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INNOVATIVE TECHNOLOGIES OF OIL AND GAS

RESEARCH ON SHALE RESERVOIR CHARACTERIZATION AND CONTROL FACTORS

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This study focuses on the characterization of shale gas reservoirs in the Longmaxi Formation and their controlling factors in the east Sichuan Basin. Detailed mineralogical and reservoir characterization of shale samples in the region was carried out by various methods, including X-ray diffraction analysis and nitrogen adsorption experiments. The results show that the shale is mainly composed of clay minerals (illite content ranges from 34.9% to 55.7%), quartz and calcite. In terms of reservoir characteristics, the shale mesopore morphology is mainly "slit-type", with BET specific surface area ranging from 7.12-25.63 m^2/g and BJH pore volume from 0.0095-0.0262 mL/g. These reservoir characteristics show a significant positive correlation with the organic carbon content (1.82-3.87%). correlation. Petrographic analysis further reveals that the brittle mineral content has a significant effect on the brittleness, pore development and fracturing effectiveness of the rocks. In addition, diagenesis (including compaction, cementation, dissolution, and thermal evolution of organic matter) had a significant impact on the formation and characterization of shale pores. These findings provide a key scientific basis for understanding the geological characteristics and development potential of shale gas reservoirs in the Longmaxi Formation in the Sichuan Basin.

Keywords: Sichuan Basin, shale gas, brittle minerals, reservoir characteristics, controlling factors.

1. Introduction

Shale gas, as a typical autogenous self-storage gas reservoir, refers to natural gas endowed in organic matter, mineral microcracks and microporosity of mud shale formation with adsorption state and free state as the main state [1-3]. It plays a key role in changing the global energy pattern and promoting the development of green and low-carbon economy. China is very rich in shale gas resources, especially in areas such as the Upper Triassic in the Sichuan Basin, which possesses huge shale gas potential, and its proven geological resources and technically recoverable reserves rank first in the world [4-7]. In recent years, with the rapid development of the national economy and the urgent demand for clean energy, shale gas exploration and development and theoretical research in China have been emphasized and have begun to enter the development stage.

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Shale gas, as an important unconventional natural gas resource, occupies an important position in the global energy structure. Especially in China, the exploration and development of shale gas resources have received increasing attention. Globally, especially in the United States and Canada, the exploration and development of shale gas has made remarkable achievements, bringing far-reaching impacts to the global energy market. These countries have accumulated rich experience in shale gas development through extensive drilling and testing, and have gained in-depth knowledge of shale gas reservoir characteristics, storage mechanisms, and gas adsorption and flow properties [8-12]. However, due to the differences in geological conditions, the international experience is not fully applicable to shale gas fields in China. Especially in the east Sichuan area of the Sichuan Basin, the widely distributed black shales of the Wufeng Formation-Lower Silurian Longmaxi Formation have become an ideal area for China's shale gas pilot test area and for conducting shale gas reservoir studies due to their high organic carbon content, high degree of thermal evolution, and high content of brittle minerals [13-17]. However, there are still many deficiencies in the research of reservoir characteristics and main controlling factors. Currently, there are fewer studies on the mineralogical characteristics, pore structure, and physical properties of shale gas reservoirs in the region, which limits the possibility of a comprehensive understanding of shale gas reservoirs in the region. In addition, although some preliminary studies on shale gas reservoir characteristics have been conducted at home and abroad, the effects of brittle minerals on shale gas reservoirs and the causes of brittle minerals have not been studied deeply enough [18-20]. Organic matter and mineral compositions are recognized as important factors controlling shale pore development, but the specific understanding of their influence mechanisms is not clear [21, 22]. The complexity of deposition and diagenetic processes in fine-grained sediments is also a weak point in current sedimentological studies [23-25].

In view of this, the present study is aimed at characterizing the black shale reservoirs of the Wufeng Formation-Longmaxi Formation and their controlling factors in the east Sichuan Basin, with the aim of filling the gaps in related studies at home and abroad. We comprehensively analyzed the mineral genesis, pore characteristics, and organic geochemical properties through the techniques of core observation, thin-section analysis, X-ray diffraction, nitrogen adsorption, mercury compression, and scanning electron microscopy, with the aim of revealing the main factors affecting the pore development of the black shale. This study not only helps to better understand the geological characteristics of shale gas reservoirs in the Longmaxi Formation in the east Sichuan Basin, but also has important theoretical and practical significance in guiding the exploration and development of shale gas in the region and even in the whole country.

2. Research materials and methods

The research rock samples used in this paper were taken from wells QY-3, DZT-6, CNSH3, CNN1, Y6, Y8, Y9 and B1 in the east Sichuan Basin. Fifty typical samples were selected from them, of which the rocks used for SEM experiments were taken from 3015.51m in well Y6 and 2270.03m in well Y8. The cores used for X-ray diffraction experiments were taken from QY-3 well at 2960.96, DZT-6 well at 2733.75m, CNSH3 well at 2943.47m and CNN1 well at 2583.66 m.

In this paper, the black shale from the Wufeng Formation-Longmaxi Formation outcrop section and the related drilling wells in the east Sichuan area is taken as the object of study, and the mineralogical, petrological and reservoir physical characteristics of the black shale are investigated in detail from qualitative to quantitative and from macroscopic to microscopic through the combination of field observation and indoor analytical tests.

Core observation and sample collection. In this paper, the black shale at the bottom of the Wufeng Formation-Longmaxi Formation in the east Sichuan area is the object of study, and detailed core and outcrop observations are carried out to describe in detail the structural and tectonic characteristics of the black shale and to complete the sampling work.

Shale pore characterization methods. Qualitative and semi-quantitative characterization of shale pores: before observing the samples with field emission scanning electron microscope, the mud shale samples were dissected with argon ions, and the shale pores were qualitatively analyzed for pore types, sizes, distribution locations, and distribution patterns with the help of energy spectroscopy; nitrogen adsorption can be used to analyze the pore structures, specific surface areas, and pore volumes of some micropore, mesopore, and macroporous pores: quantitative characterization of macropores with the use of pressed mercury is more precise and can be used to analyze the macroporous volume. The quantitative characterization of macropores is more accurate and can be used to analyze the volume of macropores.

Methods for analyzing the mechanism of brittle minerals' influence on shale pore space. Based on the results of preliminary tests, the mechanism of brittle minerals' influence on shale inorganic porosity is analyzed by combining quantitative and qualitative means. Based on the analytical tests, analyze the relationship between brittle mineral content and total porosity, pore type, pore size distribution, pore volume and specific surface area, analyze the influencing factors of shale inorganic porosity, and clarify the influence of brittle mineral content on shale inorganic porosity. Combined with qualitative means such as scanning electron microscope and microscope, the influence mechanism of shale inorganic porosity is analyzed.

