink-bottle-like.

To characterize the shale pores more intuitively, we used SEM to further determine the pore types. Two-dimensional features of nano- to micro-scale pores in shales can be observed using high-resolution SEM. The qualitative description of pore types, morphology, and

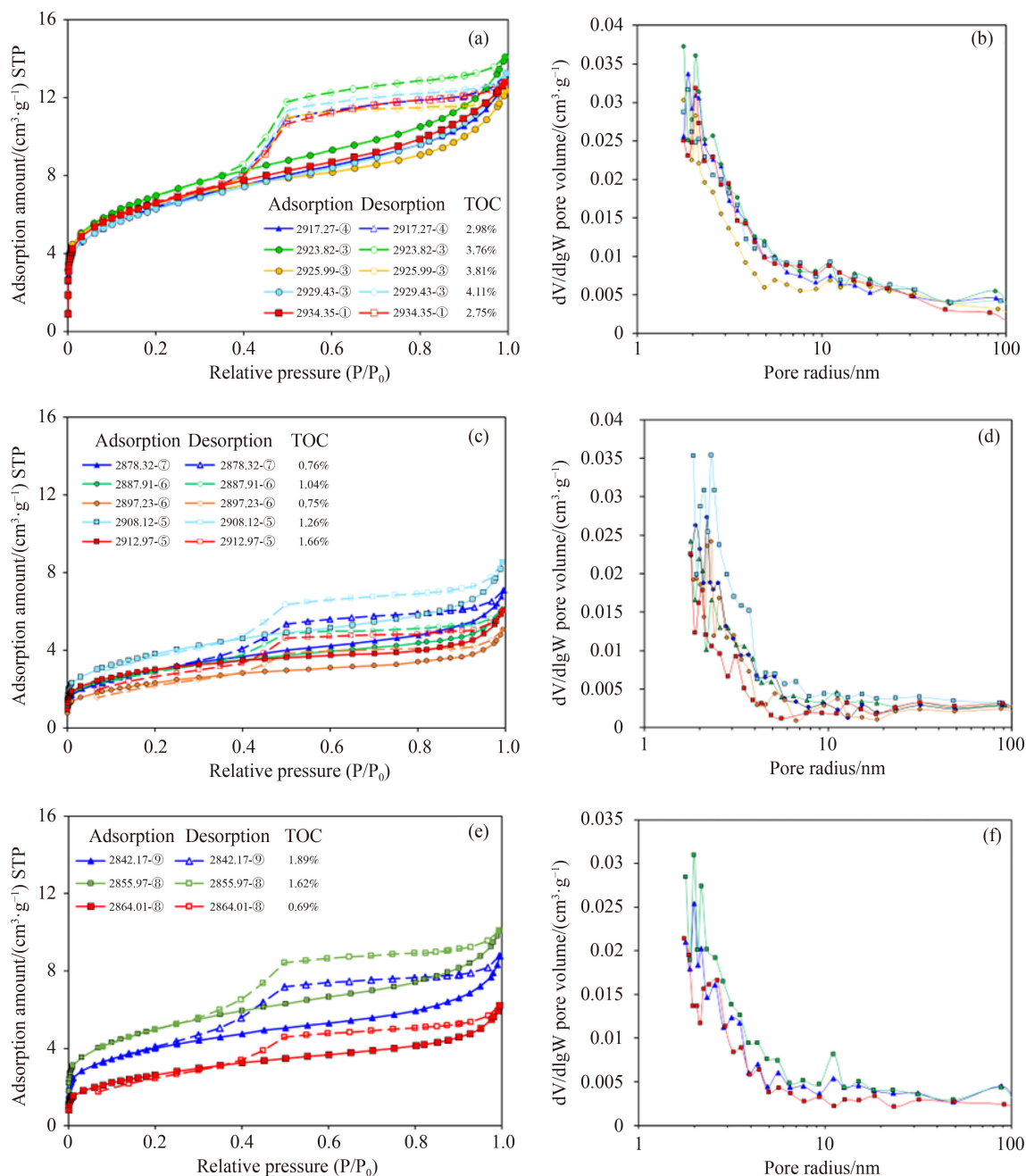


Fig. 3 Nitrogen adsorption-desorption isotherms and Pore diameter distribution of Long 1 Member shale samples. (a–b) Layers 1–4; (c–d) layers 5–7; (e–f) layers 8–9.

contact relationships between mineral particles in reservoirs is based on SEM images. According to the pore classification scheme of Zhao (2020), the pore types of the Long 1 Member shale were classified into three categories by studying SEM images: organic pores, inorganic pores, and microfractures (Figs. 4 and 5).

The organic pores in the reservoir were further classified into five categories: kerogen nanopores (Fig. 4(a)), graptolite OM pores (Fig. 4(b)), framboidal pyrite inter-crystalline pores (Fig. 4(c)), clay mineral-OM aggregates (Fig. 4(d)), and solid bituminous OM pores (Fig. 4(e)). Pyrite in the Long 1 Member occurred mainly in framboidal, granular, and irregular shapes. Within the organic-rich shale section there was a large quantity of framboidal pyrite, and a large number of organic pores were developed between these crystals. The granular and irregular-shaped pyrite was cemented by clay minerals, housing clay mineral pores and organic pores. These pores provided storage space for the preservation of shale gas. We also found that solid bitumen had developed among mineral particles; this was mainly elliptical and irregular in shape and contained organic nanopores.

Inorganic pores within the Long 1 Member mainly took the form of brittle mineral-related pores (Figs. 5(a), 5(b)) and clay mineral inter-crystalline pores. The brittle mineral-related pores included edge pores and dissolution pores. The main cause of dissolution pores in brittle minerals is the dissolution of feldspars and carbonates by acidic fluids generated via raw hydrocarbons, and results in the formation of irregularly shaped pores (Fig. 5(a)) (Zhou et al., 2022). Brittle mineral edge pores were mostly developed between brittle minerals and clay

minerals, and the pore shapes were mostly slit-like or irregular (Fig. 5(b)). Clay interlayer fractures dominated the clay mineral inter-crystalline pores, with the pores mainly taking the form of narrow-slit and lamellae shapes (Figs. 5(c), 5(e)). In addition, clear OM shrinking fractures (Fig. 5(d)) were visible at the contact surfaces between minerals and OM, which were slit-like with an uneven pore-size distribution. Microfractures can accommodate small quantities of free and adsorbed gas and provide a particular type of space for gas storage in shale reservoirs. The shale microfractures in the Long 1 Member were mainly developed at the mechanically weak surface between different material components, and their lengths were of the micrometer scale (Fig. 5(e)). The causes of microfracture formation are mainly dissolution, stress compression, and cracking of mechanically weak surfaces (Guo et al., 2016).

