Research Article

Three-Dimensional Morphology and Connectivity of Organic Pores in Shale from the Wufeng and Longmaxi Formations at the Southeast Sichuan Basin in China

Tao Jiang, Zhijun Jin, Zongquan Hu, Wei Du, Zhongbao Liu, and Jianhua Zhao

1State Key Laboratory of Shale Oil and Gas Enrichment Mechanisms and Effective Development, Beijing 100083, China
2Key Laboratory of Shale Oil/Gas Exploration and Production, SINOPEC, Beijing 100083, China
3Petroleum Exploration and Production Research Institute, SINOPEC, Beijing 100083, China
4SinoProbe Center, Chinese Academy of Geological Sciences and China Geological Survey, Beijing 100037, China
5Institute of Energy, Peking University, Beijing 100871, China
6School of Geosciences, China University of Petroleum (East China), Qingdao 266580, China

Correspondence should be addressed to Tao Jiang; jiangt813@cags.ac.cn

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Organic pores play an important role in shale reservoirs. Organic pores occur where shale gas was produced and accumulated. However, there is little scientific understanding of the distribution and connectivity of organic pores. Organic pore types and their structural characteristics were studied using a total organic carbon (TOC), thin section, focused ion beam scanning electron microscope (FIB-SEM), and nano-CT. The samples were from the Wufeng Formation in the Upper Ordovician and Longmaxi Formations from the lower Silurian. The results show that organic matter is mainly concentrated in the Wufeng Formation and the bottom of the Longmaxi Formation and that the middle and upper parts of the Longmaxi Formation contain a low amount of organic matter. The shale of the Wufeng-Longmaxi Formation has high maturity, and its organic pores are well developed. There are three types of organic pores: algae, graptolite, and pyrobitumen pores. The pore connectivity of shale with a high organic content is better than that of shale with a low organic content. The volume of the organic pores accounts for more than 50% of the volume of the organic matter. Majority of the organic pores have an aperture smaller than 100 nm and are round, nearly circular, and elliptical in morphology. Most of the organic pores in a shale formation are developed in pyrobitumen, and most of the larger organic pores are concentrated at the center of solid pyrobitumen. The organic pores in pyrobitumen have the best connectivity and are the most favorable reservoir spaces and migration channels for shale gas, which is a crucial point of reference for future research of shale gas.

1. Introduction

In recent years, shale gas has undergone significant development as an unconventional resource [1]. With the success of industrial shale gas development in the United States, the research and development of shale gas have become important for increasing global oil and gas resources [2]. With rising energy demand, increasing pressure on energy, and growing environmental awareness, it is urgently necessary to explore and develop shale gas resources. China is the third country after the United States and Canada to develop and utilize shale gas resources. To date, China has made significant progress in the research and development of shale gas [3–7]. In Jiaoshiba Chongqing, the first large-scale shale gas field in China was built and has reached 100 billion cubic meters [8].

Organic pores are widely developed in shale that is rich in organic material and are an important part of the shale reservoir space [9]. Shale gas can be stored in a free phase in organic pores and can be adsorbed onto the surface of
organic matter in an adsorbed phase [10]. The porosity of the organic matter directly determines the distribution of the gas [7]. The type and maturity of organic matter are the geological conditions used to judge the commercial developmental value of shale gas [11]. Therefore, the study of organic pores is important for shale gas research and the evaluation of shale gas as a resource.