3. Results and discussion

3.1. Geological background of the study area

The Sichuan Basin, located in the Yangzi Plate, is a foreland basin evolved from the Craton Basin, covering an area of about 1.8×10^5 km^2 . The basement of this complex marine-land superposed basin consists mainly of a pre-Aurignan metamorphic facies, over which marine carbonates, mudstones, sandstones, and evaporites of the Aurignacian to Middle Triassic, as well as terrestrial clastics of the Late Triassic to Eocene, were deposited. The basement of the basin has a dual structure: the older part consists of mixed gneisses from the Paleozoic to Early Proterozoic, while the newer part consists of shallowly formed metamorphosed green schists from the Middle to Upper Proterozoic. The sedimentary cover mainly consists of two sets of stratigraphic layers: one is the marine sedimentation from the Aurignacian to the Middle Triassic, with a total thickness of 4-7 km; the other is the terrestrial sedimentation after the Late Triassic, with a total thickness of 2-6 km. The basin is surrounded by a large fracture control, including the Great Bashan Fault Belt in the north-east, the Longmenshan Terrace Marginal Fault Belt in the north-west, the Yunnan-Guizhou-Chuan-E-Tai Fold Belt in the south-east, and the Liangshan Block Fault, the Emeishan and Loushan Fault Belts and the Basha Fault Zone, all of which are arranged in order from east to west. Emeishan, Loushan Fracture Zone and Bafangshan Fracture Zone. During the Cenozoic, the Sichuan Basin was in an area of strong extrusion fold uplift activity, and the main sediments were distributed in western Sichuan, with a thickness of about several hundred meters.

3.2. Mineralogical characteristics

X-ray diffraction (XRD) analysis tests on samples collected from drill cores and four outcrop sections of black shale of the Wufeng Formation-Longmaxi Formation in the study area revealed that the mineral composition of black shale of the Wufeng Formation-Longmaxi Formation in the study area is complex, mainly including clay minerals, quartz, calcite, dolomite, feldspar, and pyrite, etc. The mineral composition of black shale of the Wufeng Formation-Longmaxi Formation in the study area is also complex.

In shale gas reservoirs, clay minerals not only affect the reservoir storage properties and gas content, but also are an important factor affecting the fracturing effect of the reservoir. X-ray diffraction analysis shows that the clay minerals in the study area are mainly composed of illite, illite-mixed layer and chlorite (**Figure 1**), of which: illite content ranges from 34.9% to 55.7%, with a mean value of 48.43%; chlorite content ranges from 12.6% to 31.4%, with a mean value of 21.52%; illite content ranges from 22.1% to 35.4%, with a mean value of 30.63%; and illite content ranges from 22.1% to 35.4%, with a mean value of 30.63%. 31.4%, with a mean value of 21.52%; and ilmenite mixed layer ranged from 22.1% to 35.4%, with a mean value of 30.63%. There are six basic modes of clay mineral distribution, and the clay mineral assemblage in the study area is dominated by illite + chlorite + immonite (I+C+I/S), which belongs to the I-type - normal transformation type, indicating that the longitudinal spreading characteristics of the clay minerals are mainly affected by the effect of diagenesis.

The mineral composition of the black shale of the Wufeng Formation-Longmaxi Formation varies greatly in different areas. In this paper, through the X-ray diffraction analysis test on the samples collected from the black shale of the Wufeng Formation-Longmaxi Formation in the drilling cores and outcrop sections in the study area, the results of the study on the mineral distribution characteristics of the shale in the study area are shown in **Figure 2**.

The analysis of the black shale of the Wufeng Formation-Longmaxi Formation (Fig. 2a) reveals the following regular conclusions: quartz and clay minerals are the main constituents, accounting for more than 80% of the total content, and the quartz content varies significantly among wells. wells QY-3 and DZT-6 have higher quartz contents of 55.25% and 69.73%, respectively,

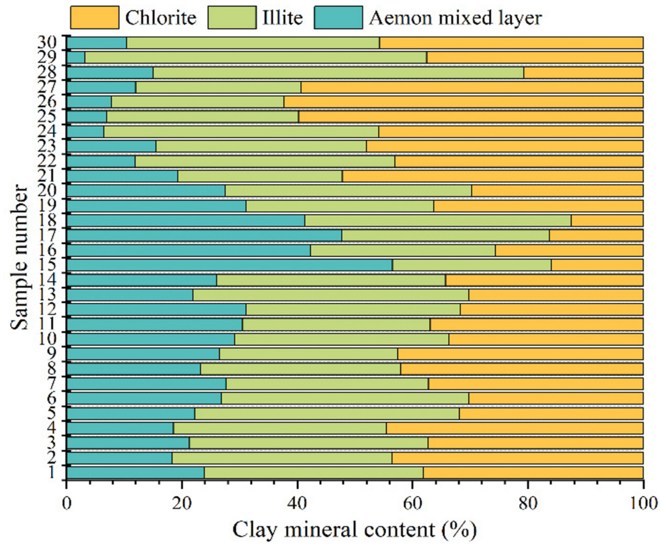

Fig. 1. Clay mineral composition of black shale of Wufeng Group-Longmaxi Group in the study area

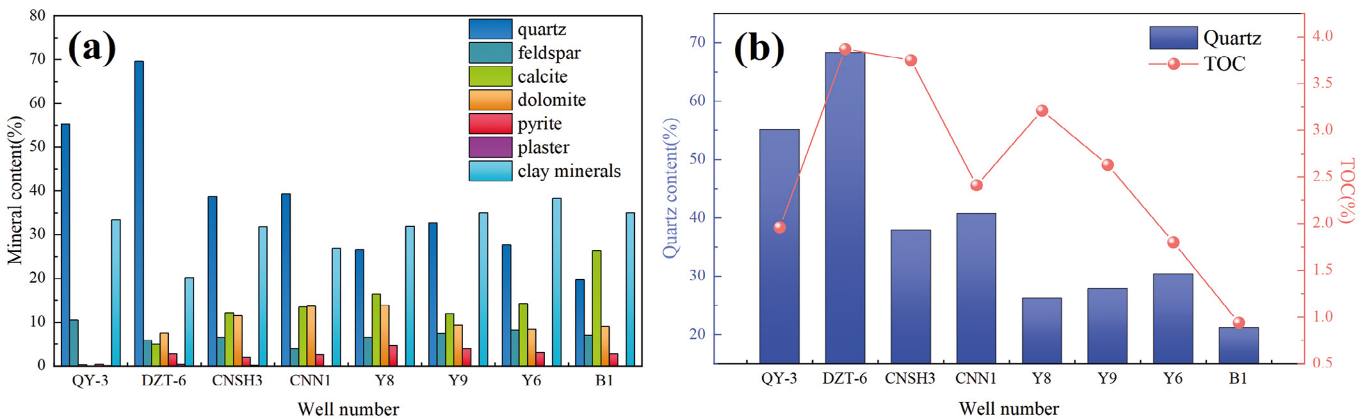

Fig. 2. Mineral composition of the black shale of the Wufeng Formation-Longmaxi Formation and the comparison of quartz and TOC contents in the study area. a) Mineral composition; b) Quartz and TOC contents comparison

which are mainly biogenic quartz, while terrestrial source detrital quartz is less. In contrast, well B1 has the lowest quartz content of 19.73%. wells Y6, Y8, and Y9 have intermediate levels of quartz content, and the detrital quartz content is usually less than 5%. In addition, the organic carbon (TOC) contents of these wells are relatively stable, ranging from 1.82% to 3.87%, indicating a correlation between quartz content and TOC, which supports the biogenicity of quartz (Fig. 2b). Taken together, the mineral compositions of the black shales of the Wufeng Formation-Longmaxi Formation show obvious differences between wells, reflecting the changes of sediments under different geological conditions.