5.2 Quantitative characterization of microscopic pores

In this study, we performed quantitative characterization of shale pores using N_2 adsorption, but the differences in experimental results caused by different types of pores could not be accurately deduced. Therefore, we used additional SEM imaging for the quantitative evaluation of different pore types. Zhao et al. (2018a) qualitatively analyzed pores at different spatial scales and quantitatively analyzed pores using *Image-Pro Plus* software, providing a new way to study pores at the microscale in shale reservoirs. Herein, we refer to the quantitative approach of Zhao et al. (2018a). Thirteen representative SEM images of 13 shale samples from

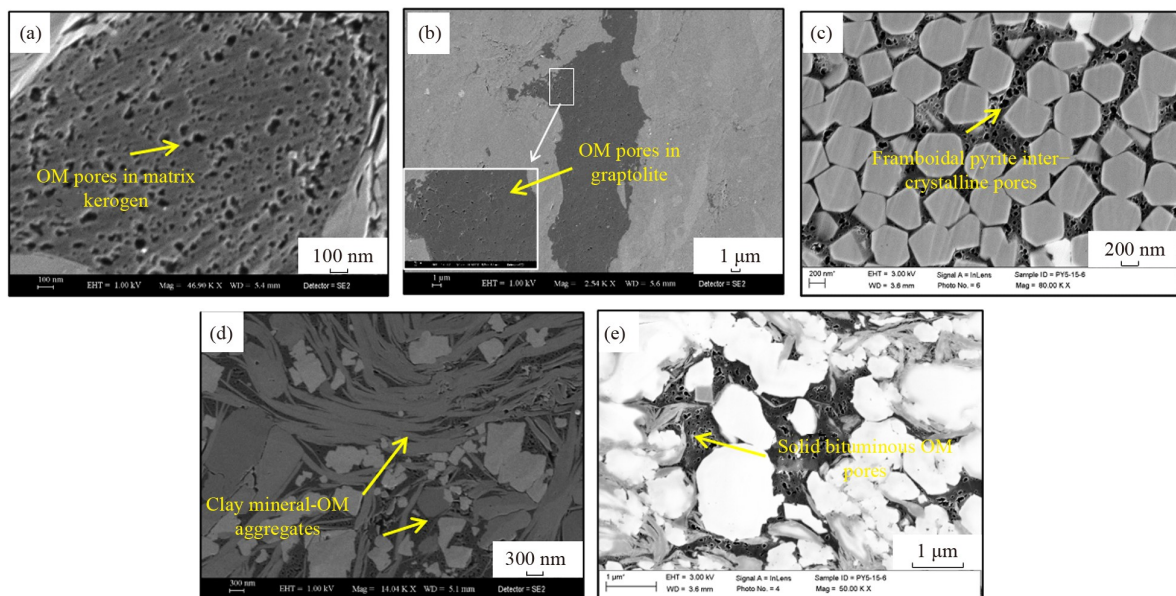


Fig. 4 Organic pore types of shale sample Long 1 Member, Well PY-A. (a) Nano-organic pores in matrix kerogen, layer 9 (2842.17m), TOC (1.89%); (b) OM pores in graptolite, layer 8 (2864.01m), TOC (0.69%); (c) framboidal pyrite inter-crystalline pores, layer 3 (2923.82m), TOC (3.76%); (d) clay mineral-OM aggregates, layer 5 (2908.12m), TOC (1.26%); (e) solid bituminous OM pores, layer 3 (2923.82m), TOC (3.76%).

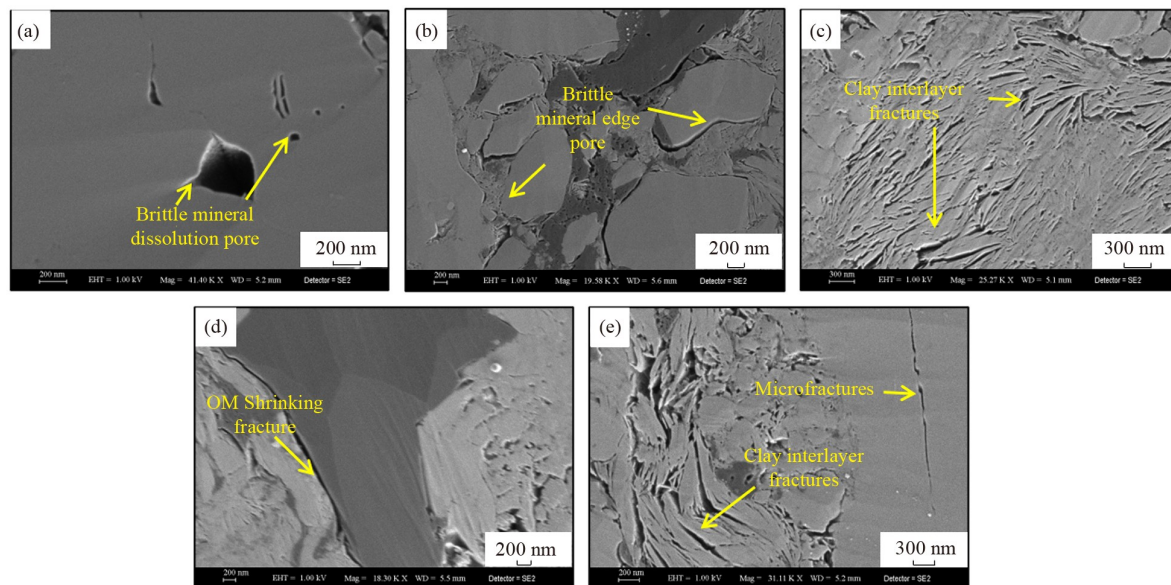


Fig. 5 Types of inorganic pores and microfractures in the shale of Wufeng–Longmaxi Formation, Well PY-A. (a) Brittle mineral dissolution pore, layer 6 (2897.23 m), TOC (0.75%); (b) brittle mineral edge pore, layer 8 (2864.01 m), TOC (0.69%); (c) clay mineral intercrystallite pores, layer 6 (2897.23 m), TOC (0.75%); (d) OM shrinking fractures, layer 6 (2887.91 m), TOC (1.04%); (e) microfractures, layer 3 (2925.99 m), TOC (3.81%).

layers 1–9 were selected based on the degree of pore development and pore distribution characteristics. The field of view of these images was $1 \mu\text{m}^2$. Among them, three images of the same sample with a field of view of $1 \mu\text{m}^2$ were chosen for repeated quantification to verify the accuracy of the method. The error of the repeated quantification results was $< 2\%$. *ImageJ* mapping software was used to analyze the images, providing information regarding the areas and scales of different pores, and permitting the quantification of information from different types of pores.