There are many types of pores in shale. There are primary pores, such as matrix cracks and intergranular pores, and there are secondary pores, such as organic pores, dissolved pores, and organic microfractures [12]. Organic pores are important because they are one of the largest reservoir spaces. Organic pores have various shapes, and they can be elliptical, circular, or irregular polygons, but are primarily in the form of ellipses, with pore sizes between 5 and 200 nm [13–15]. Well-developed organic pores have good connectivity, forming interconnected pore networks that can contain large shale gas reservoirs. Pores in two adjacent organic materials or in different parts of the same organic material can be different [16, 17]. In addition, there are many forms of organic matter in shale, mainly occurring in the presence of clay and brittle minerals. Pores can comprise up to 50% of the volume of the organic matter. The formation of organic pores is a product of the maceral content of organic matter and the degree of host shale maturity. Some macerals generate hydrocarbons and a large number of pores as its maturity increases [18, 19]. When the maturity of the organic matter ($R_o$) is less than 0.90%, organic pores are not well developed. In the gas window, liquid hydrocarbons begin to crack, and organic pores begin to develop [20]. Loucks et al. [19] and Slatt et al. [12] indicated that organic pores formed when $R_o = 0.60$. The maturation of organic pores can be divided into three stages: the formation period ($0.60% < R_o \leq 2.00%$), the development period ($2.00% < R_o \leq 3.50%$), and the destruction period ($R_o > 3.50$). The density of organic pores is a function of the TOC content, maturity, and the type of organic matter such as maceral or pyrobitumen [21–23].

Currently, there is no detailed description or classification scheme for organic pores in shale. What characteristic differences may exist between different types of shale remains unclear. It is necessary to know the connectivity of organic pores in shale formations. In particular, the current understanding of the three-dimensional morphology and connectivity of pores in different types of organic matter is insufficient. Therefore, various organic pores in shale were analyzed to classify the types of organic matter present and the morphology and connectivity characteristics. The present study augments our understanding of shale gas storage space and will assist in the research and development of shale gas.

2. Geological Background

The Sichuan Basin is located at the northwestern edge of the Yangtze Platform [24]. The basin has experienced two stages: the Craton stage (from the Early Paleozoic to Middle Triassic) and the foreland basin stage (from the Late Triassic to Cenozoic) [25]. The Sichuan Basin began to form during the early Indosinian Movement and, eventually, formed a diamond-shaped sedimentary basin after the Himalayan movement (Figure 1). The Sichuan Basin is surrounded by a contiguous range of mountains. The Sichuan Basin is a complex, superimposed basin with terrestrial and marine sediments [26]. The shale deposits in the central Sichuan Basin are thin, and the shale deposits in the eastern, southeastern, and southwestern Sichuan Basin are relatively thick.

The southeast Sichuan Basin is composed mainly of ejective folds that consist of high-steep anticlines, loose synclines, and fault zones [26]. The basement of the southeast Sichuan Basin is composed of Presinian metamorphic rocks. The Devonian, Carboniferous, Cretaceous, and Paleogene rock strata were absent from the entire area. During the Early-Middle Ordovician, the area transformed from an open sea to a restricted sea surrounded by uplifts, resulting in a low-energy and anoxic sedimentary environment [27, 28]. During the Early Silurian, the southeastern Sichuan Basin was a semioccluded stagnant basin with a deep shelf environment. A black, organic-material-rich shale was deposited, forming the best layer for shale gas investigation in the region [29, 30].

The lower section of the Wufeng-Longmaxi Formation is primarily composed of a black graupelite shale, and the upper section is primarily a gray argillaceous siltstone [31]. During the Longmaxi’s Formation period, the Sichuan Basin was shallow and has graupelite shale deposits and a short deposit range compared with that of the Wufeng’s Formation period [32]. The Wufeng-Longmaxi Formation in the southeast Sichuan Basin is relatively thick, and the terrigenous detrital content increases from the lower to the upper section. At present, the Wufeng Formation and the lower Longmaxi Formation are the main shale gas reservoirs [33].

![Figure 1: Tectonic setting and location of the southeast Sichuan Basin. (a) Location of the Sichuan Basin in China. (b) Tectonic setting of the southeast Sichuan Basin.](image)
3. Sampling and Experimental Methods

The total organic carbon (TOC) content was tested with a Leco analyzer. The inorganic carbon in the sample was removed with diluted hydrochloric acid and then burned in a high-temperature oxygen oven to convert all the organic carbon to carbon dioxide. The TOC content was then measured by an infrared detector. The type and maturity of organic matter were observed under a LABORLUX 12 POL fluorescence microscope. Samples larger than 4 mm were cemented with a non-fluorescent cement to a microscope slide and observed using the microscopes oil immersion lens. A high-pressure mercury vapor or xenon lamp was used as the excitation source, and then, a blue or ultraviolet light was used to excite the filter. Organic matter with fluorescence was isolated and tested. The reflectance of the graptolite was determined. Then, the type and maturity of the organic material were obtained.

