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Research Article

Reservoir Characteristics and Gas Content of Black Shale of Wufeng-Longmaxi Formation in Qianjiang Area, Southeast Chongqing, China

Bin Wu

Chongqing Industry Polytechnic College, Chongqing 401120, China

Correspondence should be addressed to Bin Wu; wubin@cqipc.edu.cn

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The shale gas exploration in Changning, Weiyuan, Fuling Jiaoshiba, and other areas has achieved great success in the Sichuan Basin, and the basin margin of the Sichuan Basin will be an important target in the future. In view of the lack of understanding of shale gas reservoir characteristics of the basin margin area of the Sichuan Basin, this paper conducts a study on reservoir characteristics and gas-bearing properties of this area using qualitative and quantitative techniques. The results show that the thickness of the black shale reservoir in the Qianjiang area is up to 33 m, the kerogen sapropelic type (I) is dominant, and Ro ranges from 2.2 wt% to 3.39 wt%. The mineral composition is relatively complex, mainly composed of quartz and clay minerals. Quartz content is in the range between 9.4 wt% and 79 wt% (average = 47.48 wt%), while clay content is in the range between 10.02 wt% and 43.6 wt% (average = 29.41 wt%). There are various types of reservoir spaces, mainly including organic pores dominated by matrix pores, intergranular and intragranular pores, intergranular and intragranular dissolution pores, secondary dissolution pores, and microfractures, among which organic pores are the main pore types. The pore volume of nanopores is 0.009897-0.017177 cm^3/g (average = 0.01304 cm^3/g), and the specific surface area ranges from 12.1 m^2/g to 35.1 m^2/g (average = 20.47 m^2/g). Micropores and mesopores are the main pore sizes for the development of organic pores. Quartz particles promote the development of reservoir space, while clay minerals are not conducive to the development of pores. The total gas content ranges from 0.5 m^3/t to 4.43 m^3/t (average = 1.69 m^3/t). Its enrichment is controlled by many factors. The increase of organic matter abundance is the material basis for shale gas enrichment. The development of organic pores provides high-quality storage space for shale gas preservation. The development of different mineral components restricts the enrichment of shale gas.

1. Introduction

As a clean and efficient natural gas resource, shale gas is favored by major oil and gas resource countries in the world and has achieved success in the United States, Canada, China, and other countries. In 2021, the annual output of shale gas in the United States reached 7721×10^4 m^3, accounting for 80% of the total natural gas output of the year, which has become the main force of natural gas supply in the United States. However, China's shale gas exploration started late, and the annual output of shale gas in 2021 also reached 230×10^4 m^3 [1–4], which is relatively backward in development. At present, the exploration and development of marine shale gas in China has made breakthroughs in Changning, Weiyuan, Fuling, Zhaotong, and other regions in the Sichuan Basin, establishing 10 billion shale gas fields [5, 6]. However, no substantial progress has been made in the areas around the Sichuan Basin that are largely covered with black organic shale, and only low-yielding shale gas wells have been obtained in some areas, such as well PY1 in Pengshui and well NY2 in Wulong [7–9]. It is necessary to further study the geological theory of shale gas in view of these substantive issues. Therefore, in view of the lack of understanding of shale gas reservoir characteristics of the Wufeng-Longmaxi Formation in the Qianjiang area, southeast Chongqing, this paper carried out a study on

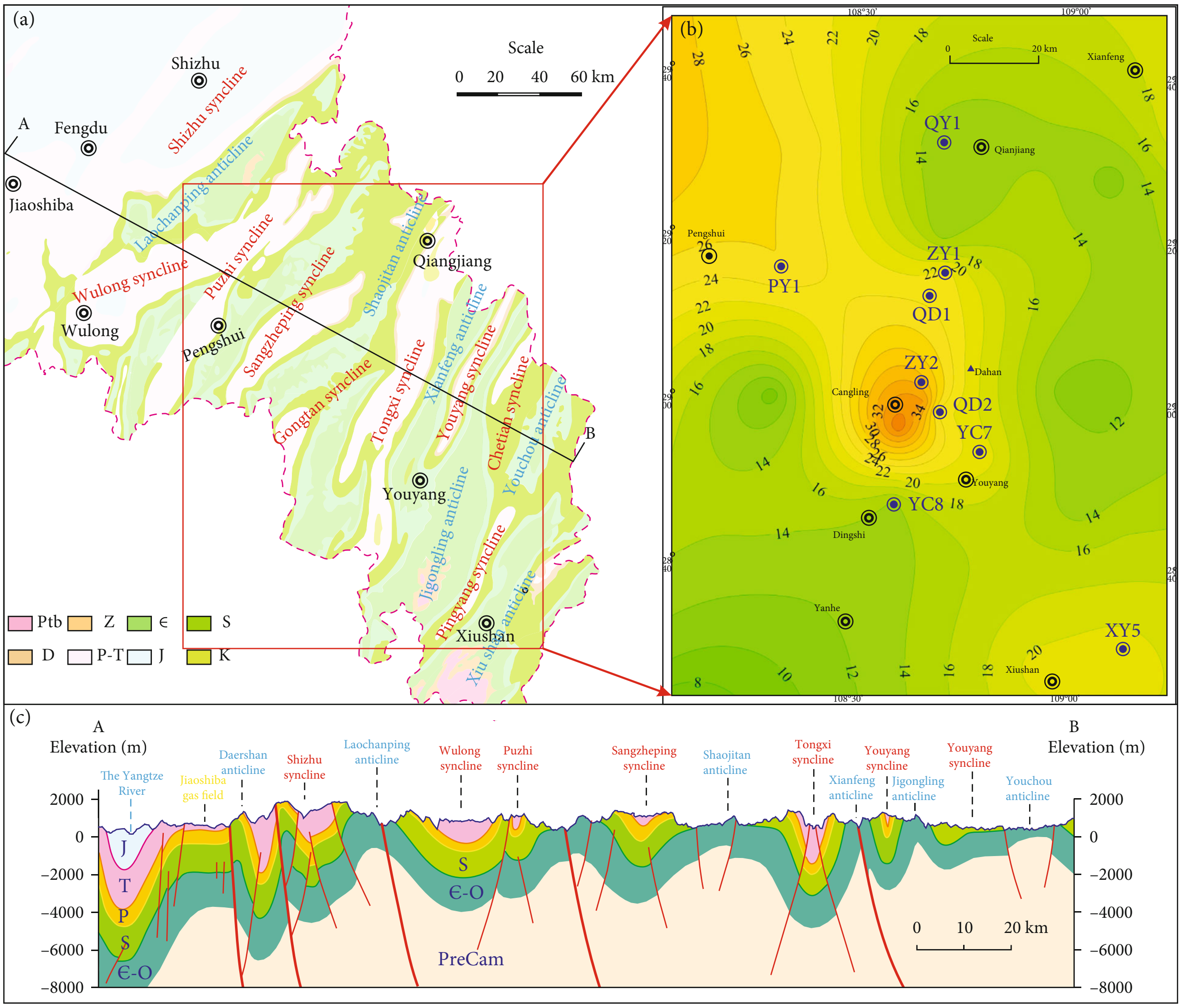


Figure 1: (a) Tectonic geological map of southeast Chongqing. (b) Thickness distribution map of high-quality shale in Wufeng-Longmaxi Formation, Qianjiang area. (c) Profile of tectonic in southeastern Chongqing.

the characteristics and enrichment laws of shale gas reservoirs in the basin margin area, such as the difficulty of selecting favorable areas and the unknown exploration potential due to the strong heterogeneity of gas content and the unknown influencing factors. It is hoped to provide beneficial geological theory support for shale gas exploration and development in the basin margin area.

2. Regional Geological Setting

The study area is located in the southeast of Chongqing. The west side is restricted by the Qiyao Mountain, the east side is restricted by the Xuefeng Mountain, and the south side is also affected by the Qianzhong Uplift. The fission track shows that [10–13], after the tectonic movements of Jinning, Chengjiang, Caledonian, Variscan, Indochina, Yanshan, Xishan, etc., the area has formed a series of NE and NNE asymmetric anticlinal fold belts and associated faults, and the structural deformation is very complex. Several third-order structural units are developed, including the Sangzheping syncline, Tongmayuan anticline, Tongxi syncline, the northern end of the Xianfeng anticline, Youyang syncline, Jigongling anticline, Youchou anticline, etc. The anticlinal core is mainly exposed to the Cambrian system, and the synclinal core is mainly the Middle Triassic. The anticline-synclinal wing is often developed with "Y-shaped" conjugate inverse faults [14, 15]. Most of the main faults are deep to the basement and tend to be SE. The Qianjiang area shows a typical trussed or transitional fold anticline with a narrow syncline, wide anticline, or equal width between the anticline and syncline (Figures 1(a) and 1(c)).

During the Late Ordovician Wufeng-Early Silurian Longmaxi sedimentation period, affected by the Chuanzhong-Qianzhong-Xuefeng Mountain uplift, the Sichuan Basin was a restricted shallow sea shelf sedimentary environment [16–23]. The sedimentary water body gradually became shallow from the Fuling Jiaoshiba in the basin to the southeast of Chongqing in the basin margin. The black shale sedimentary environment gradually changed from the deep water shelf to the shallow water shelf. The Pengshui-Cangling area belongs to the deep water shelf, and the Qianjiang area is located in the transition area from the shallow water shelf to the deep water shelf; the east of the Youyang-Xiushan area is located in the shallow water shelf, especially the Youyang East and Xiushan areas which are significantly affected by the Xuefeng provenance area, with high black shale sand content. Controlled by the influence of the sedimentary environment, the thickness of black high-quality shale (TOC > 2%) deposited in the Wufeng Formation and the early Longmaxi Formation in southeast Chongqing is NW-SE. Compared with the Jiaoshiba area in the basin, the thickness of black high-quality shale in southeast Chongqing is reduced, and only the Pengshui-Cangling area is relatively thicker, generally greater than 23 m (Figure 1(b)).