We applied quantitative characterization using multi-scale SEM images to characterize the microstructure of the pores as accurately as possible. Organic pores were generally nanoscale, making them difficult to identify on microscale SEM images (Figs. 6(a), 6(d), 6(g)). To ensure the accuracy of the results, the OM areas were individually scaled to the nanoscale (Figs. 6(b), 6(e), 6(h)) and quantitatively characterized to obtain nanoscale pore area porosity (porosity being the ratio of pore area to scan plane area). The area porosity was substituted into the total OM area to calculate the OM pore area at the microscale of Figs. 6(a), 6(d), and 6(g). For the software-identified pores, manual comparison was performed to correct the pore edges for accuracy (Fig. 6); the quantitative characterization results are shown in Fig. 7.

The pore types were characterized qualitatively using SEM images from Well PY-A. There were substantial differences in the distributions of different types of pores in the vertical plane of the Long 1 Member. Organic pores dominated in layers 1–4, accounting for $> 46\%$ of all pore area; brittle mineral edge pores, dissolution pores, and clay mineral inter-crystalline pores were also present.

The main reservoir pore types in these layers were organic pores, brittle mineral edge pores, and dissolution pores. Pores in layers 5–7 were dominated by brittle mineral-related pores; compared with layers 1–4, the contribution from organic pores was markedly lower owing to the lower TOC content. The proportion of clay mineral inter-crystalline pores increased in layers 5–7 compared with layers 1–4. The main reservoir pore types in layers 5–7 were brittle mineral edge pores, dissolution pores, and clay mineral inter-crystalline pores. In layers 8–9, clay mineral inter-crystalline pores were the dominant pore type and organic pores were more common. The main reservoir pore types in these layers were clay mineral inter-crystalline pores and organic pores. These results are consistent with those of the nitrogen adsorption experiments.

The contributions of different types of pores to porosity were calculated based on our quantitative characterization results. Organic pores contributed most of the porosity in layers 1–4. Layers 5–7 were dominated by brittle mineral-related pores, and showed low porosity resulting from the low TOC content. Brittle mineral pores contributed more to the porosity of layers 1–9, with an average of 46.1%. Clay mineral inter-crystalline pores contributed most of the porosity in layers 8–9. But the clay mineral pores contributed less to the porosity of layers 1–7. The calculated results show that clay mineral inter-crystalline pores contribute less porosity (Fig. 7).

5.3 Exploration of the main control factors of vertical heterogeneity of microscopic pores

On the basis of our nitrogen adsorption results, we

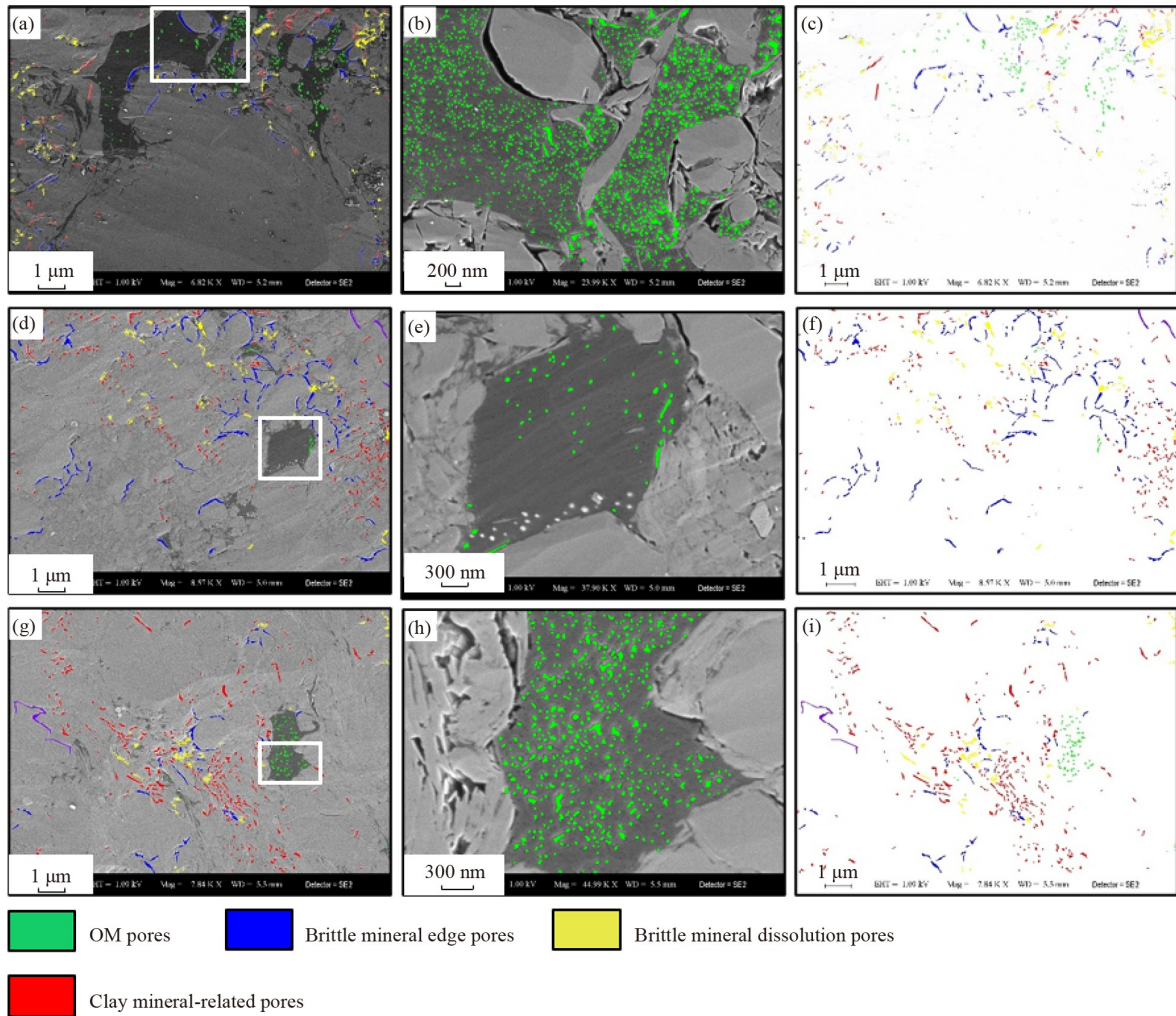


Fig. 6 Quantitative characterization of organic and inorganic pores in different layers of the Wufeng–Longmaxi Formation. (a–c) layer 3 (2925.99 m), TOC (3.81%), porosity (5.109%), OM areal porosity (17.43%); (d–f) layers 7 (2878.32 m), TOC (0.76%), porosity (1.719%), OM areal porosity (3.52%); (g–i) layer 8 (2855.97 m), TOC (1.62%), porosity (4.715%), OM areal porosity (16.44%).