3.3. Reservoir characterization

3.3.1. Analysis of shale mesopore morphology

The isothermal adsorption curves of gases classified by the International Chemical Union in 1985 were divided into six classes (I-VI) [26], and Sing et al [27] who judged the pore shapes by the characteristics of hysteresis loops, classified them into four classes (H1-H4) based on the characteristics of the curves. The nitrogen adsorption curves of the black shales of the Wufeng Formation-Longmaxi Formation in the study area are shown in **Figure 3**. It can be found that the adsorption curves of the organic matter-rich fine-grained shales have obvious hysteresis loops, which is a type IV curve, indicating the development

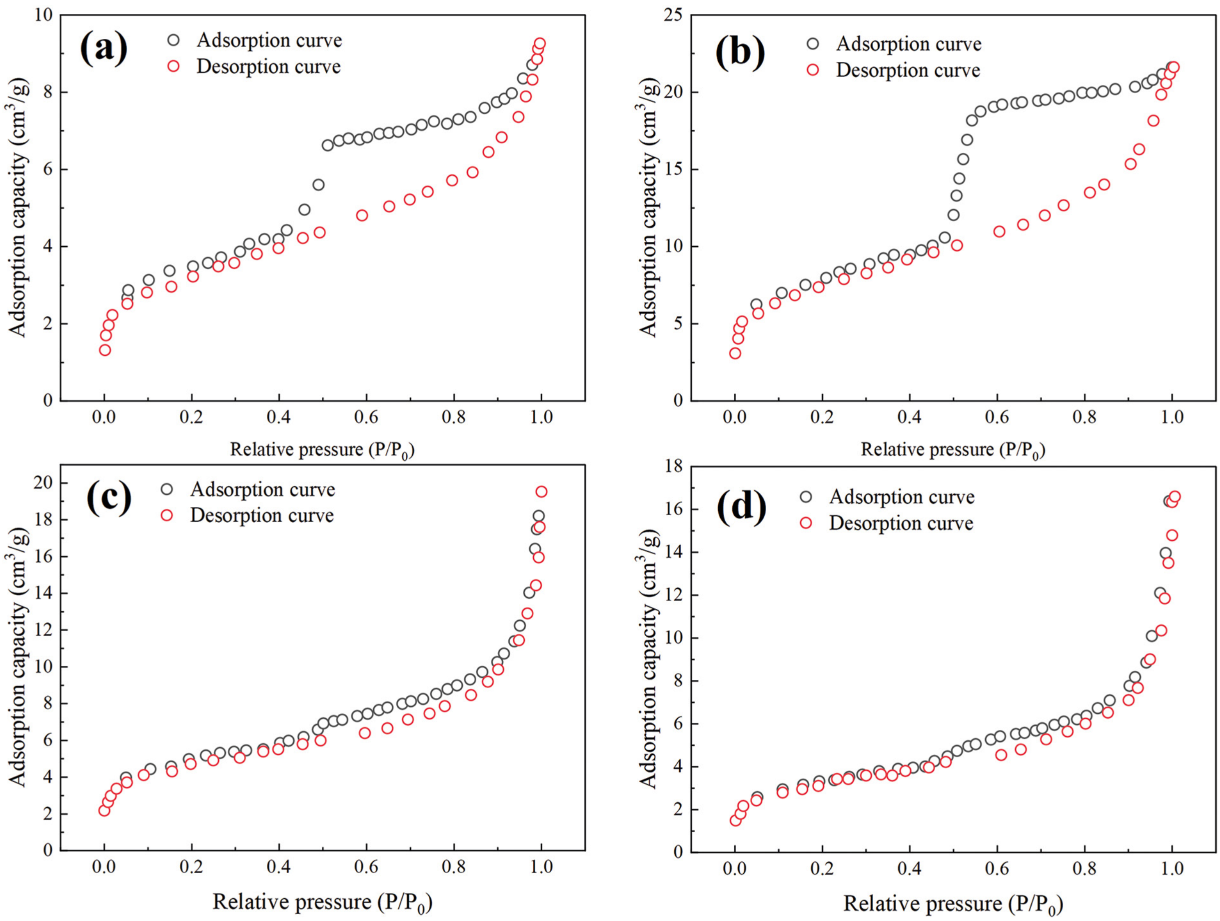

Fig. 3. Characteristics of ammonia absorption curve attached to the black shale of Wufeng Formation-Longmaxi Formation in the study area. a) Y8-2; b) Y8-3; c) Y8-7; d) Y8-12

of mesopores, and the hysteresis curves are similar to those of the type H3, and the shape of the pores is interpreted as "slit-type". The nitrogen adsorption-desorption curves of all the black shales show a "forced closure" phenomenon when the P/P_0 is between 0.45 and 0.5, which is related to the "tensile strength effect", and indicates that these samples have pores smaller than 4 nm. The nitrogen adsorption curves of low organic matter carbonate fine-grained rocks are of type II, which is related to the development of more macropores.

3.3.2. Specific surface area, pore volume and pore size distribution

According to the nitrogen adsorption experiments, the BET specific surface area of the black shale samples from the Wufeng Formation-Longmaxi Formation in the study area was 7.12-25.63 m^2/g (mean value 13.88 m^2/g), and the pore volume of the BJH was 0.0095-0.0262 mL/g (mean value 0.0159 mL/g), and the cumulative pore volume and the specific surface area increased dramatically in the range of the pore diameter of 3-10 nm, which indicating that pores in this range provided most of the pore volume and specific surface area (**Figure 4**).

The specific surface area and pore volume of the shale in the destination layer of the study area have a strong positive correlation with the organic carbon content, indicating that organic matter is the main source of the pore volume and specific surface area of the shale mesopores, while the average pore diameter is inversely proportional to the organic carbon, indicating that the diameter of organic matter pores is smaller than that of the mineral-related pores (**Figure 5**). The pore distribution of shale is mostly concentrated around 4 nm, with one peak present in most samples and two peaks visible in some (**Figure 6**).