3. Characteristics of Black Shale Reservoir

3.1. Samples and Experimental Test Method. This sample is mainly collected from the Qianjiang shale gas block in Chongqing, and all sample collections are strictly in accordance with the oil and gas industry standards. The cores of well ZY1, well QD1, well ZY2, and well QD2 were collected for analysis and research; 105 samples were collected; and XRD, kerogen type, TOC, Ro, FIB-SEM, isothermal adsorption, gas content test, and porosity and permeability analyses were carried out. After all the samples were collected, they were sent to the experimental center of East China Exploration and Development Research Institute of Sinopec for testing in strict accordance with the shale gas sample analysis specifications.

3.2. Mineralogy. Shale gas belongs to a "continuous" gas reservoir [24]. Its gas components mainly exist in black shale with the form of adsorption, free, or dissolution [24–26]. The enrichment degree of shale gas is closely related to the mineral composition. It is particularly important to study the brittle mineral content and clay mineral composition of black shale. This time, the mineral composition and content of black shale in the Wufeng-Longmaxi Formation are quantitatively analyzed by means of X-ray diffraction, thin-section microscope, and scanning electron microscope. The mineral composition is relatively complex, mainly composed of clay minerals, quartz (Figures 2(a) and 2(c)), feldspar (Figure 2(a)), calcite (Figure 2(b)), dolomite, etc., with a small amount of pyrite (Figure 2(d)). The clay minerals are mainly composed of illite, illite and montmorillonite-mixed layer, and chlorite.

3.2.1. Characteristics of Brittle Minerals. According to the X-ray diffraction analysis, the content of brittle minerals in the study area is relatively high, ranging from 40.8 wt% to 88.7 wt% (average = 67.02 wt%). The siliceous quartz is the main mineral type, ranging from 9.4 wt% to 79 wt% (average = 47.48 wt%), followed by potassium feldspar and plagioclase, with an average content of 12.82%. The content of carbonate minerals is generally very low, with an average of 6.7%, and the content of other minerals is relatively low, less than 5%. Vertically, the black shale of the Wufeng Formation is mainly siliceous shale, with a relatively high siliceous content, greater than 40%, increasing gradually from bottom to top. The siliceous content of the Longmaxi Formation is more than 25%, increasing gradually from bottom to top (Figure 3). According to the test results of TOC, the high-quality shale section in the Wufeng-Longmaxi Formation has a high content of brittle minerals (TOC > 2 wt%), ranging from 47.5 wt% to 87.9 wt% (average = 59.19 wt%). The average content of authigenic brittle minerals is 11.72%, with good compressibility, which is conducive to the implementation of hydraulic fracturing and the formation of the fracture network. The content of brittle minerals in ZY1, ZY2, QD1, and QD2 wells in the area shows good consistency. The content of authigenic brittle minerals is stable. The data points in the triangle map are close to the end of clastic minerals (Figure 4(a)). When the TOC increases, the content of clastic minerals increases to a certain extent. The data points in the triangle map are close to the end of clastic minerals (Figure 4(b)).

3.2.2. Characteristics of Clay Minerals. The composition of clay minerals in the study area is stable, mainly illite and an illite and montmorillonite-mixed layer. The clay mineral content is relatively low, ranging from 10.02 wt% to 43.6 wt% (average = 29.41 wt%). Its components are mainly illite, ranging from 53 wt% to 80 wt% (average = 69.7 wt%), followed by an illite and montmorillonite-mixed layer, with an average of 23%. The content of chlorite is small, generally less than 10% (Figure 2). In the Wufeng-Longmaxi Formation high-quality shale section (TOC > 2%), clay mineral content is in the range between 13.4 wt% and 33.9 wt% (average = 28.89 wt%) (Figure 3).

According to the above quantitative analysis of mineral composition, the black shale in the area has good reservoir-fracturing quality. The reservoir-fracturing brittleness index of the high-quality black shale section in the area ranges from 40 wt% to 75 wt% (average = 58.5 wt%), which is far higher than the hydraulic-fracturing requirement of the brittle mineral index in North America which must be greater than 30% [27, 28]. At the same time, TOC content of the high-quality black shale section ranges from 2 wt% to 6.9 wt% (average = 4.5 wt%). The abundance of organic matter is high, which is conducive to the occurrence of shale gas. Therefore, the high-quality black shale reservoir segment in the study area has higher reservoir quality and fracturing quality, which is a favorable target segment for hydraulic fracturing.

3.2.3. Lithofacies Division and Characteristics. The study of the material composition and lithofacies of black shale is conducive to the development of shale gas. Previous studies have shown that shale lithofacies exercise important controls on the reserve characteristic of gas shale [29]. The study of mineral composition shows that the minerals in the lower part of the Longmaxi Formation are mainly siliceous, with relatively low clay mineral content and high organic carbon content (Figure 4). Referring to the lithofacies classification scheme of Yang et al. [29], the study area can be divided into siliceous shale lithofacies (siliceous content > 60 wt%), argillaceous/siliceous mixed shale

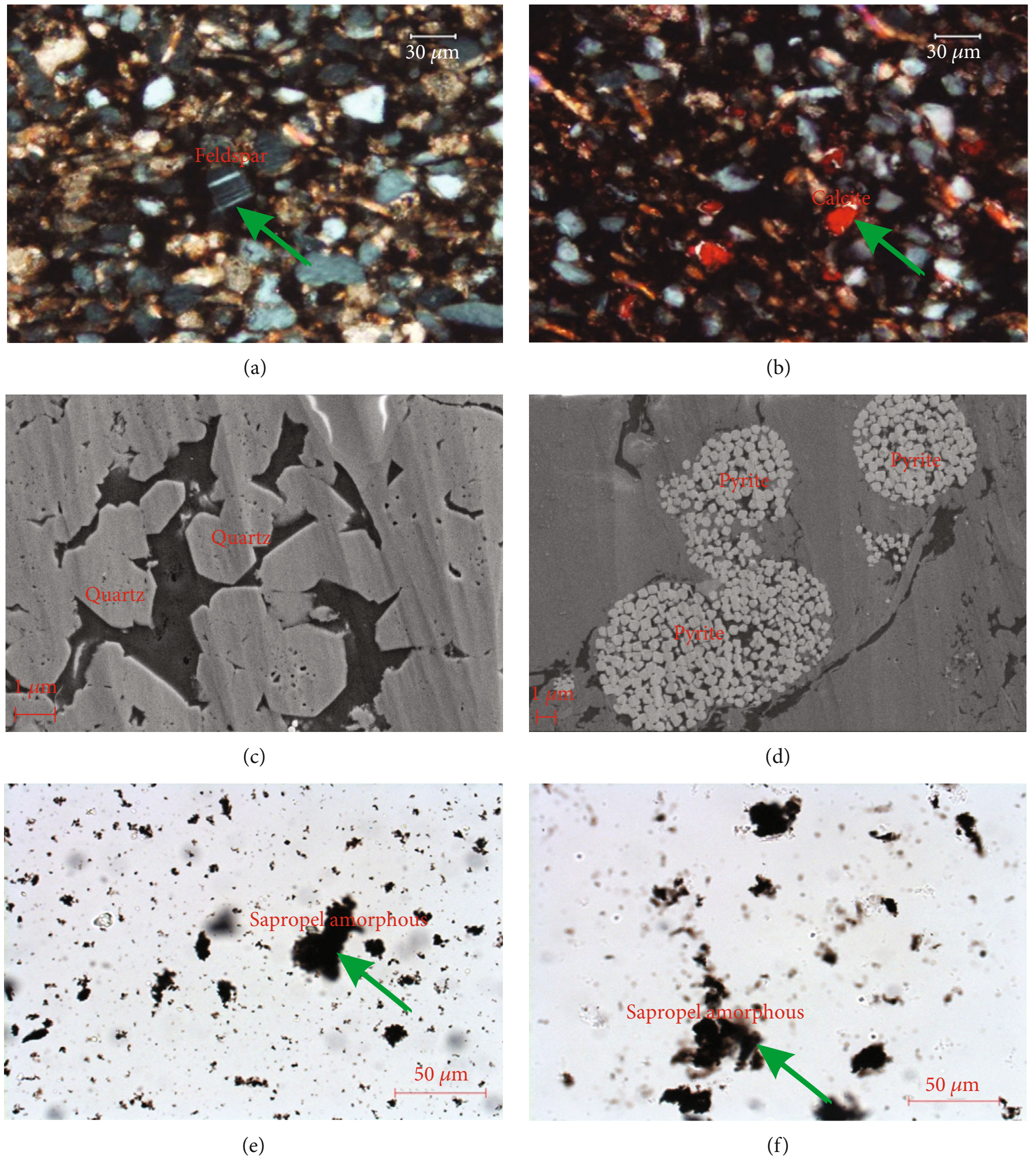


Figure 2: Mineralogical characteristics of black shale in Wufeng-Longmaxi Formation. (a) Well QD1, 1098.92 m, feldspar; (b) well QD1, 1101.38 m, calcite; (c) well ZY1, 2620.01 m, quartz; (d) well ZY1, 2622.07 m, moldy spherical pyrite; (e) well ZY1, 2618.01 m, sapropel amorphous; (f) well ZY1, 2616.81 m, sapropel amorphous.

lithofacies (siliceous, calcareous, argillaceous < 60 wt%), and silicon-rich argillaceous shale lithofacies (argillaceous content less than 60 wt%).