conducted correlation analysis of TOC content and pore parameters for different pore-size ranges. We found that TOC content showed a strong positive correlation with pore volume and specific surface area for some pore-size ranges (Fig. 8). Pore volume and specific surface area of micropores (< 2 nm) and mesopores (2–50 nm) showed a strong positive correlation with TOC content, indicating that OM pores dominated the pore type for these size ranges. The volume and specific surface area of macropores (> 50 nm) did not correlate significantly with TOC content, reflecting the fact that the influence of OM on the development of pores becomes more limited with increasing pore size.

On the basis of our quantitative characterization SEM results, we found that the different types of pores in each layer were influenced by different factors (Fig. 9). The pore type of the shale was mainly controlled by the TOC content, despite the high content of brittle minerals in layers 1–4. Therefore, the pore type in layers 1–4 was

dominated by organic pores, with a lesser percentage of brittle mineral-related pores (Fig. 9(a)). Layer 1 showed an increased clay content and decreased brittle mineral content compared with layers 3–4. Although layers 1–4 were all dominated by organic pores, the percentage of clay mineral inter-crystalline pores was highest in layer 1. The brittle mineral content of layers 5–7 was similar to that of layers 3–4, but the reduced TOC content in these layers led to a higher proportion of pores associated with brittle minerals. The average TOC content of layers 8–9 was only 0.35% higher than that of layers 5–7, but the porosity in layers 8–9 was much higher. Thus, mineral composition appears to be the main factor accounting for the difference in pore types. Clay content had a positive correlation with the percentage of clay mineral pores (Fig. 9(c)). The pore type was dominated by clay interlayer fractures in layers 8–9 that had a higher clay content. In layers 8–9, the mineral composition of the shale at the base of layer 8 was similar to that of layer 7,

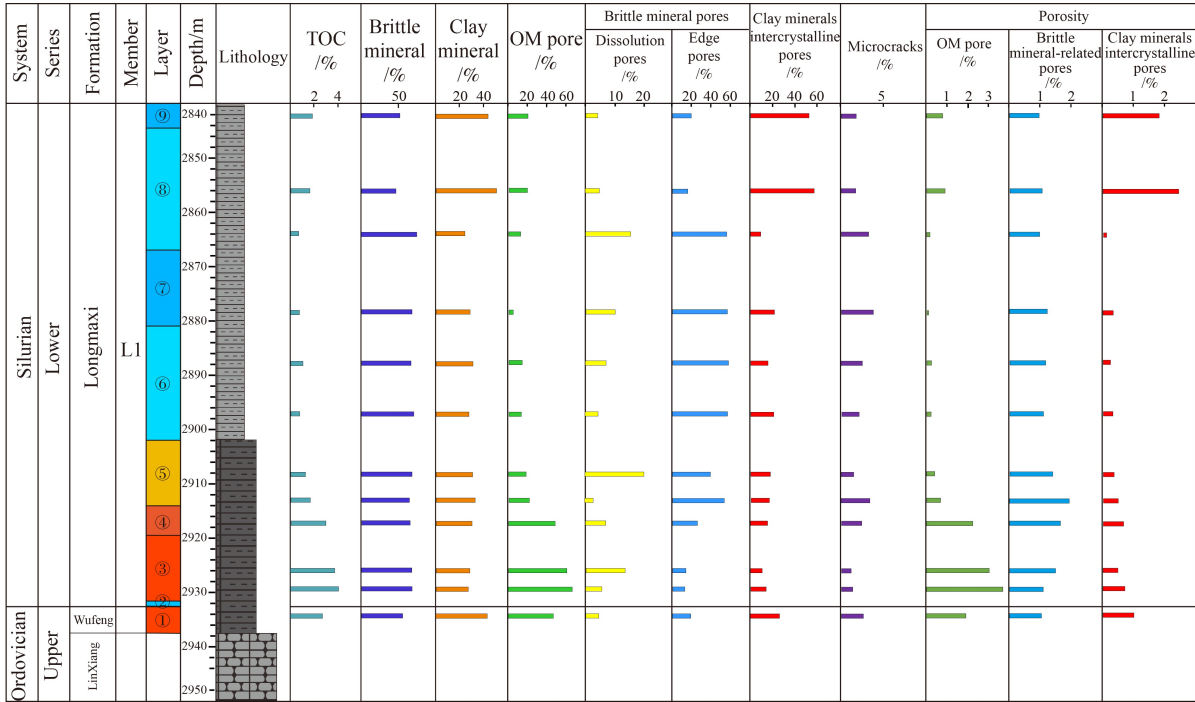


Fig. 7 Percentage composition diagram of organic pores, inorganic pores, and microfractures in different shale layers of Wufeng–Longmaxi Formation, Well PY–A.

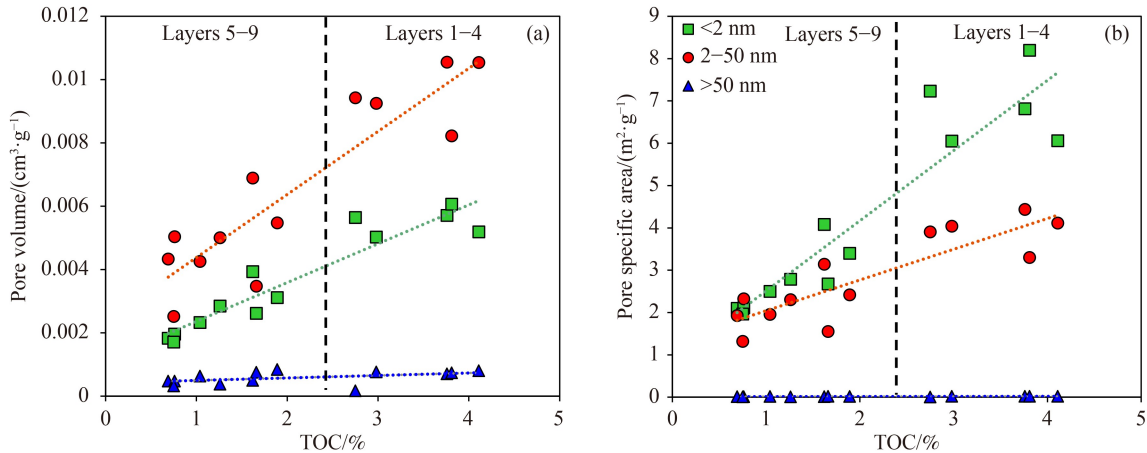


Fig. 8 Correlation analysis of TOC content of shale samples with (a) pore volume and (b) the pore-specific surface area of different pore sizes.

with a high brittle mineral content and low clay content. Therefore, the pore type at the base of layer 8 was dominated by brittle mineral-related pores.