The two-dimensional micromorphology, structural characteristics, and three-dimensional connectivity of the samples were studied using an FEI Quanta 200F focused ion beam scanning electron microscope (FIB-SEM) in the high-vacuum scanning mode. The electron image was obtained from the secondary electron signal of the sample.

Figure 2: Comparison sections of the TOC content of the Wufeng-Longmaxi Formation shale in the southeast Sichuan Basin. (a) Section of the TOC content from northeast to southwest. (b) Section of the TOC content from east to west.

Figure 3: TOC distribution of the shale in the Wufeng-Longmaxi Formation at the southeast Sichuan Basin.
surface through an ion beam generated by a liquid metal (Ga) ion source. The sample was observed to have dimensions of $4.5 \mu m \times 8 \mu m \times 8 \mu m$. The surface of the sample was polished with a JEOL IB-09010 argon ion sample polishing machine. Pores with apertures larger than 5 nm could be observed by the FIB-SEM, and ImageJ was used to calculate the face rate based on gray recognition.

A Zeiss Metrotom nano-CT was used to study the three-dimensional characteristics of the samples. Sixty-five micrometer diameter cylinder samples were selected. The resolution of the nano-CT in the present study was 65 nm. Therefore, pores larger than 65 nm could be imaged. The working principle is that the X-ray source and the detection receiver scan the samples synchronously. When each scan was completed, the scanning rack rotated to the next angle and then performed the next scan. Through digital image processing and three-dimensional reconstruction of the CT single-image and CT image sequences, the porosity of the samples was calculated, and an image was obtained of the three-dimensional distribution and connectivity of pores.

Figure 4: Graptolite photographs and energy spectrum of the Wufeng-Longmaxi Formation shale in the southeast Sichuan Basin. The fragment is graphitic, that is, mainly composed of carbon. The fragment is encased in clay minerals and carbonate minerals.

Figure 5: Pyrobitumen morphology photograph and energy spectrum of the Wufeng-Longmaxi Formation shale in the southeast Sichuan Basin. (a) Fluorescence microscope photo. (b) Polarized light microscope photo. (c) SEM photo and energy spectrum characteristics.
4. Results

4.1. Organic Matter Characteristics

4.1.1. Organic Matter Content. The organic matter in the southeast Sichuan Basin has many different characteristics in both the horizontal and vertical space [34]. The thickness of the TOC > 2% shale varies significantly from northeast to southwest (Figure 2(a)). From the northeast to the southwest, the thickness gradually declines. The thickest area is around the JY-1 well in the northeast, approximately 40 m thick, while the shale with TOC > 2% in the southwestern area is less than 20 m thick. The JY-1 well area and LY-1 well area have the thickest shale from east to west (Figure 2(b)). In the vertical direction, the TOC > 4% shale is mainly concentrated at the Wufeng Formation and at the bottom of the Longmaxi Formation, and the TOC content gradually decreases from the bottom to the top in the Longmaxi Formation. The average TOC content of the Wufeng Formation is 4.3%, while the average TOC content of the bottom of the Longmaxi Formation is 3.2% (Figure 3).

4.1.2. Organic Matter Type. Based on light microscopy and FIB-SEM observations, the majority of organic matter in the southeast Sichuan Basin is algae, pyrobitumen, and graptolite. A small amount of biodetritus was also observed, such as acritarchs, chitinozoans, and sponge spicules (Figures 4–6). Most of the graptolite has a thin, dense carbonaceous structure (Figure 4), and its pores are poorly developed. There are contraction fractures between the graptolite and surrounding minerals. Energy spectrum analysis showed that the graptolite is mainly carbonaceous.