The siliceous shale lithofacies is mainly located at the top of the Wufeng Formation and the bottom of the Longmaxi Formation and is the main reservoir of shale gas. Silica content > 60 wt%, argillaceous and calcareous content < 20 wt%, TOC > 3 wt%, gas content > 2 m^3/t, and organic pores are very developed. According to the development experience of shale gas in recent years, siliceous shale lithofacies is the preferred section for horizontal well drilling and fracturing. The shale/siliceous mixed shale lithofacies is mainly located at the bottom of the Wufeng Formation and the middle of the Longmaxi Formation, and the gas-bearing property of shale gas is poor. Silica content < 60 wt%, argillaceous content > 20 wt%, TOC ranges from 1 wt% to 3 wt%, gas content < 1 m^3/t, and organic pores are relatively developed. The silicon-rich argillaceous shale lithofacies are mainly located in the middle and upper parts of the Longmaxi Formation, which is the cap rock of shale gas enrichment. The argillaceous content is high, generally >40 wt%; the siliceous content ranges from 40 wt% to 60 wt%; and the TOC and gas content are poor, generally <1 wt%. In general, siliceous shale lithofacies is characterized by high siliceous content, high organic carbon content, good gas-bearing property, and well-developed organic pores and is the main preferred interval for shale gas development at present.

3.3. Organic Geochemical Characteristics. In this study, kerogen maceral analysis and scanning electron microscopy were used to determine the kerogen type. A large amount of

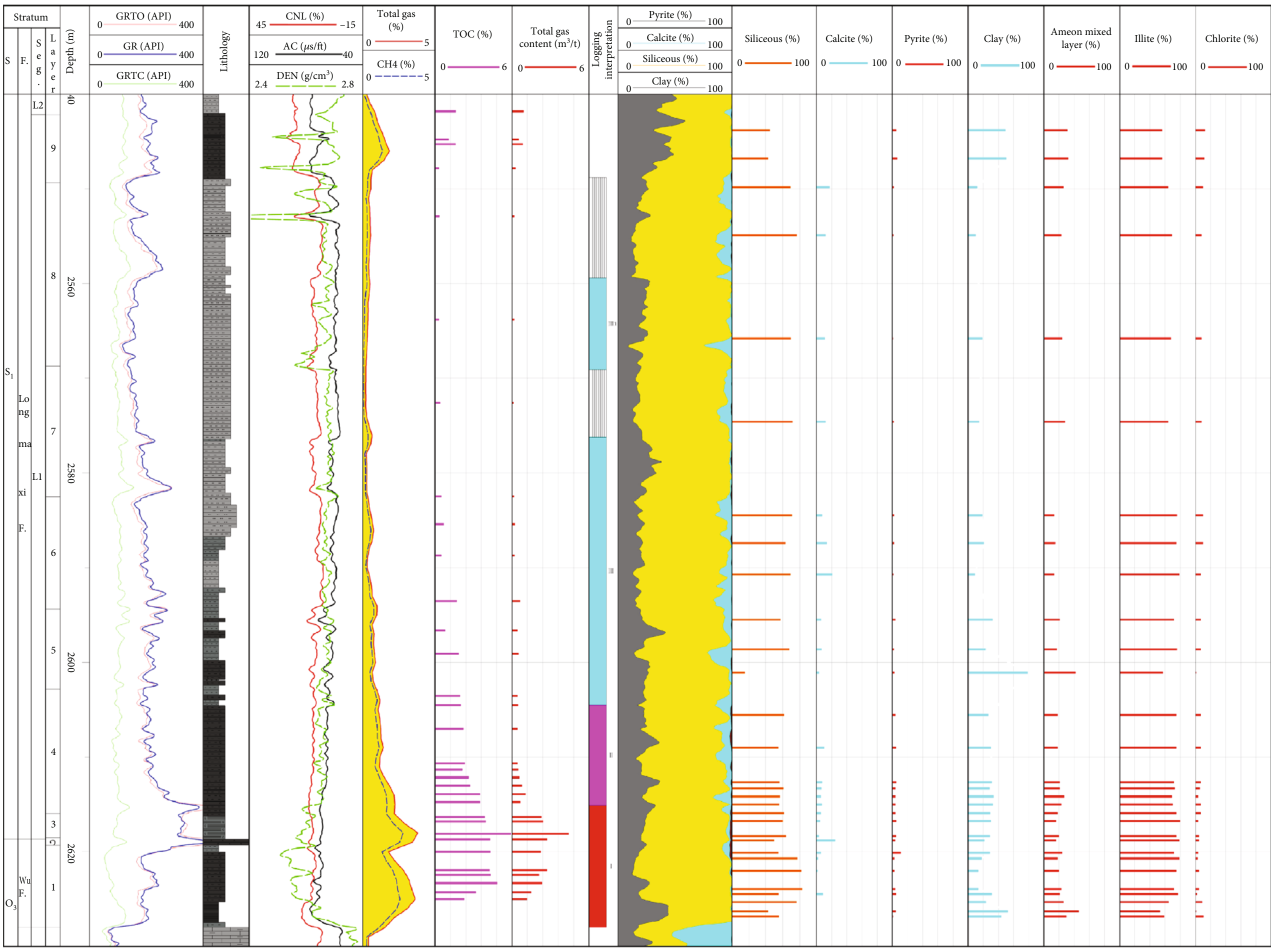
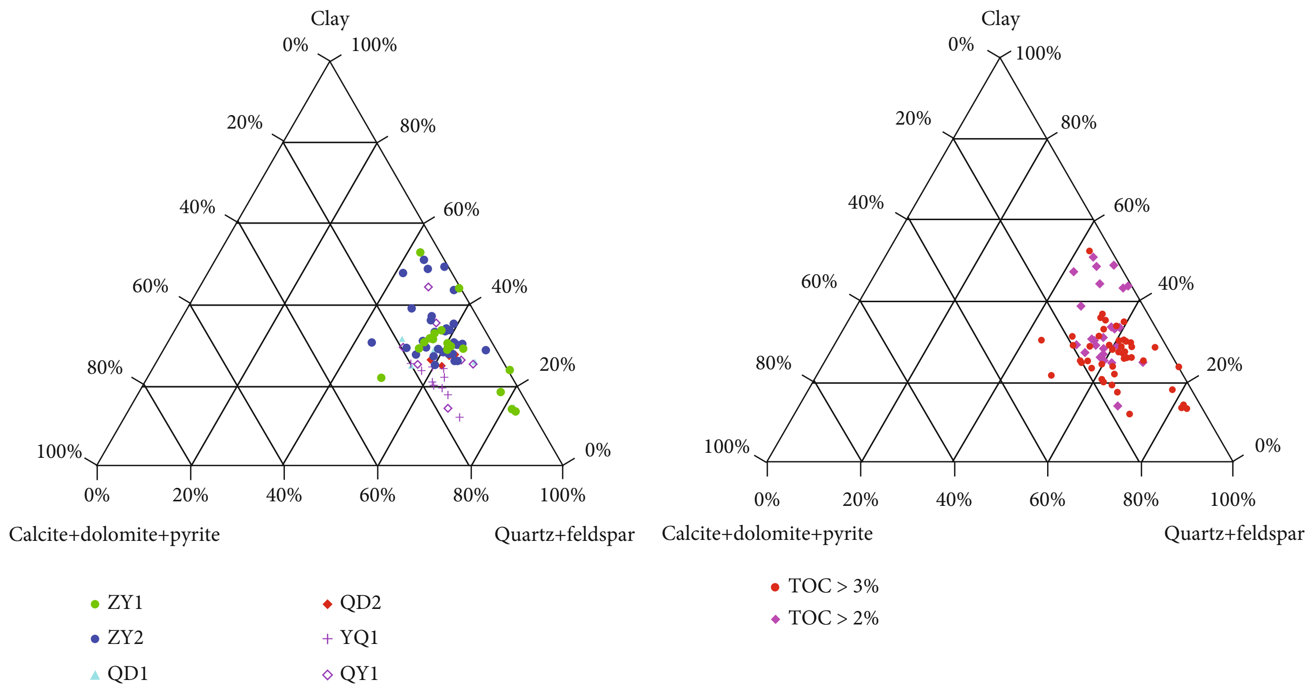


Figure 3: Comprehensive analysis of mineral composition of well ZY1 in Wufeng-Longmaxi Formation in Qianjiang area, southeast Chongqing.

sapropelite and a small amount of vitrinite, inertinite, and other organic macerals were detected in shale samples. The TI indicates that there are mainly sapropelite (I) and humic (II1) kerogen types in the Wufeng-Longmaxi Formation. Sapropelic type (I) accounts for more than 85% (Table 1). Kerogen microscopic examination mainly shows amorphous flocculent and granular aggregates (Figures 2(e) and 2(f)), indicating that the organic matter in the black shale of the Wufeng-Longmaxi Formation is type I, with a small amount of type II1, and the parent material is mainly from plankton, bacteria, and algae. The core, thin sections, and outcroppings of the black shale in Qianjiang also indicate that plankton graptolites are the main biological fossils of the Wufeng-Longmaxi Formation, and lower aquatic organisms such as siliceous sponge bone needles, brachiopods, cyanobacteria, globuletes, keratophytes, and ostracodes are also found. These lower aquatic plankton, algae, and animal bodies have laid a good material foundation for the enrichment of shale gas in this area, and the enrichment of organisms has also greatly improved the pore structure and porosity of organic matter, providing a good reservoir space for free gas.