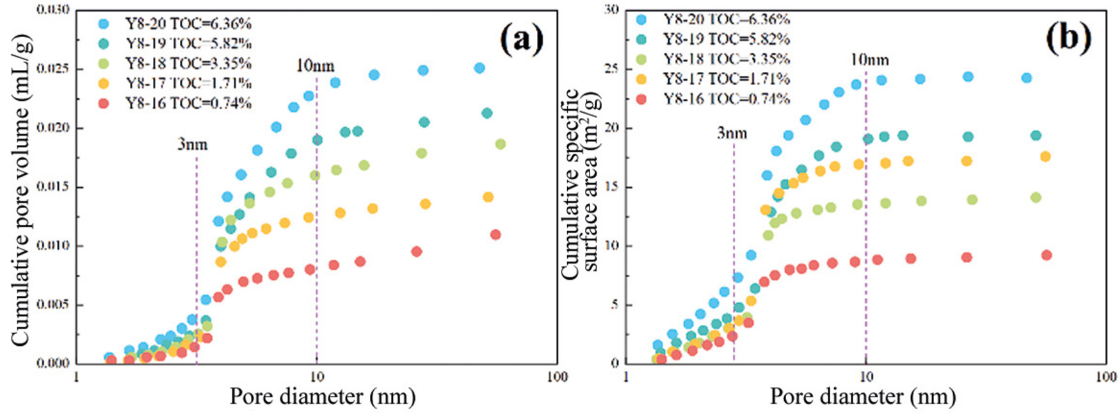

Fig. 4. Cumulative pore volume and specific surface area of black shale of Wufeng Formation-Longmaxi Formation in the study area. a) Cumulative pore volume; b) Cumulative specific surface area

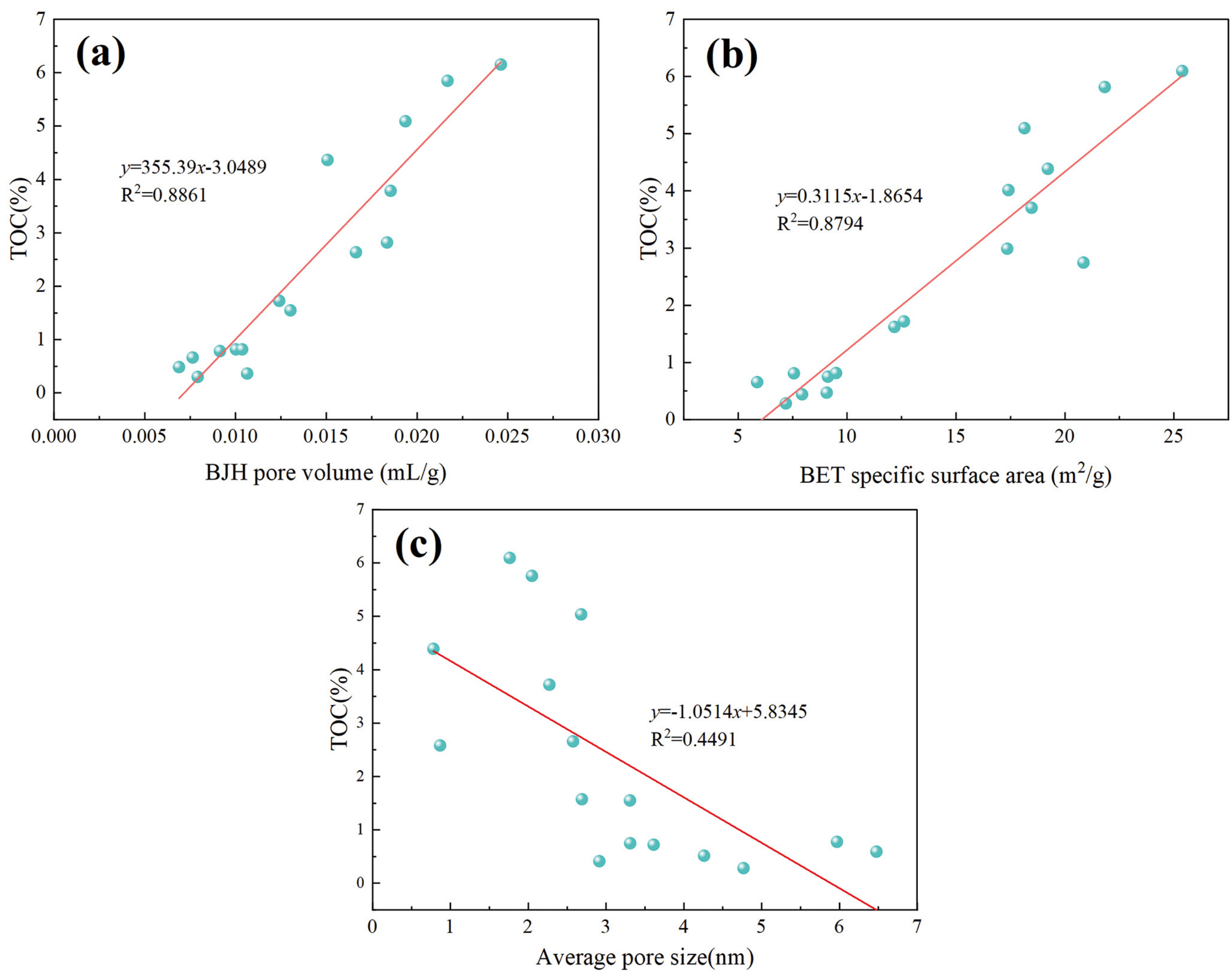

Fig. 5. Relationship between pore volume, specific surface area and average pore diameter and TOC of black shale of Wufeng Formation-Longmaxi Formation in the study area. a) Pore volume; b) Specific surface area; c) Average pore diameter