As shown in Fig. 2, the porosity of layers 1–4 and 8–9 was higher than that in other layers. The porosity increased with increasing TOC content (Fig. 10(a)). Porosity of the organic-rich shales from layers 1–4 and organic-poor shales (TOC < 2%) from layers 5–9 was controlled by TOC content. In addition, the porosity of shales in layers 5–9 was also secondarily influenced by clay content (Fig. 10(b)). This is consistent with a previous study in north-western Guizhou (Cao et al., 2020). The development of clay mineral lamellar

structures generates a large number of micropores between clay crystals, which provide a large number of adsorption sites for gases, contributing to greater porosity (Zhang et al., 2013; Tang and Fan, 2014; Wang et al., 2014a).

As quartz is biogenic in this region, the TOC content of layers 1–4 positively correlated with quartz (Yi et al., 2020). In contrast, the TOC content of layers 5–9 did not correlate with quartz and positively correlated with clay minerals (Fig. 11), indicating that the sedimentary period was influenced by detrital materials of terrestrial origin. This is consistent with a previous study (Wu et al., 2017).

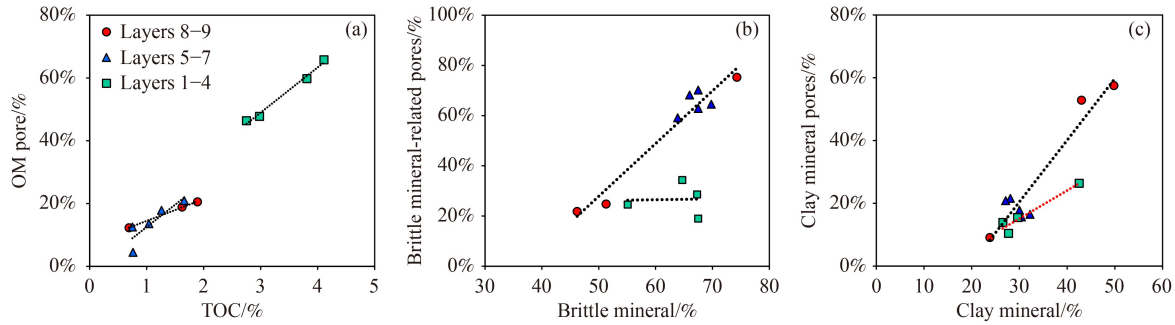


Fig. 9 Correlation analysis of organic matter pore (a), brittle mineral related pore (b), and Clay mineral inter-crystallite pores (c) percentage with TOC and mineral composition.

Different pore types can affect the connectivity and permeability of a reservoir. Micro-CT scanning can clearly show the connectivity and permeability of sample pores. However, the resolution of micro-CT is limited to approximately 0.7 μm , so some connected nanopores cannot be identified. The micro-CT scan model showed that the pores of layer 3 shale samples were more developed than those of layer 5 (Fig. 12). The connectivity was higher because brittle mineral-related pores dominated the pore type in layer 5. The permeability characterization results of the CT scans agreed with our experimental permeability measurements, and indicated that the pores associated with brittle minerals contributed more permeability.

In summary, TOC content is the main factor controlling the vertical differences in organic pores and porosity within the Wufeng–Longmaxi shales; TOC content, together with mineral composition, controls the distribution characteristics of different types of pores and the distribution of vertical permeability.

6 Pore development model

During the Late Ordovician–Early Silurian period, the Upper Yangzi region took the form of a foreland basin at

the front edge of an orogenic belt owing to the influence of the collision of the Huaxia and Yangzi plates (Feng et al., 2021). A series of dark shales of deep-water shelf sub-facies were deposited at the beginning of the Late Ordovician (Wang et al., 2015; Huang et al., 2017). The Wufeng–Longmaxi Formation in the study area went through a burial stage from the beginning of deposition until the end of the Garriodon, then began to slowly uplift in the early Hesperian. Devonian and Carboniferous strata are missing, and a small amount of erosion (approximately 300 m) occurred during the Indochina movement (Lu et al., 2007). Tectonic uplift occurred during the Yanshan and Himalayan movements, and the total amount of strata erosion reached > 4000 m (Yuan et al., 2014; Xu et al., 2015). According to the lithofacies division scheme of Wang et al. (2014b), lithofacies of the Long 1 Member are dominated by siliceous shale (Fig. 13). During the deposition of the Wufeng Formation, intense volcanic activity played an important role in the enrichment of OM (Wu et al., 2018), resulting in a relatively high OM content in the lower part of the Longmaxi Formation and upper part of the Wufeng Formation. The base of layer 1 comprises claystone with poor hydrocarbon generation potential. The underlying Ordovician Baota/Linxiang Formation is composed of limestone. Therefore, this claystone at the base of layer 1,

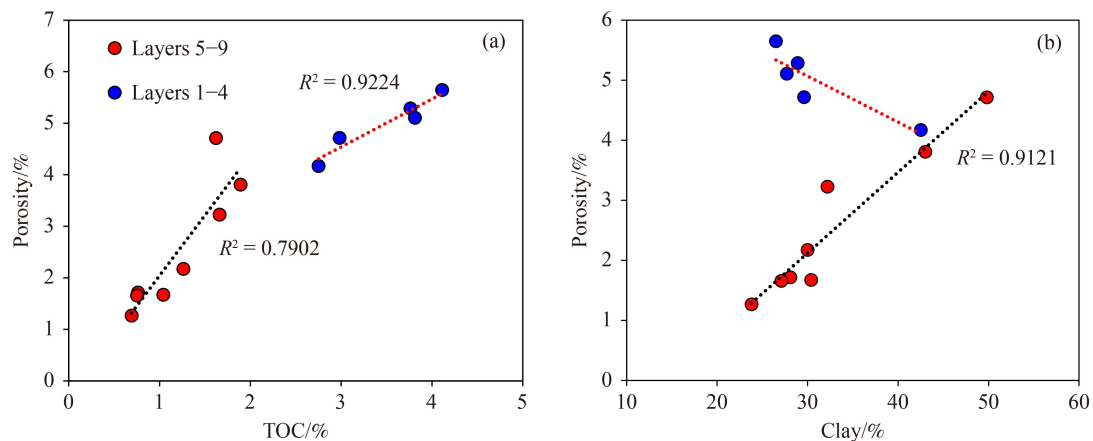


Fig. 10 Correlation analysis of porosity with TOC (a) and clay content (b).