Due to the lack of vitrinite in the Lower Paleozoic in the Sichuan Basin, it is difficult to obtain vitrinite reflectance; asphalt reflectance is used to characterize the thermal maturity of organic matter in the Wufeng-Longmaxi Formation [30–32]. The test results show that the thermal maturity of black shale samples in the study area is generally high, and the equivalent vitrinite reflectance (Ro) of most wells is above 2 wt% and range between 2.2 wt% and 3.39 wt% (average = 2.64 wt%) (Figure 5), reaching a highly mature evolution stage dominated by dry gas. The thermal evolution degree of organic matter is similar to the Haynesville shale gas in North America. The vitrinite reflectance of the Haynesville shale ranges from 2.2 wt% to 3.2 wt%, which is in the highly mature stage, and the average initial production is about $39.645 \times 10^4\ m^3/d$. At present, it has become the shale gas area with the highest shale gas production in North America. The vitrinite reflectance of well JY1 in the Jiaoshiba area ranges from 2.2 wt% to 3.13 wt% (average = 2.65 wt%), which indirectly reflects that the highly mature stage of organic-rich shale is not the most important factor restricting shale gas enrichment.

The black shale of the Wufeng-Longmaxi Formation in Qianjiang has the characteristics of high organic matter content and high maturity, and the thickness of the high-quality shale section (TOC > 2%) is relatively large, which is comparable with Pengshui. The minimum TOC value of the high-



(a) According to drilling statistics

(b) According to TOC statistics

FIGURE 4: Triangle diagram of mineral composition of black shale in Wufeng-Longmaxi Formation in Qianjiang area, southeast Chongqing.

TABLE 1: Statistics of lithofacies characteristics of black shale in Wufeng-Longmaxi Formation in Qianjiang area.

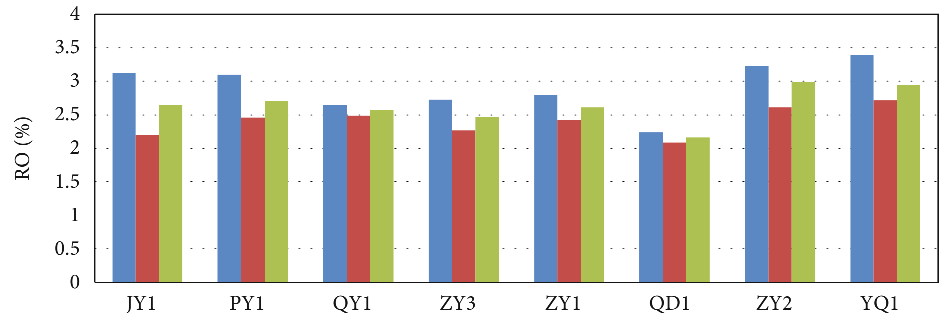
Well	Formation	Sapropelite (%)	Chitinite (%)	Vitrinite (%)	Inertinite (%)	TI	Organic matter type
ZY1	O_3w-S_1l	54.9~97.13 85.64 (7)	0	2.87~45.4 1.63 (7)	0	9.2~94.26 71.29 (7)	I-II_1
QD1	O_3w-S_1l	95~98 96.75 (7)	0	1~3 1.63 (7)	1~3 1.63 (7)	90.50~96.25 93.91 (7)	I
QD2	O_3w-S_1l	94~98 96.24 (17)	0	1~3 2.0 (17)	1~3 1.76 (17)	88.75~96.25 92.97 (17)	I
YC8	O_3w-S_1l	80~95 88.15 (13)	0	2~8 5.0 (13)	2~15 6.85 (13)	61.25~90.5 77.56 (13)	I-II_1

Note: when kerogen maceral analysis is used to classify kerogen types, weighted type index TI value is used to determine the kerogen types, namely, $TI = (a \times 100 + b \times 50 + c \times (-75) + d \times (-100))/100$, where a, b, c, and d represent the percentage of sapropelite, chitinite, vitrinite, and inertinite, respectively.

quality shale section of sublayers 1-5 ranges from 0.92 wt% to 2.35 wt%, the maximum ranges from 4.26 wt% to 5.91 wt%, and the average ranges from 2.72 wt% to 3.92 wt%. There is little difference in organic matter content in Jiaoshiba-Pengshui-Qianjiang, but there is a difference in the thickness of shale with organic carbon content greater than 2%. Vertically, the TOC of the high-quality black shale section shows a bimodal structure, with 2 wt%-3 wt% and 3.5 wt%-4.5% as the main distribution range, while 3 wt%-3.5 wt% and 4.5 wt%-6.0 wt% are relatively low. Horizontal comparison shows that the thickness of high-quality shale with TOC > 2% in ZY2 is 33 m, and the thickness of ZY1 is 24 m. When TOC > 3%, ZY1 is 15 m, ZY2 is 19.8 m, JY1 is 38 m, and PY1 is 19.7 m (Figure 6). In general, in the sedimentary environment of detention and anoxia, the Jiaoshiba of the deepwater shelf is conducive to the enrichment of black graptolite shale rich in organic matter. The black shale with TOC > 3% has a large sedimentary thickness, while the organic matter enrichment in the Pengshui to Qianjiang area is poor, and the black shale with TOC > 3% has a thin sedimentary thickness.

3.4. Pore Types and Structural Characteristics

3.4.1. Pore Types and Morphological Characteristics. As an unconventional gas reservoir, black shale has the characteristics of large resources and poor quality of reservoir space. Understanding the types and characteristics of its storage space is conducive to finding out the occurrence state of natural gas in the black shale reservoir and promoting the



	JY1	PY1	QY1	ZY3	ZY1	QD1	ZY2	YQ1
Max	3.13	3.1	2.65	2.73	2.79	2.24	3.23	3.39
Min	2.2	2.46	2.49	2.27	2.42	2.09	2.61	2.72
Average	2.65	2.71	2.57	2.47	2.61	2.16	2.99	2.95

FIGURE 5: Comparison histogram of average value of thermal maturity (Ro) of organic matter in Wufeng-Longmaxi Formation in southeast Chongqing.

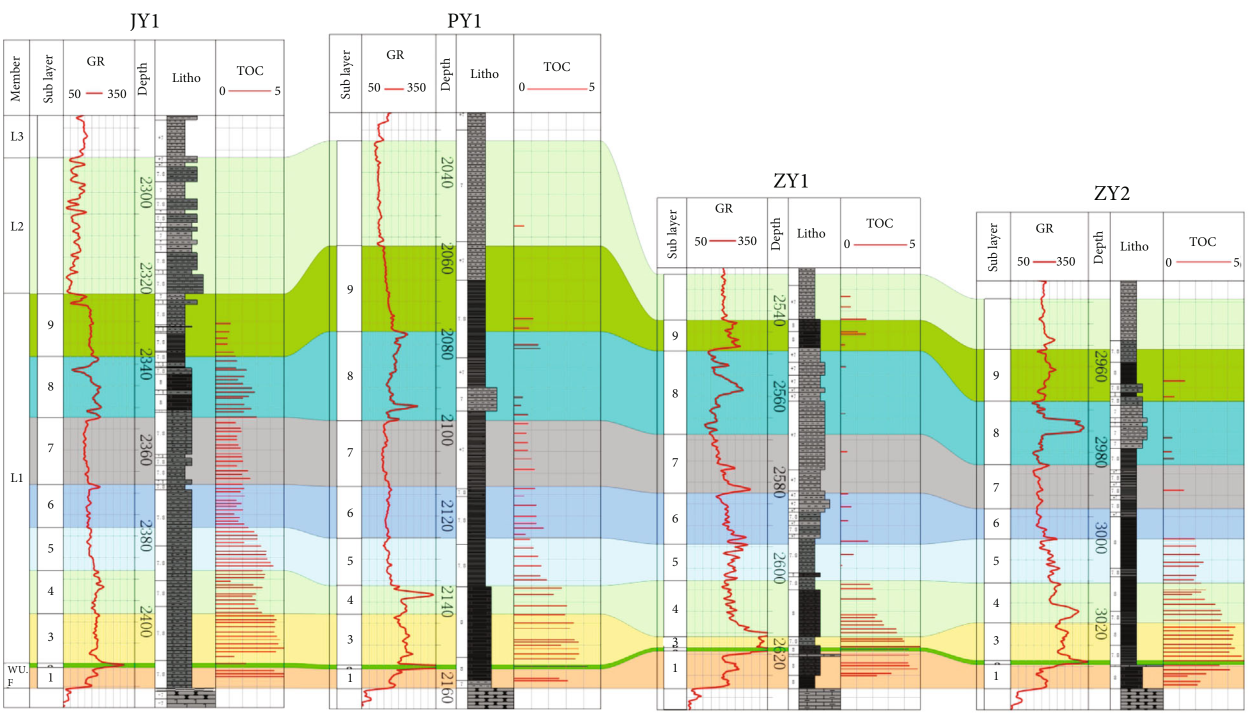
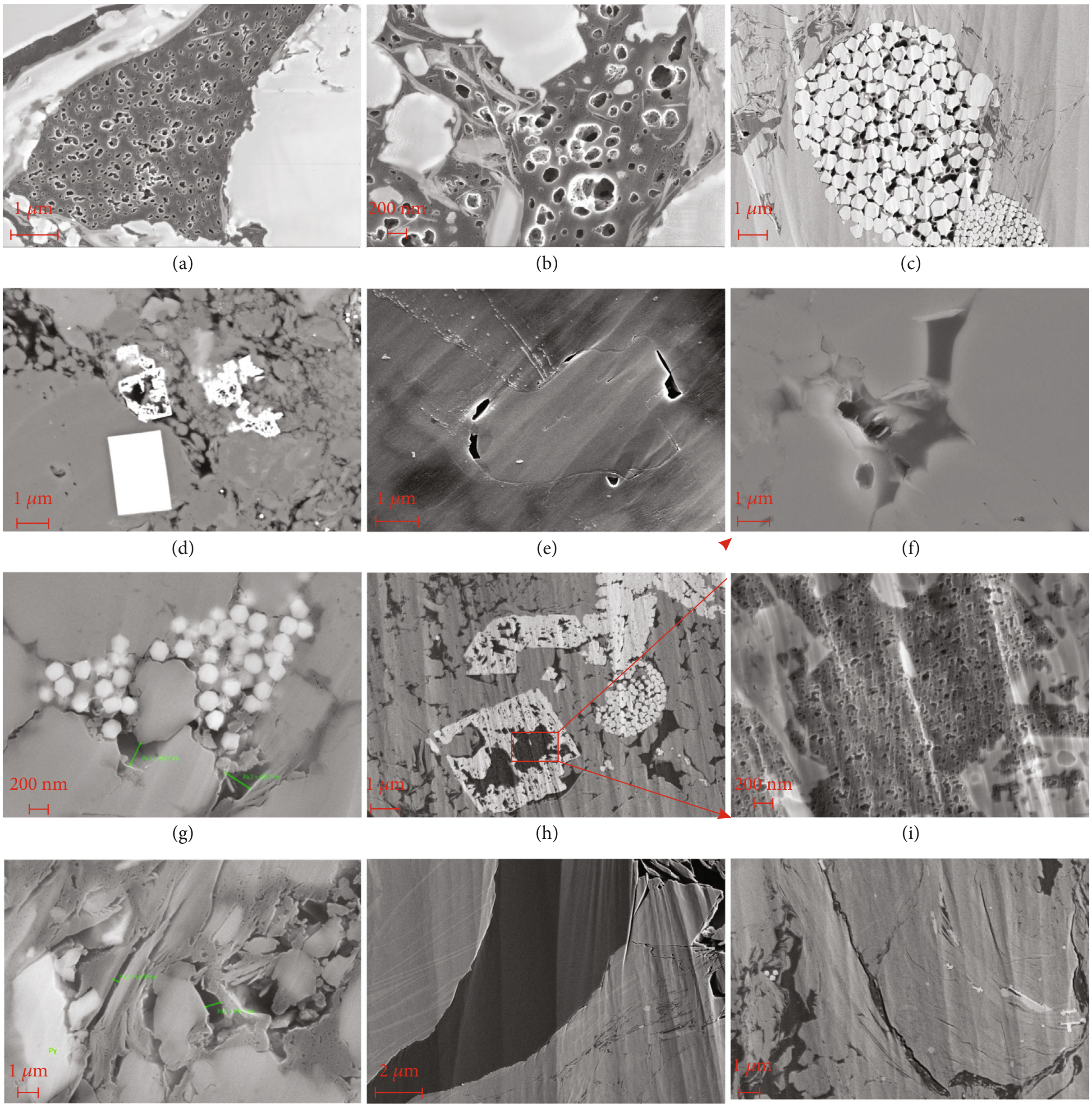