Pyrobitumen generally fills the space between mineral particles and has no fixed form (Figure 5). Compared with the dense structure of graptolite, pyrobitumen has a loose structure and more well-developed pores (Figure 5(c)). Energy spectrum analysis showed that the main elemental composition of pyrobitumen is also carbonaceous (Figure 5(d)).

Algae are generally secondary components that postdate hydrocarbon generation and are covered with lamellate clay minerals. The clay minerals do not have a fixed orientation and look like petals (Figure 6). The algae are also relatively loose, and energy spectrum analysis showed that the elemental composition of the algae is mainly carbonaceous (Figure 6(c)).

Energy spectrum analysis showed that the biodetritus contains mostly phosphorus and calcium, with a relatively complete structure and morphology (Figure 7; [35]). The biodetritus is relatively dense, and its pores are either not developed or only a few nanoscale micropores are developed.

Figure 6: Algae morphological photograph and energy spectrum of the Wufeng-Longmaxi Formation shale in the southeast Sichuan Basin. (a) The dark part is algae. (b) Amplification of (a). (c) Amplification of (b). The algae are mainly carbonaceous and energy spectrum characteristics.
4.1.3. Organic Matter Maturity. The microscopic organic components that indicate the maturity of the black shale in the Wufeng-Longmaxi Formation in the southeast Sichuan Basin are mainly three types: pyrobitumen, biodetritus (predominantly graptolite), and vitrinite-like maceral (VLM). The black shale of the Wufeng-Longmaxi Formation is abundant in graptolite, with obvious morphological features. In the present study, the reflectance of the graptolite was measured. Based on 18 samples from 12 wells, the $R_o$ is between 2.22% and 3.13%, with an average of 2.71% [36, 37], indicating that the shale has a high degree of maturity (Figure 8).

4.2. Organic Pore Characteristics

4.2.1. Algae Pores. The pores in the organic matter of the Wufeng-Longmaxi Formation shale are generally well developed, but the pores in different types of organic matter show significant variability. The cell structure of the algae is poorly preserved, and most of it have been degraded and heavily micronized. This type of organic matter has a distinct shape, and the inorganic minerals produced by the internal metasomatism have an obvious biological structure. The organic matter pores inside the algae mostly have a local concentration, and the surface porosity is approximately 15% (Figure 9).

4.2.2. Graptolite Pores. Generally, graptolite is poorly preserved and displays a fixed, rigid shape. Fractures can form between the graptolite and inorganic minerals. Small quantities of inorganic minerals are observed inside the graptolite, and the pores are poorly developed, with 5% surface porosity (Figure 10).

4.2.3. Pyrobitumen Pores. Pyrobitumen does not have a fixed shape and fills the spaces in the inorganic mineral particles of the clay layers. The pores are well developed in the pyrobitumen, with a 30% surface porosity (Figure 11). The pore shape within the pyrobitumen is controlled by the morphology of the pyrobitumen. The pores are mainly concentrated in the center of the pyrobitumen. The pore morphology is mostly
Figure 9: Pore characteristics of the algae in the Wufeng-Longmaxi Formation shale at the southeast Sichuan Basin.

Figure 10: The pore characteristics of graptolite in the Wufeng-Longmaxi Formation shale at the southeast Sichuan Basin.
ellipsoidal or semiround, with a 10-300 nm long axis and a 2-100 nm short axis. The pores are less developed at the edges of the pyrobitumen, especially at the throat area adjacent to the inorganic minerals. The morphology of the pyrobitumen is mainly long strips or flat, with an aspect ratio greater than 5:1. The long axis direction of the pores agrees with the long axis direction of the pyrobitumen (Figure 11). The pyrobitumen portion contained pores ranging in size from 5 to 50 nm (Figure 12; [38]). These pyrobitumen pores are the main body of organic pores in the Wufeng-Longmaxi Formation shale.