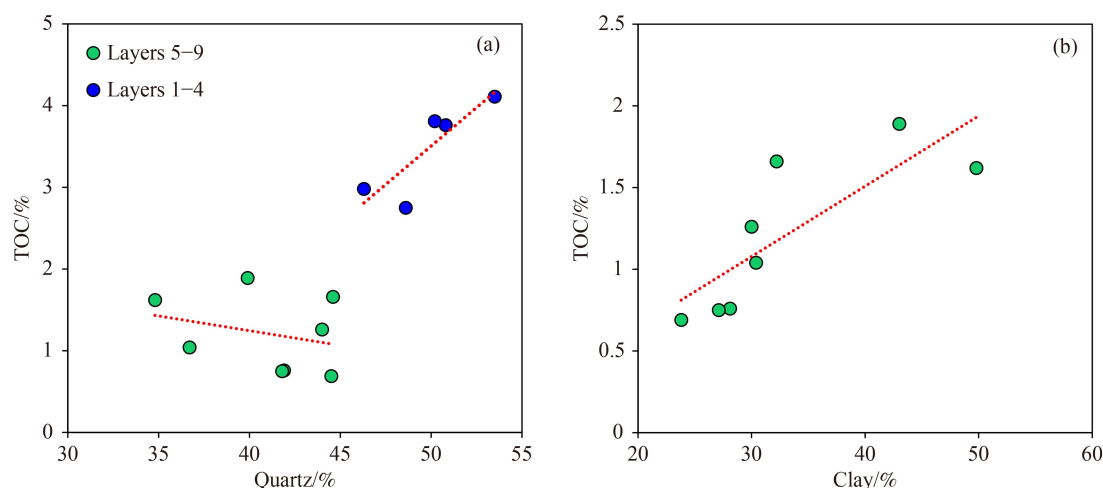


Fig. 11 Correlation analysis of TOC with Quartz (a) and clay content (b).

together with the underlying Baota/Linxiang Formation, ensures the hydrocarbon sealing of Longmaxi shale gas; it prevents hydrocarbon dispersal from the shales of the Wufeng–Longmaxi Formation, allowing OM to be enriched and preserved (Hu et al., 2014; Wei et al., 2017; Zhang et al., 2022). The top of layer 1 is characterized by a siliceous shale showing high TOC content, porosity, and gas content, and this has been confirmed to be a hydrocarbon generation layer. Layers 3–4 are silica-rich and high-TOC shales, which are the main production strata.

The end of the Hirnantian Glaciation and associated recovery of global temperatures led to a rise in sea level, which transformed the sedimentary environment of layers 3–4 into one of anoxia. Meanwhile, mild volcanic activity also favored the accumulation of OM (Wu et al., 2018). The water was warm in the study area during this period, and primitive organisms began to bloom. Planktonic microalgae sank through the water column after death, while siliceous organisms, such as graptolites, diatoms, and radiolarians, were enriched on the seafloor, and a small amount of pyrite was deposited (Yan et al., 2018). Therefore, layers 3–4 have a high OM content. The quantitative characterization results attained using SEM showed that organic pores dominate layers 3–4. Combined with the results of nitrogen adsorption experiments, micropores and mesopores have been shown to dominate the pore size. Micropores mainly comprise organic pores and clay interlayer pores (Xiao et al., 2019), and previous studies have shown that OM and clay minerals have a strong adsorption capacity for methane (Zhang et al., 2013; Tang and Fan, 2014); therefore, micropores provide a substantial amount of storage space for adsorbed gas. Correlation analysis showed that mesopores also provide a portion of the specific surface area (Fig. 8b). Mesopores consist of OM, interlayer fractures of clay minerals, and brittle mineral dissolution pores (Xiao et al., 2019), and can again provide

adsorption sites for gas molecules. Therefore, layers 3–4 comprise an excellent reservoir.

In the middle and late period of Longmaxi Formation sedimentation, the aqueous environment gradually changed from anaerobic to oxygen-rich, and the sediments changed from black shale to gray-black shale. The shale was influenced by the enhanced input of land-derived debris and the sedimentation rate accelerated considerably (Zhao et al., 2018b). Layers 5–7 are low-TOC siliceous shales, with brittle mineral-related pores as the dominant pore type and a low percentage of organic pores. The development of a large number of brittle mineral-related pores gives this section excellent pore connectivity and high permeability, which may easily lead to gas escape under complex tectonic conditions. In layers 8–9, the shale lithofacies transitions from siliceous shale to clay shale. These layers are rich in clay minerals, the content of brittle minerals is lower than that in layers 1–7, and consequently the permeability is lower. Compared with layers 5–7, these layers have higher OM areal porosity and porosity. The change in the anoxic environment indicated the gradual drop of sea level, the clastic supply from terrestrial sources (Wang et al., 2014a), and the deposition of clay-rich shale. The thickness and stable distribution of this shale makes it an excellent reservoir caprock. The gas content of layers 8–9 is also higher than that in layers 5–7, while the permeability is lower, owing to a higher proportion of the pores being clay mineral inter-crystalline pores. Meanwhile, pores are widely developed in clay mineral-OM aggregates, providing abundant shale gas storage space. Under suitable conditions for shale gas preservation in complex tectonic zones, layers 8–9 comprise potential normal-pressure shale gas target layers.