FIGURE 6: Comparison chart of TOC of Wufeng-Longmaxi Formation in southeast Chongqing.

exploration and development of shale gas [33–37]. Based on the combination of microscopic thin sections and FIB-SEM, this paper qualitatively and quantitatively studies the micropore morphology, quantity, and distribution characteristics of the Wufeng-Longmaxi Formation black shale in Qianjiang. The reservoir spaces are diverse, heterogeneous, and complex. There are mainly organic pores with matrix pores, intergranular and intragranular pores, intergranular and intragranular dissolution pores, secondary dissolution pores, and microfractures.

Nanoscale organic pores are formed during the thermal evolution of organic matter [38]. In the highly mature stage, the thermal degradation of kerogen to oil and gas generates carbon-rich residues, secondary micropores, and microcracks [39, 40]. The organic pores in the study area are distributed in honeycombed dispersion, belonging to the pores inside kerogen. The pore diameter is 5 nm-200 nm, and the main part is about 150 nm. They are regular pits, dense networks, or round oval, which occur at the boundary of organic matter and mineral particles (Figures 7(a) and 7(b)). They contribute greatly to the specific surface area and pore volume of shale, which are the main storage space of shale gas. Organic pores are mainly found in the 1-5 sublayers of the Wufeng-Longmaxi Formation, which belongs to a deepwater shelf sedimentary environment, where a large number of graptolites, algae, and other plankton flourish, laying a foundation for the enrichment of organic matter. During the process of hydrocarbon generation by diagenetic



(a) (b) (c) (d) (e) (f) (g) (h) (i) (j) (k) (l)

Figure 7: Photos of the FIB-SEM. (a) Well ZY1, 2617.82 m, honeycomb-like organic matter pores are densely developed, with good connectivity. The pores are round oval, and the pore diameter is 30-250 nm. (b) Well ZY1, 2618.18 m, organic matter pores are extremely developed, the pore diameter is mostly hundreds of nanometers, the pore roundness is high, and there are signs of dissolution at the edge. (c) Well ZY1, 2618.18 m, mold ball-shaped intergranular pore, with pore diameter of 10-200 nm. (d) Well ZY1, 2622.01 m, intragranular dissolution holes, clay minerals associated with authigenic quartz particles are corroded. (e) Well ZY1, 2622.01 m, intergranular pore. (f) Well ZY1, 2612.25 m, corrosion hole in clay mineral particles. (g) Well ZY1, 2624.92 m, corrosion hole between quartz particles and clay mineral particles, with large pore diameter, greater than 200 nm. (h, i) Well ZY1, 2616.33 m, authigenic quartz particles are filled with clay minerals and organic matter. After magnification, micropores filled with organic matter are developed. (j) Well ZY1, 2614.76 m, clay mineral intergranular pores (fractures) and organic matter pores are developed, and corrosion traces can be seen at the edge of authigenic quartz particles. (k) Well ZY1, 2613.00 m, massive organic matter pores are underdeveloped, and it can be seen that the contact contraction joint between clay minerals and kerogen is developed. (l) Well ZY1, 2618.70 m, microfractures run through the horizon, partially filled with organic matter, and several clay mineral contraction joints are developed.

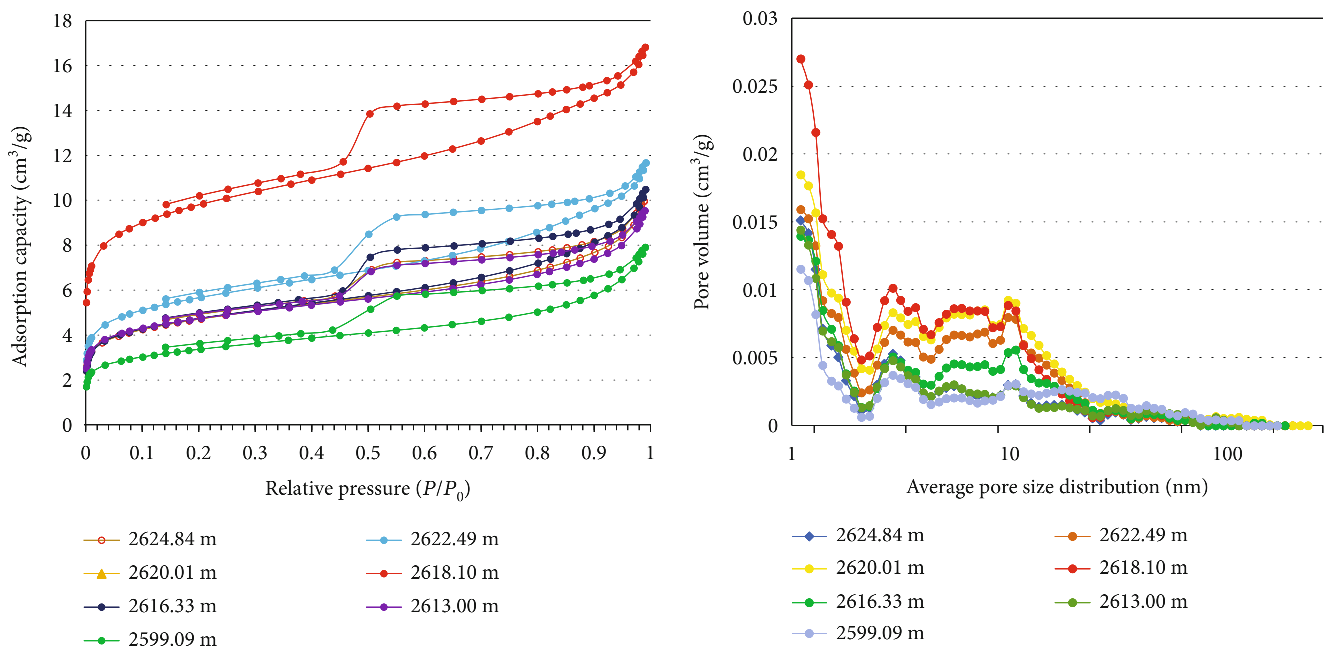
burial in black shale, a large number of organic pores are generated under the action of hydrocarbon expulsion pressure, which not only forms a channel for the migration of conventional natural gas but also leaves storage space for the occurrence of shale gas. Mold globular intergranular pores are another kind of relatively developed pores seen under the microscope (Figure 7(c)). In the early stage of Wufeng-Longmaxi Formation sedimentation, it was in a sedimentary environment of still water, low energy, and oxygen deficiency, accompanied by the development of a large number of mold globular pyrite aggregates. Mineral crystals formed a large number of intergranular micropores, and the pore diameters were mainly distributed between 10 nm and 150 nm. At the same time, some isolated intergranular pores are developed at the compaction edges of dolomite, calcite, feldspar, and other minerals (Figure 7(e)). Secondary intergranular dissolution pores, intergranular dissolution pores, and intergranular dissolution pores are developed inside and at the edges of these crystal particles (Figures 7(d), 7(f), and 7(g)). The pore diameters are generally tens of nanometers to several microns (Figures 7(h) and 7(i)). These pores are mainly formed by the transformation of compaction and water loss during the burial process, which causes the clay particles to rearrange, deform, or fracture, and the geochemical environment of the medium changes from acidic medium to alkaline medium. The clay mineral kaolinite transforms into montmorillonite, illite and montmorillonite-mixed layer, and illite, and the interlaminar pores or micropores are formed by the dehydration or acidification between the layers.