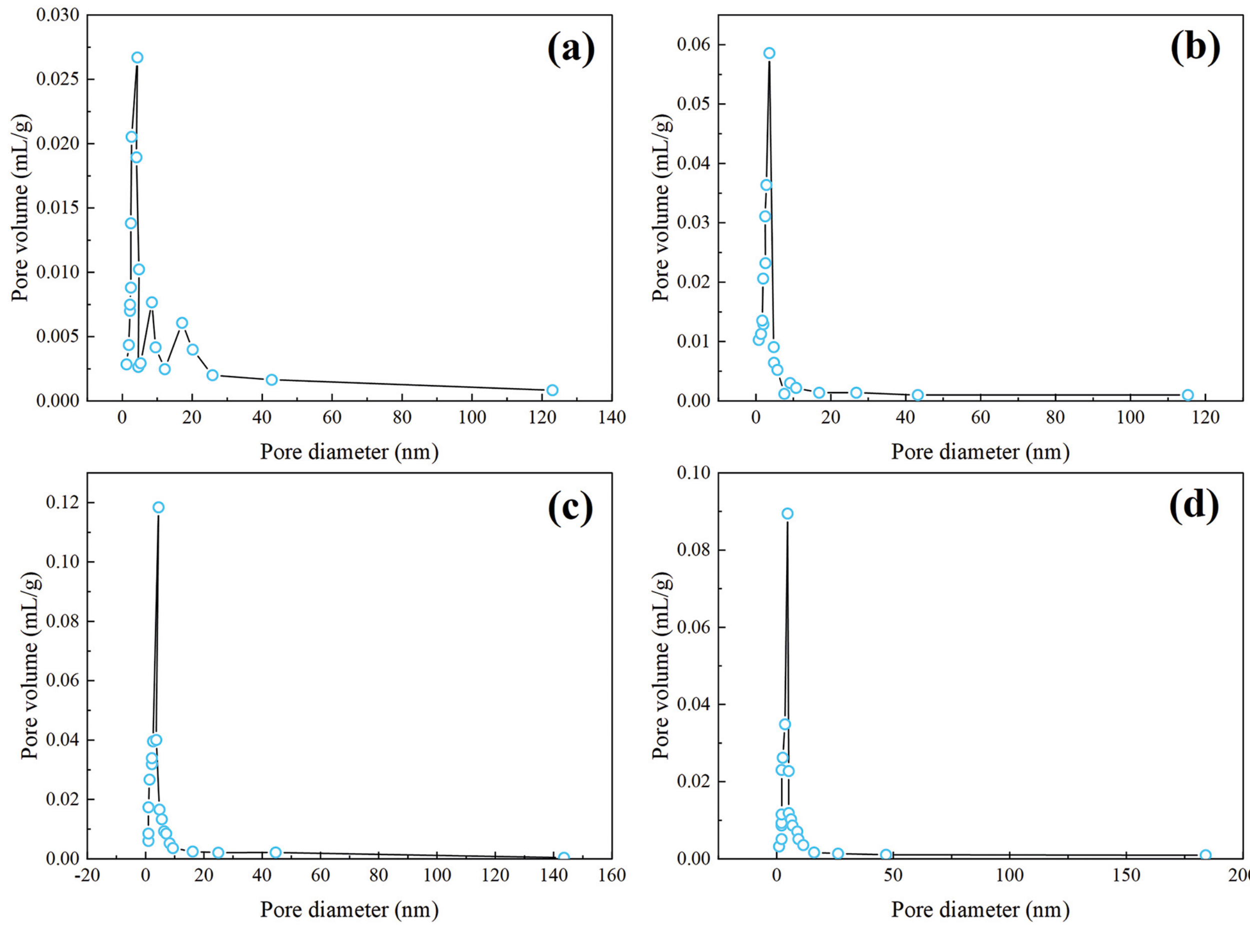

Fig. 6. Characteristics of pore size distribution of black shale of Wufeng Formation-Longmaxi Formation in the study area. a) Y9-4; b) Y9-6; c) Y9-8; d) Y9-13

3.3.3. Pore size distribution characteristics

The mercuric pressure method can determine pores with a pore size larger than 2 nm and can be used to characterize partial meso-macropores. In this paper, the maximum pressure applied is 50 MPa, which corresponds to a pore radius of about 26 nm, and can test macropores as well as some mesopores. At a pressure of 50 MPa, the saturation value of pressed mercury is 20-30%. The overall performance of the sample mercury pressure curve is low in mercury saturation and low in mercury withdrawal rate, and the proportion of nanoscale pores is around 90.5% (**Figure 7**). According to the mercury pressing experiments, it can be seen that the pore volume of macropores mainly comes from the pores with a diameter of 50-1000 nm (Fig. 7): the percentage of shale macropores volume to the total pore volume (i.e., the injected mercury saturation corresponding to the capillary radius of 25 nm) ranged from 11.2-18.3% (**Figure 8**).

3.4. Reservoir physical controls

3.4.1. Organic carbon content

In this paper, the correlation analysis of organic carbon content and clay mineral content with BJH pore volume and BET specific surface area of black shale samples from the Wufeng Formation-Longmaxi Formation collected from drilling wells in the study area revealed that organic carbon content has a strong positive correlation with BJH pore volume and BET specific surface area, with the correlation coefficients of 0.77 and 0.84, respectively (**Figure 9a, b**), and the correlation coefficient between the clay minerals and its BJH pore volume and BET specific surface area had little correlation (Fig. 9c, d). This suggests that organic matter pores are an important source of shale specific surface area and pore volume, which is also the same as previous