5. Discussion

5.1. Morphology and Connectivity of Organic Pores. The pores in the shale exist in three dimensions. Nano-CT scans can display the internal mineral composition and pore structure in 3D [39]. The connectivity of the organic-material-rich shale at the bottom of the Wufeng-Longmaxi Formation is medium-poor, and the pore size is small (Figure 13). The continuity of organic matter in three-dimensional space is good, and a large number of pores have developed in these well-connected organic materials. Figure 13(b) shows the organic pores in different regions using multiple colors. Areas with the same color indicate that the nanopores in the organic matter of this part are connected. These different-sized pores form a complex spatial network structure. The pores in the sample are mainly circular, and the shape of the pores is sill-like, sheet-like, and tubular, with moderate connectivity. The throat is needle-shaped and partially sill-like (Figure 13(c)). The sample had a total of more
than 7400 organic pores, with a total volume of $4.6 \times 10^{11}$ nm$^3$, which accounted for 5.7% of the total sample volume of $8 \times 10^{12}$ nm$^3$. The sample had 5120 organic pores (larger than 65 nm), and the total volume of the organic pores was $2.6 \times 10^{11}$ nm$^3$, which accounted for 3% of the total volume of the sample and 56.6% of the total organic matter volume.

The organic-material-poor shale in the middle and upper parts of the Wufeng-Longmaxi Formation has poor connectivity (Figure 14). The continuity of the organic matter in three-dimensional space is poor, and the connected organic pore range is obviously reduced (Figure 14(b)). The same characteristics are also visible in the organic throat distribution. The pores of the sample were mainly elongated (similar to intergranular pores) and flake-shaped with poor connectivity. The throat was needle-shaped (Figure 14(c)). The sample had a total of 2698 organic pores with an overall volume of $1.2 \times 10^{11}$ nm$^3$, which accounted for approximately 1.6% of the total sample volume of $8 \times 10^{12}$ nm$^3$. The sample had 1067 organic pores, which accounted for 0.9% of the total volume of the sample.

According to the 3D FIB-SEM data, the organic pores in the Wufeng-Longmaxi Formation shale with long axes < 100 nm accounted for approximately 89% of the total organic pores, while the organic pores with long axes > 200 nm accounted for less than 4% of the total organic pores (Figure 15). Organic pores have a relatively high proportion of small pores [40]. It was determined that the majority of the organic pores in the sample were less than 50 nm, and a large number of the small pores had developed inside the organic matter.

The organic pores with aspect (long – to – minor axis) ratios > 4 only comprise approximately 15% of the total organic pores, and more than 50% of the pores have aspect ratios < 2 (Figure 16). The morphological characteristics of the organic pores obtained by three-dimensional FIB-SEM are similar to those obtained by two-dimensional FIB-SEM: the shapes of the organic pores are primarily rounded, nearly circular, or elliptical.

5.2. Role of Pyrobitumen Pores. The majority of organic matter in the Wufeng-Longmaxi shale are migrated organic matter of amorphous form, uniform color, and nanometer-scale internal pores [41]. The migrated organic matter was injected into the mineral pores (mainly intergranular) in the form of liquid hydrocarbon during the shale oil generation stage [42]. If the TOC content in the shale is 5% by weight, the organic matter density is approximately 50% of the shale density. The organic matter then accounts for nearly 10% of the pore volume of the shale, while the organic matter contains approximately 20%-40% of the
volume occupied by organic pores. Therefore, the organic matter has an organic matter porosity of 2%-4% of the total volume of the shale. Due to its low porosity and poor permeability, it was difficult for liquid hydrocarbon to migrate out of the shale [43]. The remaining hydrocarbon separated into light hydrocarbon (primarily methane) and heavy hydrocarbon (primarily solid pyrobitumen) [44]. The pyrobitumen pores were formed during the process of gas and liquid separation.

Based on the analysis of 832 pyrobitumen enclaves in 16 samples, it was determined that the pyrobitumen pores are primarily ellipsoidal or near-circular. The long-to-minor axis ratios of the pyrobitumen pores are similar. The aspect ratios are concentrated at $< 3$, and less than 10% of the long-to-minor axis ratios are $> 3$ (Figure 17).