Nanoscale microfractures can also be seen under the microscope, mainly including dissolution fractures (Figure 7(j)), diagenetic fractures (Figure 7(k)), and hydrocarbon generation fractures (Figure 7(l)). The solution fracture is a kind of fracture formed by the acid corrosion of weak parts of the formation water along black shale bedding, such as different mineral components or arrangement of mineral particles. The corrosion joints are mostly filled with secondary minerals such as multiphase calcite, and some are filled with siliceous quartz and asphalt. Diagenetic fractures are formed at the diagenetic stage. Due to the pressure of the overlying layer and its own water loss shrinkage, dry cracking, or recrystallization, unstable minerals are gradually transformed into relatively stable minerals, resulting in a small opening, irregular fracture surface, good connectivity, and partial filling with secondary minerals or organic matter. Hydrocarbon generation fracture is the process of diagenesis and evolution. A large amount of oil and gas is generated after the kerogen is mature. According to the view of the hydrocarbon generation episode, the volume of oil and gas is much larger than the volume of the original kerogen itself. These constantly emerging fluids enter the pores, which will inevitably continue to squeeze the existing fluids in the pores, resulting in the increase of pore fluid pressure. In the relatively closed environment of the hydrocarbon source rocks, abnormally high-pressure hydrocarbon expulsion occurs. Thus, a large number of microcracks are formed on the weak surface (bedding or fissure) of the rock and filled with organic matter or asphaltene.

3.4.2. Pore Structure Characteristics. Thin-section and FIB-SEM methods can help us to qualitatively and semiquantitatively understand the pore morphology and relative pore size of black shale reservoirs. However, black shale is a complex porous medium, which has the characteristics of fine particle size, small pore size, ultralow permeability, and extreme expansion. It is difficult to determine the pore size distribution of the reservoir using the mercury intrusion method. Therefore, previous researchers used the shale pore surface to have a strong adsorption capacity for gas. The nitrogen adsorption method is used to characterize the pore volume, pore diameter, and specific surface area of the black shale reservoir pore structure [41–43]. Seven black shale samples from well ZY1 were collected, and nitrogen adsorption tests were carried out using the ASAP2020 isothermal adsorption apparatus to describe the nanoscale pore structure of the Wufeng-Longmaxi Formation shale gas reservoir. According to the classification of pores defined by IUPAC, nanopores can be subdivided into micropores (<2 nm), mesopores (2-50 nm), and macropores (>50 nm).

(1) Characteristics of Desorption-Adsorption Curves of Nitrogen Adsorption. This time, seven isotherm desorption-adsorption lines with retention loop characteristics were obtained through the nitrogen adsorption experiment (Figure 8(a)), similar to the type IV adsorption isotherm proposed by IUPAC in 1985, which is a single-layer or multilayer adsorption with capillary condensation on the surface of nonporous solids, and the retention loop also has both H_2 and H_3 conditions. In the low medium pressure ($0 < P/P_0 < 0.45$), with the increase of pressure, the adsorption capacity increases slowly, and the adsorption of a single molecular layer changes to a multimolecular layer. In the medium high pressure ($0.45 < P/P_0 < 0.95$), the adsorption capacity increases relatively quickly, the retention loop is wide, and the desorption curve is much steeper than the adsorption curve, which belongs to the H_2 situation, indicating that the pores of the black shale reservoir have a wide pore diameter and a more diverse pore type distribution. In the high-pressure ($P/P_0 > 0.95$), there is no limit to the adsorption capacity in the adsorption curve, which increases slowly with the pressure, belonging to the H_3 situation, indicating that the shale reservoir has a narrow and long fissure-like pore structure, and there are microfractures, which provide a channel for communicating organic pores.

(2) Pore Size Distribution and Specific Surface Area Characteristics Based on Nitrogen Adsorption. According to the experimental results, the pore size distribution ranges from 2 nm to 150 nm (Figure 8(b)). At 2 nm-10 nm, the curve shows multiple peak values, indicating that the probability of pore occurrence within this range is high and the structure is relatively complex. At 10 nm-50 nm, the curve decreases slowly with the pore diameter, indicating that the probability of pore occurrence in this range is small. When it is greater than 50 nm, the curve is delayed, indicating that shale contains a small amount of macropores. According to the capillary condensation model BJH theory, the pore volume is calculated as shown in Table 2. The pore volume ranges from 0.009897 cm^3/g to 0.017177 cm^3/g (average = 0.01304 cm^3/g). Among them, the volume of mesopores accounts for 60 wt% of the total pore volume, while micropores and macropores only account for 40 wt%, indicating that the black shale mesopores contribute the most to the pore volume, and a large amount of shale gas exists in the mesopores as adsorbed, while macropores contribute the least to the pore volume, with only a small amount of the free shale gas reservoir.



(a) (b)

Figure 8: Nitrogen adsorption isotherm (a) and pore size distribution diagram (b) of black shale in well ZY1 in southeast Chongqing.

Table 2: Pore structure parameter statistics of black shale in Wufeng-Longmaxi Formation of well ZY1 in southeast Chongqing.

Sample	Depth	TOC (%)	BJH pore volume (cm^3/g)				BET average pore diameter (nm)	BET specific surface area (m^2/g)
			Micropore	Mesopore	Macropore	Total pore volume		
YDN-1	2624.84 m	2.28	0.003707	0.007056	0.001202	0.011965	3.71	16.9
YDN-2	2622.49 m	4.33	0.00438	0.008471	0.001151	0.014002	3.6	20.3
YDN-3	2620.01 m	4.3	0.004839	0.008581	0.001073	0.014493	3.26	24.8
YDN-4	2618.10 m	5.91	0.006199	0.009723	0.001255	0.017177	2.97	35.1
YDN-5	2616.33 m	3.89	0.003598	0.007935	0.001299	0.012832	3.86	17.1
YDN-6	2613.00 m	2.74	0.003355	0.006578	0.000972	0.010905	3.51	17
YDN-7	2599.09 m	1.85	0.002681	0.006179	0.001037	0.009897	4.12	12.1

The black shale of the Wufeng-Longmaxi Formation in southeast Chongqing has a high specific surface area (Table 2), which is mainly distributed in the range between 12.1 m^2/g and 35.1 m^2/g (average = 20.47 m^2/g). Its significant feature is that the BET specific surface area is directly proportional to TOC. The higher the TOC, the larger the specific surface area and the enhanced adsorption capacity. For example, the TOC of sample YDN-4 (2618.1 m) is 5.91 wt%, and the specific surface area is 35.1 m^2/g, which is significantly larger than other samples. The adsorption curve shows that the adsorption amount is the largest (Figure 8(a)), and the remaining samples showing a decrease of organic carbon content.

According to the microscopic pore structure analysis of the nitrogen isothermal adsorption experiment, the nanopores of the black shale are mainly mesopores, with the largest proportion of pore volume, which is conducive to the adsorption of shale gas. However, the desorption-adsorption curve shows the characteristics of the retention loop, which also shows that the pore morphology is a thin bottleneck, closed at one end, and dominated by isolated pores (Figure 8(b)); the connectivity between pores is poor, which is not conducive to gas seepage.

4. Gas-Bearing Characteristics

The phase states of natural gas in black shale include the free state, adsorbed state, dissolved state, and other possible phase states, in which the natural gas in the adsorbed phase accounts for 20 wt%-85 wt% of the total occurrence [24]. Field analysis is the main method for the gas content test. During drilling, the shale rock samples are sealed and stored in metal analysis tanks and transported to the laboratory. The actual formation conditions are simulated by the heating method, and the core is analyzed. The gas content in the Wufeng-Longmaxi Formation of well ZY1 is relatively high. The gas content in the high-quality shale section ranges from 0.5 m^3/t to 4.43 m^3/t (average = 1.69 m^3/t). The

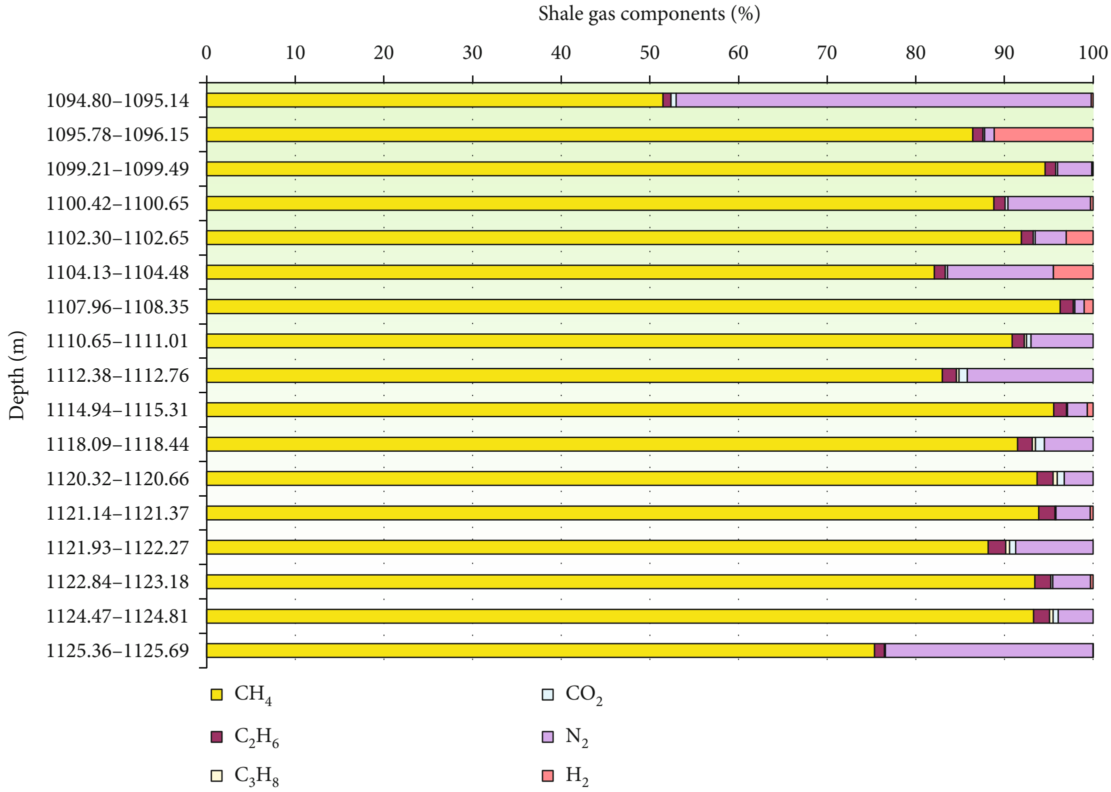


Figure 9: Analysis diagram of shale gas composition in well QD1, southeast Chongqing.