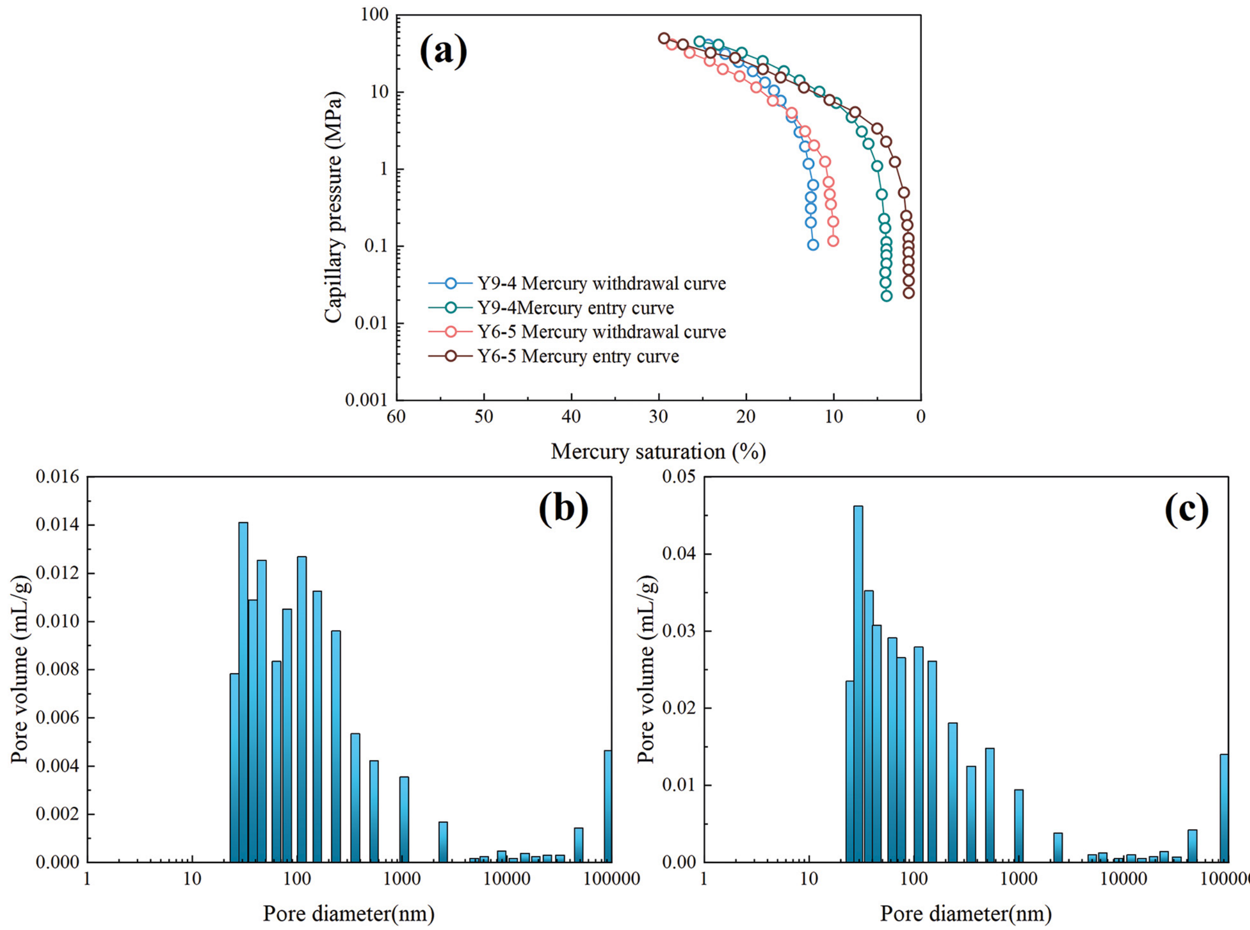

Fig. 7. Characteristics of mercury compression curves and pore size distribution of black shale of the Wufeng Formation-Longmaxi Formation in the study area. a) Mercury compression curves; b) Y6-5 pore size distribution; c) Y9-4 pore size distribution

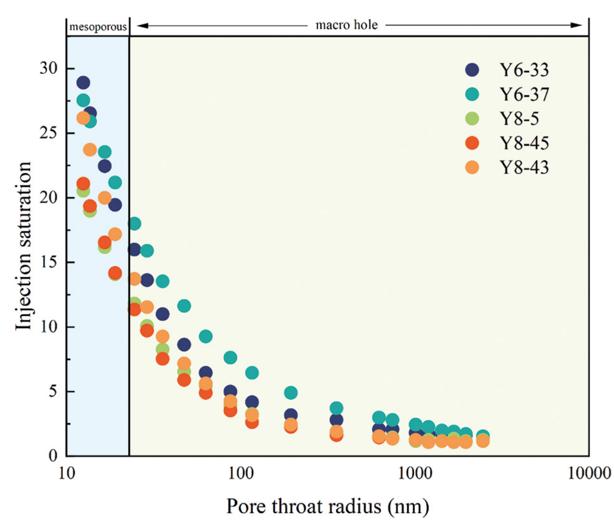

Fig. 8. Relationship between black injected Hg saturation and pore throat radius of Wufeng Formation-Longmaxi Formation in the study area

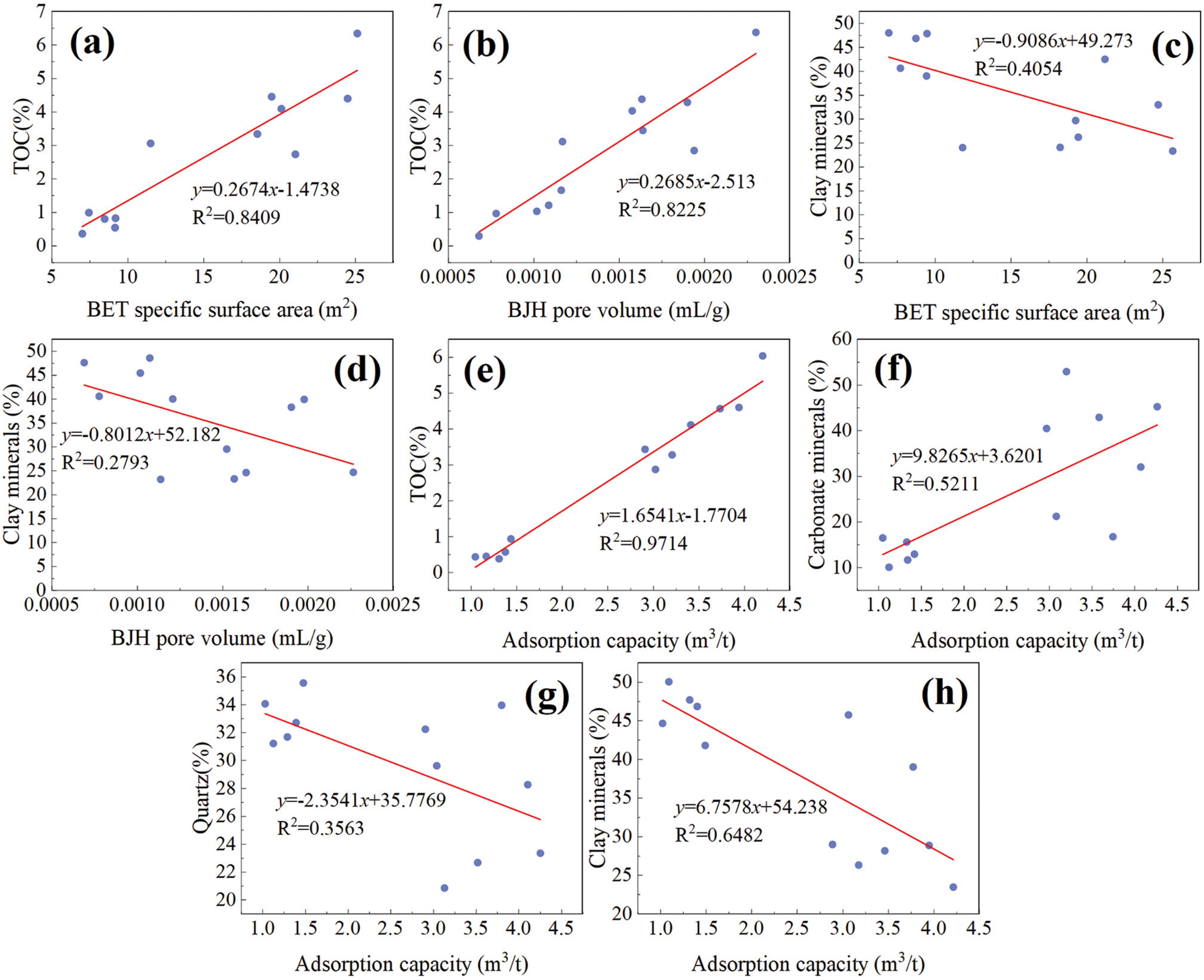

Fig. 9. Correlation analysis of organic carbon content, mineral composition and methane adsorption of black shale from Wufeng Formation-Longmaxi Formation in the study area. a) Organic carbon content vs. specific surface area; b) Organic carbon content vs. pore volume; c) Clay mineral content vs. specific surface area; d) Clay mineral content vs. pore volume; e) Methane adsorption vs. TOC; f) Methane adsorption vs. carbonate mineral content; g) Methane adsorption vs. quartz content; h) methane adsorption vs. clay mineral content