The pores in the center of the pyrobitumen are mostly rounded, and the aspect ratios are concentrate close to 1. The pores close to the throat between inorganic minerals

**Figure 14:** Organic pore characteristics of the shale based on the nano-CT. TOC is 1.6%. (a) The blue part is organic matter. (b) Interconnected pores in the organic matter. (c) Throat distribution in the organic matter.

**Figure 15:** Characteristics of the long axis of organic pores based on 3D FIB-SEM data. The majority of long axes are shorter than 100 nm.
are flat shaped or in long strips for the most part, with long-to-minor axis ratios higher than 3. The larger the volume of a single pyrobitumen enclave, the more developed the pyrobitumen pores and the larger the pore size. Additionally, the large pores are concentrated at the center of the pyrobitumen (Figure 18).

For example, Figure 18(a) shows that one 9 μm long pyrobitumen portion has abundant nanopores, and its surface...
Porosity is approximately 30% (Figure 18(a)). From the edge of the pyrobitumen to the center, the size of the pores has a pronounced increasing trend, from a majority of less than 100 nm near the edge, increasing to 500 nm further in, and increasing to greater than 500 nm at the center of the pyrobitumen. This trend may indicate that most of the gas are generated at the center of the organic matter concentrations (Figure 18(b)). The long axis direction of the pores is generally coordinated with the extension direction of the pyrobitumen. Especially in narrow pyrobitumen enclaves, the long axis direction of the pores is consistent with the extension direction of the pyrobitumen, indicating that pyrobitumen deformation has occurred on the internal pyrobitumen pores.

The pyrobitumen pores are not completely isolated from each other and have good connectivity. The large pores are generally interconnected by small pores (Figure 19(a)). In the No.1 pore, there are 2 secondary nanopores, and their aspect ratios are all lower than 1.5 (Figures 19(b)–19(d)). In the No.2 pore, there are 3 secondary nanopores, and their aspect ratios are all greater than 1.5 (Figures 19(b)–19(d)). The relationship between the large pyrobitumen pores and the secondary pyrobitumen pores indicates that they were generated by similar processes. During the formation and accumulation of shale gas, small secondary pores may accumulate within large pores, increasing the size of the larger pores.

6. Conclusion

The distribution of organic matter in the southeast Sichuan Basin has distinct variations both horizontally and vertically. The TOC > 4% shale is mainly concentrated at the Wufeng Formation and at the bottom of the Longmaxi Formation. The organic materials are primarily algae (secondary components), pyrobitumen, and graptolite, with a small amount of biodetritus, such as acritarch, chitinozoa, and sponge spicule. The Wufeng-Longmaxi Formation shale has a high degree of thermal evolution, and its $R_o$ is between 2.22% and 3.13%, with an average of 2.71%.

There are three main types of organic pores: algae pores, graptolite pores, and pyrobitumen pores. The cell structure of algae pores is poorly preserved but displays an apparent biological structure. Algae pores are locally concentrated. Graptolite is generally poorly preserved and has a fixed, rigid shape. Small quantities of inorganic minerals can be found inside the graptolite. The pyrobitumen does not have a fixed shape and fills the spaces between inorganic mineral particles and clay layers. The pyrobitumen pores are well developed, as determined by the overall morphology of the pyrobitumen enclaves.

The pores in the organic-material-rich shale are primarily circular, nanoscale pores. The majority of throats are
needle shaped, and the organic pores may account for more than 50% of the total organic matter volume. The organic matter in the organic-material-poor shale has poor continuity in three-dimensional space. The pores are mostly long and narrow, and the majority of throats are needle shaped. The organic pores may account for less than 1% of the total sample volume.

The majority of the pyrobitumen pores are ellipsoidal or near-circular, with aspect ratios concentrated at <3. The pores in the center of the pyrobitumen are mostly rounded, and the aspect ratios are predominantly close to 1. The pores close to the throat between the inorganic minerals are largely flat or in the shape of long strips, and their aspect ratios are mostly greater than 3. The large pores are concentrated in the center of the pyrobitumen, and the pyrobitumen pores are generally well connected.

Data Availability

The experimental data used to support the findings of this study are included within the manuscript.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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