high value is mainly located in the lower part of the Longmaxi Formation, generally $2\,m^3/t$ to $3\,m^3/t$, and there are few test points larger than $4\,m^3/t$ (Figure 3). The gas component chromatographic analysis shows that the shale gas in the high-quality shale section of the Wufeng-Longmaxi Formation is dominated by hydrocarbon gas. The cumulative content of CH_4, C_2H_6, and C_3H_8 can reach 91.67 wt%, of which the average content of CH_4 can reach 89.92 wt%. Nonhydrocarbon gas mainly includes N_2, CO_2, and H_2, of which the N_2 average is 7.14 wt%, belonging to the high-quality natural gas. The proportion of nonhydrocarbon components in the organic-rich shale section of the Wufeng-Longmaxi Formation is gradually higher, which is mainly reflected in the increase of the N_2 proportion. However, after being corrected to the absolute gas content, the average N_2 content is $0.11\,m_3/t$, and it is generally stable from bottom to top (Figure 9). It can be seen that the vertical change of the hydrocarbon gas in the shale gas of the Wufeng-Longmaxi Formation directly affects the change of the gas content.

5. Discussion

5.1. Influential Factors of Reservoir Space Development. Based on the study of mineralogy, organic geochemistry, and pore structure, the study area mainly develops organic pores, intergranular pores, and intragranular pores. The correlation analysis of porosity and permeability shows that they are positively correlated (Figures 10(a) and 10(b)), showing the percolation characteristics of low porosity and ultralow permeability, indicating that organic pores are the main reservoir space, mesopores are the main contributors to pore volume, and the correlation analysis of porosity and organic carbon shows that the development of organic pores in the shale gas reservoir is controlled by the enrichment degree of organic matter. The higher the TOC content, the more developed the pores are, especially the organic pores, which provide the storage and permeability space for shale gas enrichment. Therefore, the analysis of the abundance of organic matter, mineral components, pore volume, and pore specific surface area can further clarify the control factors of the storage and infiltration space.

The correlation analysis of TOC content with pore volume and specific surface area shows that the enrichment degree of organic matter is strongly positively correlated with pore volume and specific surface area, and the correlation indexes are 0.93 and 0.83, respectively (Figures 10(c) and 10(d)). Organic pores play a great role in the development of shale gas reservoir space. The pore volume of micropores, mesopores, and macropores is also positively correlated with the TOC content, but the degree of correlation is inconsistent. The correlation index of mesopores, macropores, and total organic carbon content is strong, 0.86 and 0.95, respectively, while the correlation between micropores and TOC content is weak, and the correlation index is only 0.26 (Figure 10(d)). In addition, the correlation between main minerals and pore volume and specific surface area shows that quartz is positively correlated with pore volume and specific surface area, with correlation indexes of 0.78 and 0.61, respectively (Figures 10(e) and 10(f)); clay

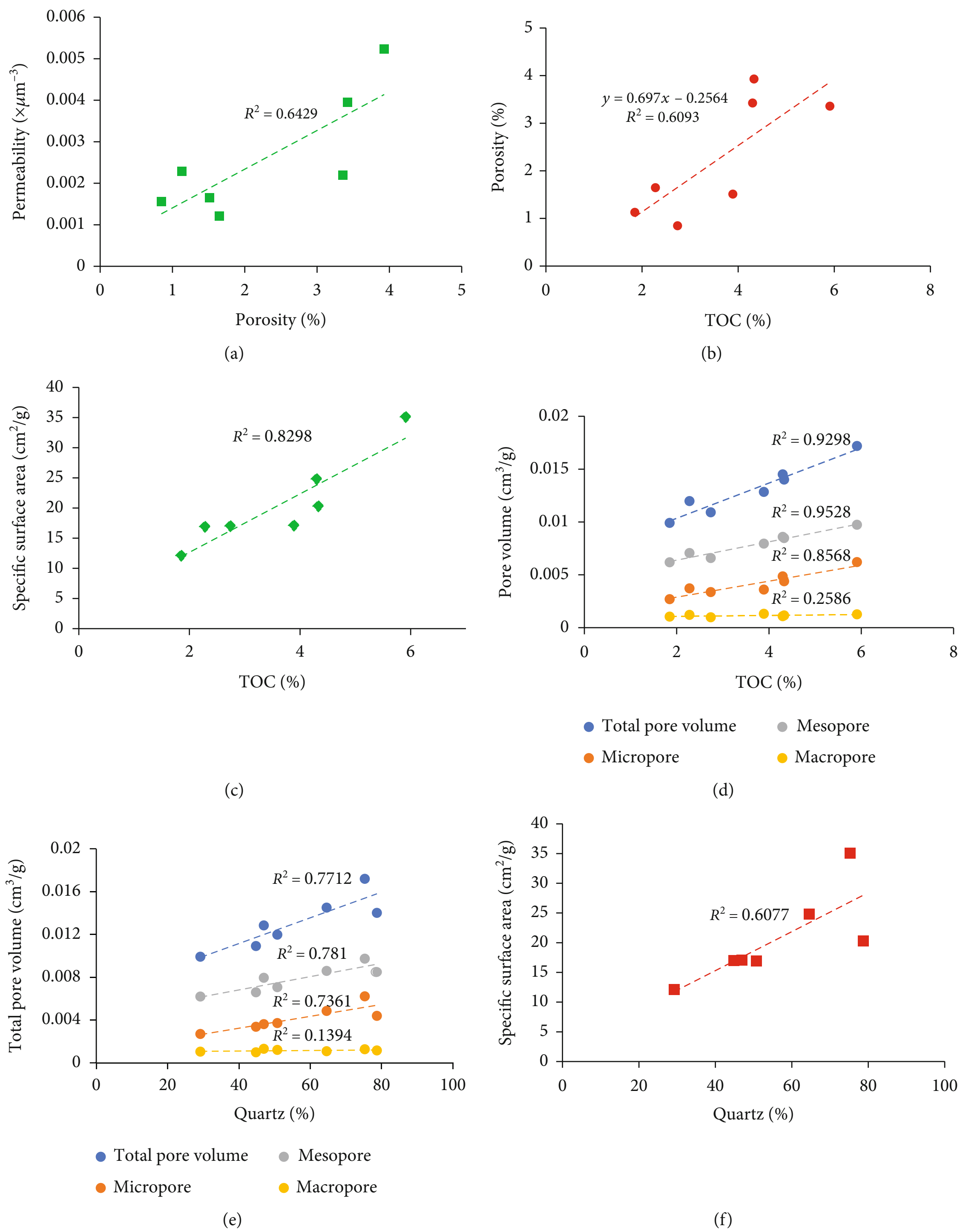
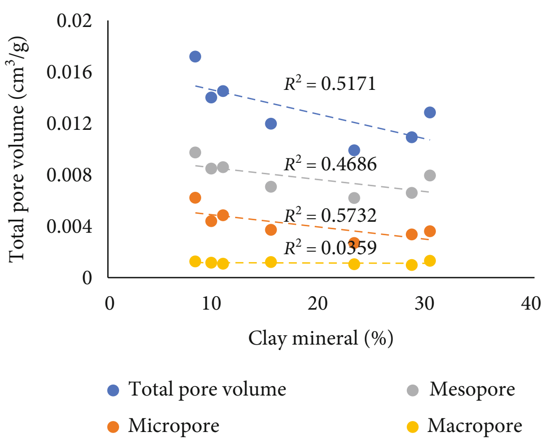
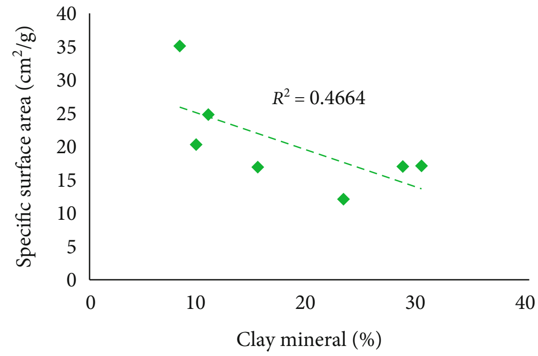


Figure 10: Continued.



(g)



(h)

Figure 10: Correlation analysis of reservoir space parameters with organic carbon and mineral components.

minerals are negatively correlated with pore volume and specific surface area, with correlation indexes of 0.51 and 0.47, respectively (Figures 10(g) and 10(h)), indicating that the enrichment degree of different mineral components is an important factor affecting the development of reservoir space, and the development of quartz particles promotes the development of reservoir space. Clay minerals are not conducive to the development of reservoir space. The correlation analysis of the pore volume of the micropore, mesopore, and macropore with quartz and clay also shows that different mineral types are important factors controlling the development of reservoir space.