findings. Methane adsorption experiments (maximum pressure 10.83 MPa, temperature 30°C) show that methane adsorption is positively proportional to organic carbon content (Fig. 9e), with a correlation coefficient as high as 0.93: the correlation is poorer with carbonate minerals, quartz, and clay minerals (Fig. 9f, g, h), which suggests that the organic matter has a stronger adsorption capacity of methane, whereas the inorganic minerals are poorer at adsorption of methane. Therefore, the adsorbed gas in the black shale of the Wufeng Formation-Longmaxi Formation in the study area is mainly related to organic matter and is less affected by inorganic minerals.

3.4.2. Petrography

In the study of black shale from the Wufeng Formation-Longmaxi Formation, the brittle mineral content has a significant effect on the brittleness, pore development, fracture formation and fracturing effect of the rock. Quartz and carbonate minerals are the main brittle minerals, and higher content means better rock brittleness and more favorable for fracturing. Conversely, rock phases with high clay mineral content are less brittle [28]. Clay fine-grained lithologies with less than 50% of brittle minerals are the least brittle, while biosilica fine-grained lithologies and carbonate fine-grained lithologies with more than 50% of brittle minerals are better brittle.

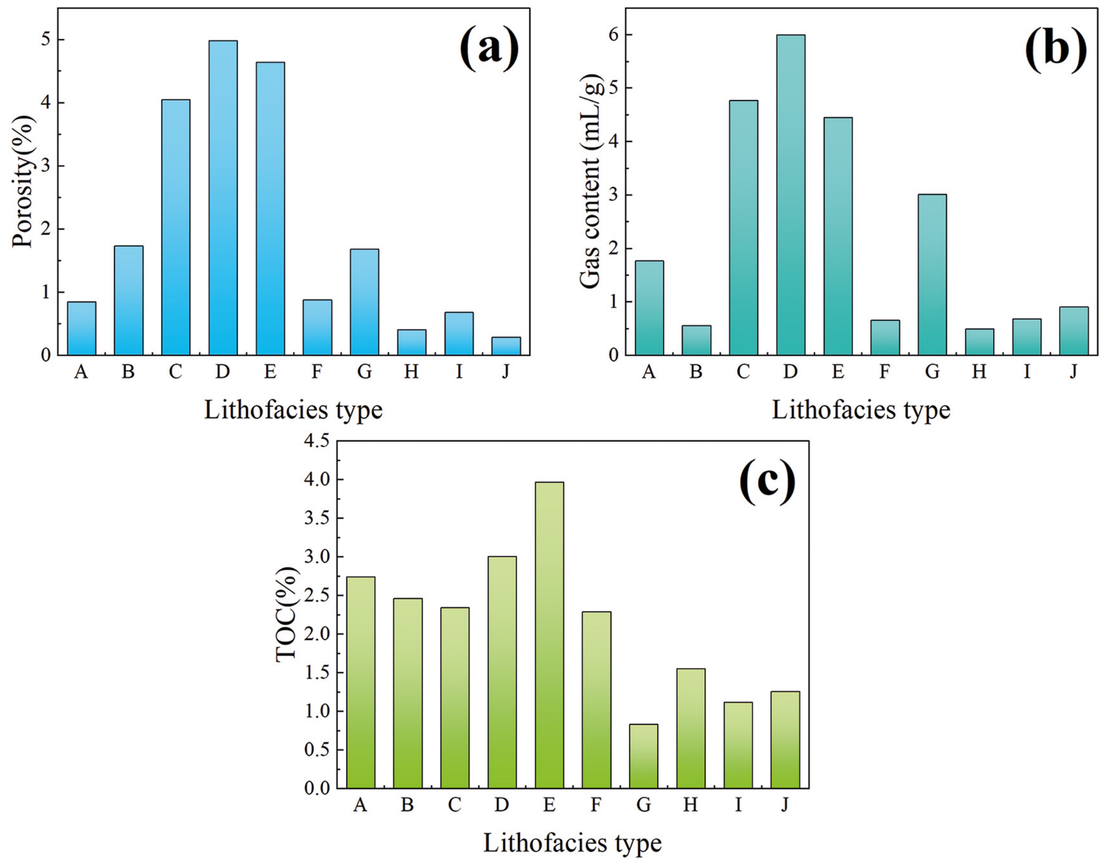

Fig. 10. Comparison of porosity, measured gas content and TOC of each lithofacies of Wufeng Formation-Longmaxi Formation in the study area. a) Lithofacies porosity; b) Measured gas content; c) TOC

Figure 10 shows the differences in physical properties and gas content of different lithologies. The poor physical properties and gas content of carbonate mixed fine-grained rock and clay mixed fine-grained rock are mainly due to the easy dissolution of carbonate minerals to block the pore space, and the clay minerals are greatly affected by compaction. On the other hand, Fig. 10c shows that the chalky carbonate fine-grained rock has well developed pores and moderate physical properties and gas content due to the richness of land-sourced detrital quartz and pulverized crystalline dolomite.

Although the organic carbon content of some lithologies is similar, such as clay-mixed fine-grained rocks, carbonate-mixed fine-grained rocks, biosilica-mixed fine-grained rocks, and clayey biosilica-fine-grained rocks, their physical properties and gas content differ significantly. The better gas content of biosilica-mixed fine-grained rocks and clayey biosilica-fine-grained rocks is mainly attributed to free gas in the inorganic pores, which is a key factor in their excellent gas content. The biogenic quartz in these lithologies was formed during the early stages of diagenesis and acts in conjunction with microbial-originated dolomite to resist compaction, protect the pore space, and improve physical properties. Among them, A-J in Fig. 10 respectively correspond to: A – carbonate mixed fine-grained rock, B – clay mixed fine-grained rock, C – bio-silica mixed fine-grained rock, D – clay bio-silica fine-grained rock, E – Biosilica fine-grained rock, F – biosiliceous clay fine-grained rock, G – silty carbonate fine-grained rock, H – clay fine-grained rock, I – carbonate fine-grained rock, J – clay carbonate Salt fine-grained rock.

Therefore, the comprehensive analysis concludes that: biosilica mixed fine-grained rock, biosilica fine-grained rock and clayey biosilica fine-grained rock with high porosity and high free gas content are the most favorable shale gas reservoirs in the study area, followed by chalky carbonate fine-grained rock.