In summary, the characteristics of the Wufeng–Longmaxi Formation in the study area are mainly controlled by the sedimentary environment. Continuous changes in the

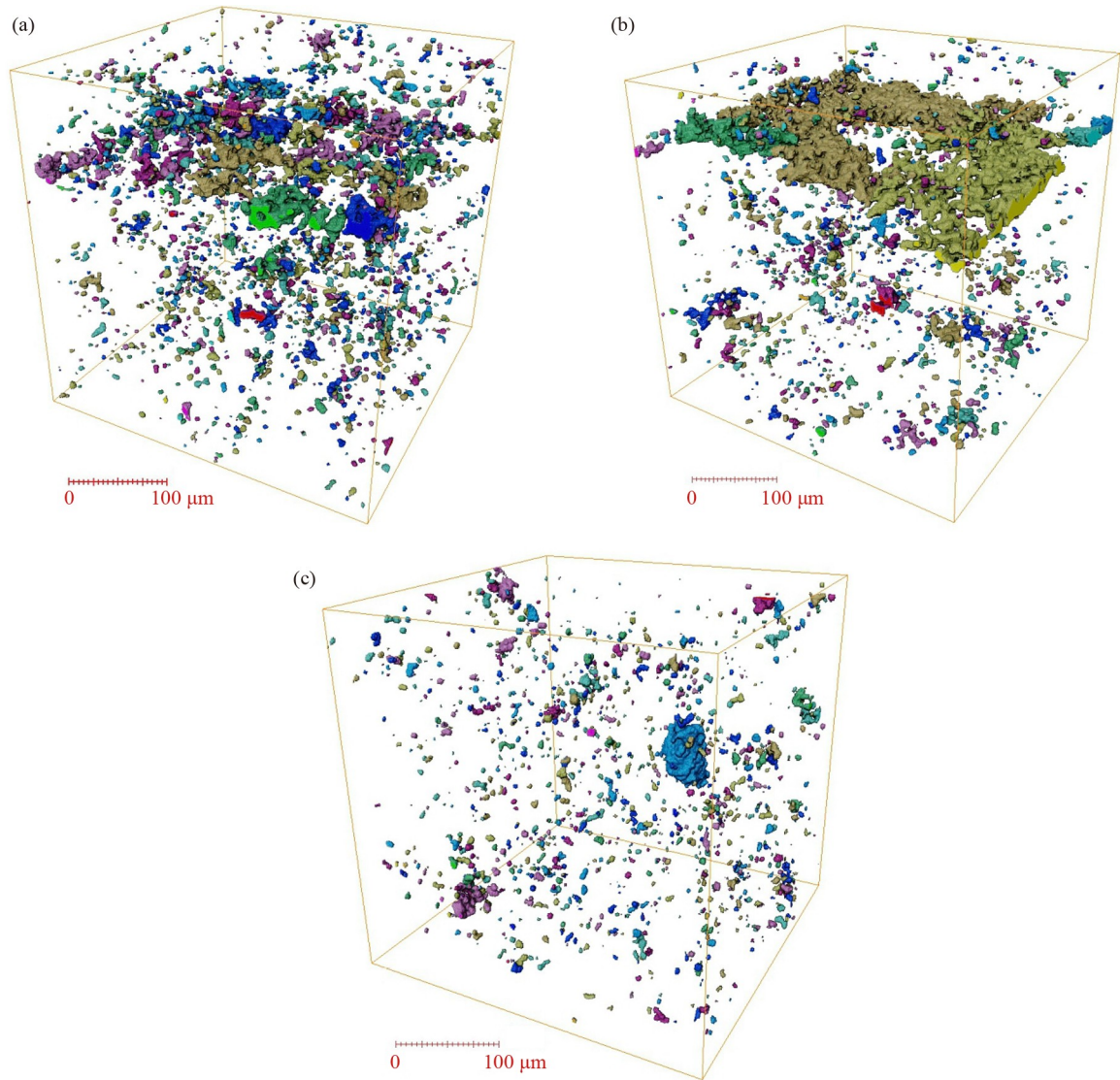


Fig. 12 Micro-CT scanning 3D model of Wufeng-Longmaxi Formation (same color for the same type of pore). (a) 2925.99 m, sample of layer 3; (b) 2908.12 m, sample of layer 5; (c) 2855.97 m, sample of layer 8.

sedimentary environment have led to the heterogeneous vertical distribution of OM and rock mineral composition. This is also the reason behind the differences in microscopic pore development of shales in different layers.

The reservoirs of the Longmaxi Formation shale have been studied previously, but most scholars have proposed models based on the sedimentary evolution of the reservoirs without outlining the complete reservoir development process (Chen et al., 2023; Dong et al., 2023). Based on this previous understanding, in this paper have proposed a model for the development of pore and mineral distribution characteristics of layers 1–9. The development model of the shale reservoirs in the Long 1 Member is shown in Fig. 14. Organic pores dominate the upper part of layer 1 accompanied by a high TOC content and brittle minerals. The base of layer 1 has a high clay

content accompanied by low permeability and porosity, which is conducive to the sealing and preservation of shale gas (Fig. 14(a)). Layers 3–4 have well-developed organic pores and a high TOC content, which benefits shale gas enrichment and preservation (Fig. 14(b)). The high brittle mineral content also facilitates fracturing during late production gas. Layers 5–7 have a high brittle mineral content, poor organic pore development, and a large number of brittle mineral edge pores and dissolution pores (Fig. 14(c)), which are inconducive to shale gas enrichment and preservation. Pore development in layers 8–9 is jointly controlled by TOC and clay minerals, and organic pores and clay mineral inter-crystalline pores are dominant here. The quartz content decreases from the bottom to the top of Long 1 Member, and a substantial amount of graptolite OM is present. Pyrite in the upper part of Long 1 Member is lower and mostly cemented

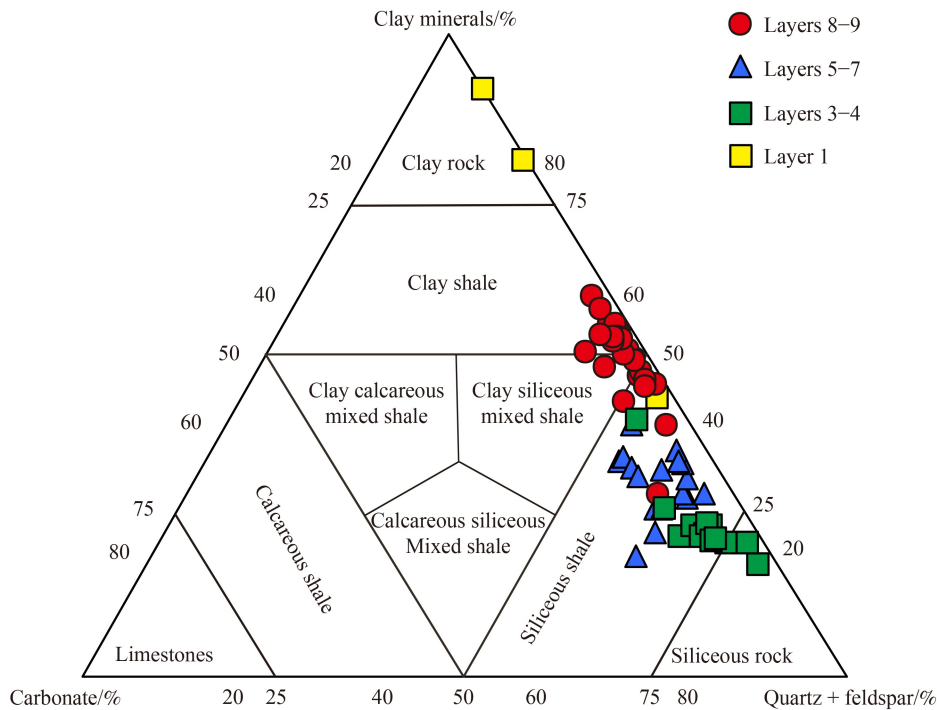


Fig. 13 Classification characteristics of shale lithofacies of layers 1–9. Lithofacies division scheme from Wang et al. (2016).