Based on the analysis of organic matter abundance, mineral composition and pore volume, and pore specific surface area, it is shown that the enrichment degrees of organic matter and mineral composition in black shale are important factors affecting the development of the shale gas reservoir space. Macropores and mesopores are the main pore sizes for the development of organic pores. Quartz particles promote the development of reservoir space, while clay minerals are not conducive to the development of pores.

5.2. Influencing Factors of Gas Content. The exploration experience of Barnett shale gas in North America shows that the enrichment degree of shale gas is closely related to the TOC and is also controlled by the development degree of the reservoir space, which usually shows a positive correlation. The correlation analysis of the total gas content, TOC, and porosity of well ZY1 shows that (Figure 11) the enrichment degree of shale gas in the Qianjiang area has similar characteristics. The total gas content is positively correlated with TOC and porosity, and the correlation index is 0.43 and 0.3, respectively, but the correlation is weak (Figures 11(a) and 11(b)). There are two main reasons for this. On the one hand, after the organic matter generates hydrocarbons, it not only generates high-pressure protective pores but also forms organic pores during the hydrocarbon generation and evolution process. At the same time, the shrinking of the organic matter volume will generate micro-fissures, which is conducive to the preservation of shale gas. The research of Jarvie et al. [38] also confirmed that the shale with organic carbon content of 7% is consumed by 35% during the hydrocarbon generation process; it can increase the shale porosity by 4.9%. Therefore, there is a good correlation between TOC and porosity. On the other hand, the hydrocarbon generation process of organic matter generates a large number of micropores and microfractures, and more free natural gas is stored in these spaces. The higher the TOC, the larger the total volume of micropores, and the larger the surface area available for adsorption of adsorbed gas [44], thus increasing the total gas content.

The development degree of reservoir space is also an important factor affecting the gas-bearing property. According to the correlation analysis of total gas content, pore volume, and specific surface area, the total gas content is strongly positively correlated with the pore volume and specific surface area, with correlation indexes of 0.93 and 0.88, respectively. Microporous, mesoporous, macroporous, and total gas content have similar correlation characteristics, and microporous, mesoporous, and total gas content have strong correlation. However, the correlation of macropores is poor (Figures 11(c) and 11(d)), which also indicates that the pore volume of micropores and mesopores provides the main storage space for the occurrence of shale gas, while the presence of macropores itself is small and contributes little to the occurrence of shale gas.

According to the previous analysis, shale gas itself is a kind of unconventional natural gas generated and stored by itself. Therefore, the mineral components will directly affect the storage capacity of shale. According to the correlation analysis of total gas content, quartz, and clay, quartz is positively correlated with total gas content, and the correlation index is 0.58, which is a weak positive correlation (Figure 11(e)). This is related to the source of quartz in black shale. There are mainly terrigenous clastic quartz, authigenic quartz transformed by diagenesis and biogenic quartz. The plankton in the Longmaxi Formation is relatively developed,

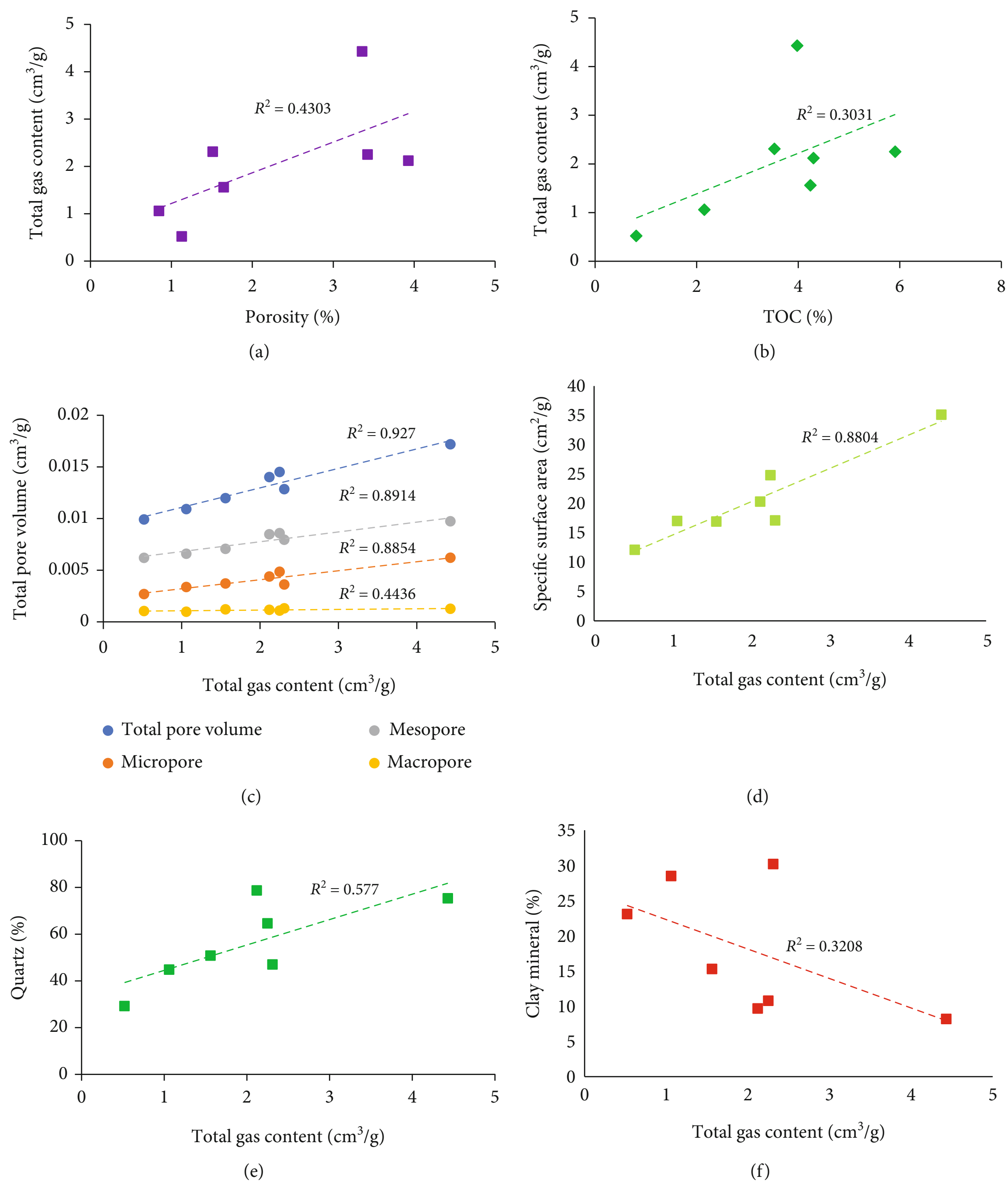


FIGURE 11: Correlation analysis of gas content with TOC and reservoir space.

and the quartz in the shale gas reservoir should mainly come from biogenesis. Clay minerals are negatively correlated with the total gas content, and the correlation index is 0.32, indicating that the increase of clay mineral content is not conducive to the occurrence of shale gas (Figure 11(f)). Therefore, the correlation between quartz and total gas content is related to the enrichment of organic matter. According to the previous analysis, the siliceous quartz of the Longmaxi Formation in the Qianjiang area is relatively developed, which is conducive to the enrichment of organic matter, while TOC is positively related to the total gas content, so quartz controls the enrichment of shale gas to a certain extent, and the increase of clay minerals is not conducive to the accumulation of shale gas.

In general, the enrichment of the total gas content of shale gas in the Qianjiang area is controlled by many factors. The increase of organic matter abundance is the material basis for the increase of the total gas content. The development of organic pores provides high-quality storage space for shale gas preservation. The development of different mineral components restricts the enrichment of shale gas.

6. Conclusion

(1) The thickness of black shale in the Qianjiang area is up to 33 m (TOC > 2%), kerogen is sapropelic type (I), and the Ro ranges from 2.2 wt% to 3.39 wt%, which is a highly mature stage. The mineral composition is relatively complex. Quartz content is in the range between 9.4 wt% and 79 wt% (average = 47.48 wt%), while clay content is in the range between 10.02 wt% and 43.6 wt% (average = 29.41 wt%). High silica content provides good reservoir-fracturing quality for hydraulic fracturing

(2) The black shale reservoir has various pore types, mainly developing organic pores, intergranular and intragranular pores, intergranular and intragranular dissolution pores, secondary dissolution pores, and microfractures. Organic pores are the main occurrence space of shale gas in the Qianjiang area. The nanopore structure can be divided into micropores, mesoporous pores, and macropores, and the pore volume ranges from 0.009897 to 0.017177 cm^3/g (average = 0.01304 cm^3/g). The mesoporous pore volume accounts for 60% of the total pore volume, which contributes the most to the enrichment of shale gas. The specific surface area ranges from 12.1 m^2/g to 35.1m^2/g (average = 20.47 m^2/g). The desorption curve shows the characteristics of the retention loop, and the pore shape is a fine bottleneck; one end is closed, with mainly isolated pores and poor connectivity, which is not conducive to gas seepage

(3) The enrichment degrees of organic matter and mineral composition of black shale in the Wufeng-Longmaxi Formation are important factors affecting the development of the shale gas reservoir space. Micropores and mesopores are the main pore sizes for the development of organic pores. Quartz particles promote the development of reservoir space, while clay minerals are not conducive to the development of pores

(4) The total gas content of shale gas reservoirs in Qianjiang ranges from 0.5 m^3/t to 4.43 m^3/t (average = 1.69 m^3/t). Its enrichment is controlled by many factors. The increase of organic matter abundance is the material basis for shale gas enrichment. The development of organic pores provides high-quality storage space for shale gas preservation. The development of different mineral components restricts the enrichment of shale gas

Data Availability

The [data type] data used to support the findings of this study are included within the article.

Conflicts of Interest

The author declares that he has no conflicts of interest.

Acknowledgments

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