3.4.3. Diagenesis

The black shale clay mineral of the Wufeng Formation-Longmaxi Formation in the study area consists of illite, ilmenite and chlorite. The content of illite is as high as 57%, and the montmorillonite mixing ratio in the ilmenite mixing layer is less than

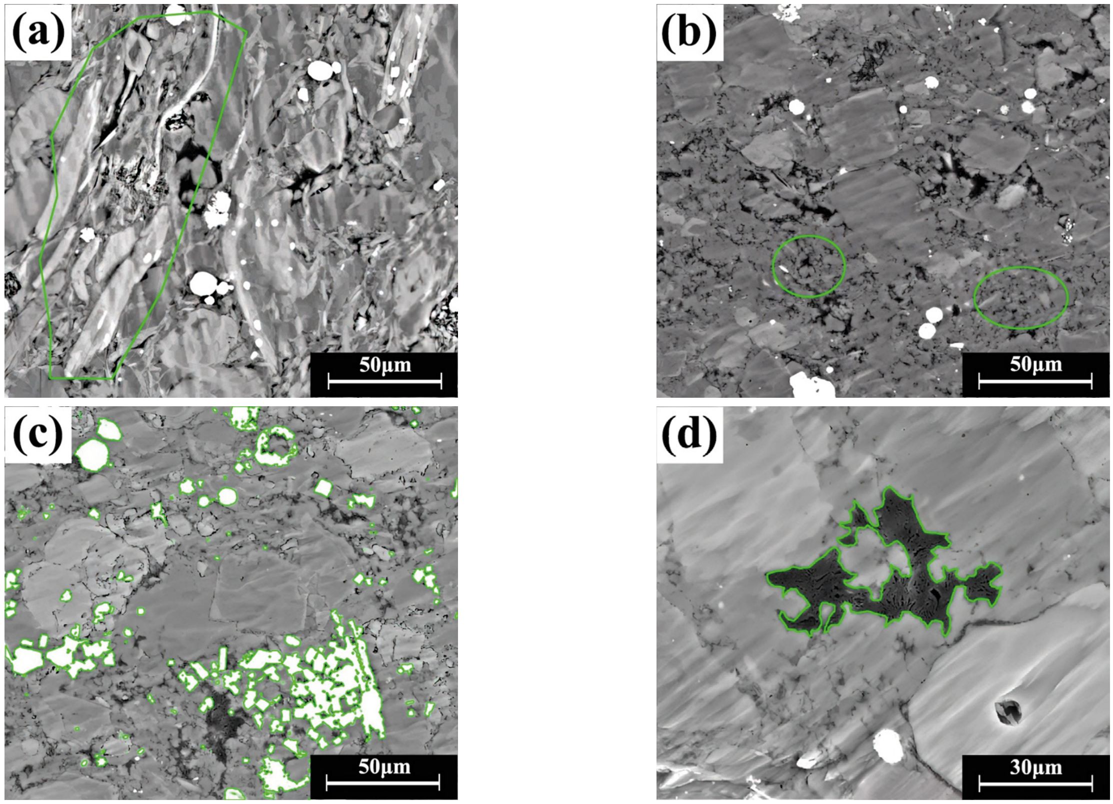

Fig. 11. Characteristics of diagenesis of black shale of Wufeng Formation-Longmaxi Formation in the study area. a) Lineament contact relationship between feldspar, quartz and clay minerals; b) Siliceous cementation in black siliceous shale; c) Dissolution pores of late diagenetic carbonatite minerals in black calcareous shale; d) Organic matter pores subjected to stress in a flattened form

10%, which indicates that it has entered the middle orogenic stage B. The Wufeng-Longmaxi Formation clay minerals in the study area are composed of illite and chlorite. The black organic-rich shale of the Wufeng-Longmaxi Formation in the Sichuan Basin and its peripheral Upper Yangzi region experienced deep burial and strong diagenesis, including compaction, cementation, dissolution, and thermal evolution of organic matter, which had an important impact on the formation and characterization of shale pores, and the results are shown in **Figure 11**.

Compaction. It mainly occurred at burial depths of less than 3000 m, resulting in densification of the mudstone and reduction of pore space. However, in the Sichuan Basin and its periphery, the paleoburial depth of the Wufeng-Longmaxi Formation generally exceeded 6500 m, and thus experienced strong compaction, preserving only the original porosity between rigid grains (quartz and carbonate grains) (Fig. 11a).

Cementation. The main manifestation is chemical cementation, mainly calcareous cementation and siliceous cementation, which reduces the intergranular pores and deteriorates the connectivity. The strength of cementation varies in shales of different lithologies, and calcium cementation is usually stronger than silica cementation (Fig. 11b).

Dissolution. It occurs mainly in the early stages of diagenesis, and as diagenesis progresses, the shale becomes denser and the pore fluid decreases, making dissolution difficult. Rock rupture in late diagenesis may also cause dissolution and the formation of dissolution pore spaces (Fig. 11c).

Thermal evolution of organic matter. Organic matter pores formed by thermal evolution of organic matter are important pore types, which are mainly formed by hydrocarbon production from mature casein. The black shales of the Wufeng-Longmaxi Formation in the Sichuan Basin have reached the overmature stage (Ro of 2.2%-3.5%), and thus a large number of organic matter pores have been formed (Fig. 11d).

Together, these diagenetic effects determine the pore characteristics of the black shales of the Wufeng-Longmaxi Formation and the quality of the shale gas reservoirs in the Sichuan Basin and its periphery.

4. Conclusion

The study reveals the complexity of mineral compositions in the shale reservoirs of the Longmaxi Formation in the east Sichuan Basin, mainly consisting of clay minerals such as illite, illite-mixed and chlorite, accompanied by significant content of brittle minerals such as quartz and calcite. The longitudinal distribution of clay minerals shows six basic patterns, reflecting the significant influence of diagenesis on pore development. This finding emphasizes the important role of diagenesis in shale pore formation and characterization.

The results of gas adsorption experiments and mercury pressure tests show that the pore volume and specific surface area of the Longmaxi Formation shales in the Chuandong area of the Sichuan Basin are significantly positively correlated with the organic carbon content. Organic matter pores are the main source of shale specific surface area and pore volume, and their average pore size is inversely proportional to organic carbon content, indicating that organic matter pore diameters are generally smaller than mineral-related pores. These findings provide an important quantitative basis for understanding the pore characteristics of shale gas reservoirs.

The study emphasizes that the brittle mineral content has an important influence on the physical properties of shale reservoirs in the Longmaxi Formation in the east Sichuan Basin, especially on the brittleness of the rock, pore development, fracture formation and fracturing effect. In addition, reservoir physical properties are jointly regulated by petrographic properties and diagenesis, which determine the characteristics and development potential of shale gas reservoirs. Therefore, brittle minerals and organic matter content are the key controlling factors of shale gas reservoir physical properties.

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