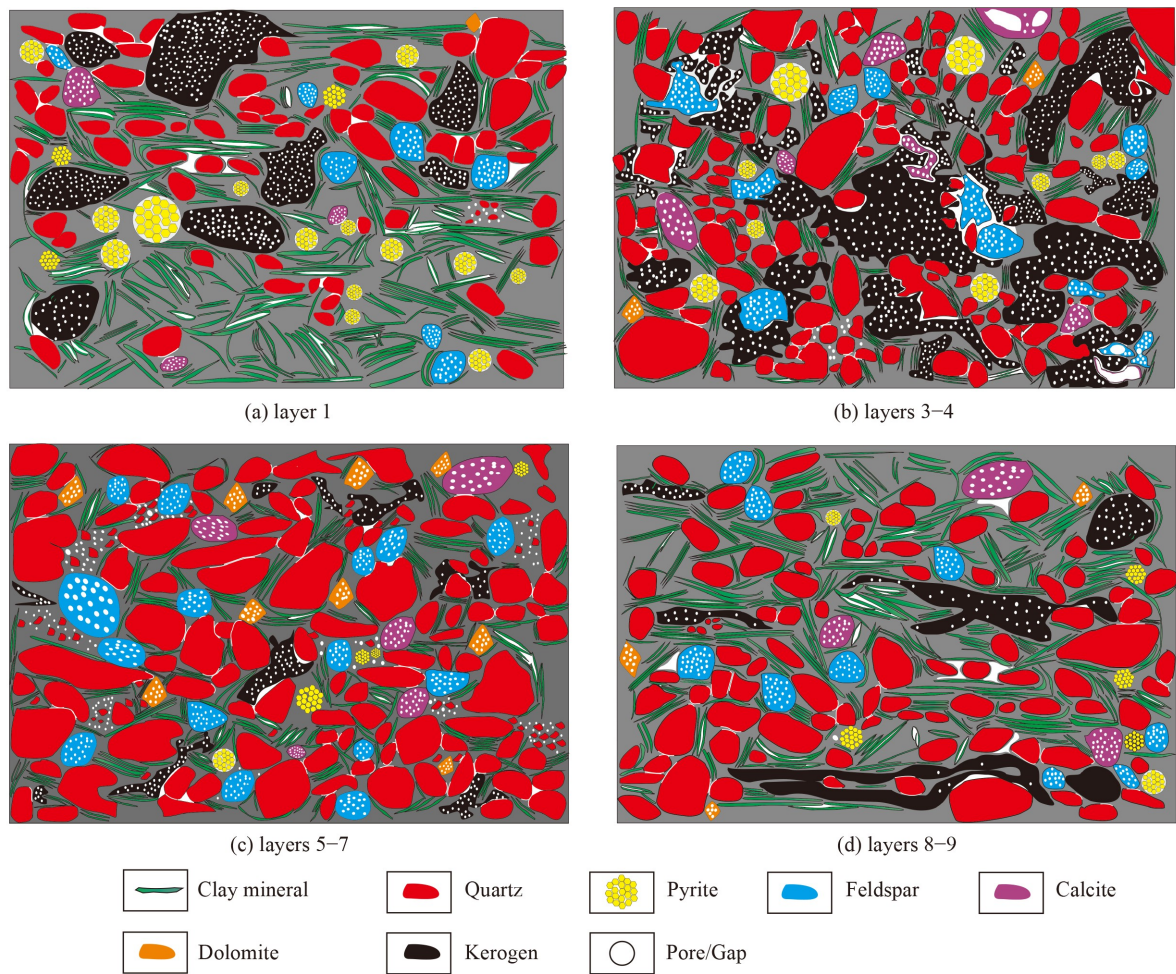


Fig. 14 Mineral composition and pore type model of shales in layers 1–9.

with clay minerals, providing a certain degree of support to some of the clay mineral inter-crystalline pores. The mineral and pore compositions of the base of layer 8 are consistent with those of layers 5–7 (Fig. 14(d)), dominated by quartz, brittle mineral-related pores, and clay mineral inter-crystalline pores.

In conclusion, layers 1–4 comprise the most favorable development section in the study area. Despite the decrease in TOC content, layers 8–9 have a good overall gas content; moreover, the quartz content is > 30%, ensuring fracturability, and allowing the exploration and development of layers 8–9 in this normal-pressure shale block with TOC > 2%.

7 Conclusions

1) The Long 1 Member in the Pengshui area shows substantial vertical variability, controlled by the changing sedimentary environment at the time of deposition. The OM abundance and total gas content of reservoirs in layers 1–4 were highest, averaging 3.15% and 5.55 cm³/g, respectively. The reservoirs of layers 5–7 were the poorest, while the TOC content and total gas content of layers 8–9 were higher than those of layers 5–7. The TOC and total gas content of the Long 1 Member showing a three-stage trend of “two highs and one low”.

2) Microscopic pores in the shales were affected by various factors. Differences in TOC content and mineral composition between layers led to differences in the distribution of different types of pores. Layers 1–4 were dominated by organic pores; layers 5–7 were dominated by brittle mineral-related pores; and layers 8–9 were dominated by clay mineral inter-crystalline pores. Pore sizes were mainly microporous and mesoporous.

3) The differential distribution of pore types affected the reservoir properties of the shale. A large number of clay mineral inter-crystalline pores in layers 8–9 provided good sealing conditions and allowed the preservation of a certain amount of shale gas. Meanwhile, brittle mineral-related pores in layers 5–7 allowed the easy escape of gas, while the organic-rich shale of layers 1–4 exhibited good enrichment and reservoir formation conditions. In addition, the excellent roof and floor conditions of the reservoir enabled the preservation of the generated shale gas.

4) On the basis of organic geochemical characteristics and a pore development model, we consider layers 1–4 to be excellent production strata, while layers 8–9 can be used as potential target production strata.

This study provides a theoretical basis for the future exploration and development of normal-pressure shale blocks.

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