Structural characteristics and porosity estimation of organic matter-hosted pores in gas shales of Jiaoshiba Block, Sichuan Basin, China

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Abstract
Nanoscale organic matter-hosted pores (OM pores) are the dominant pore type in the marine, organic-rich shales of the Upper Ordovician Wufeng Formation and the Lower Silurian Longmaxi Formation's first member in the Jiaoshiba Block, which provide important reservoir space for shale gas accumulation. In this study, field emission scanning electron microscopy (FE-SEM), statistical analysis using ImageJ software, and gas adsorption tests were conducted to investigate the characteristics of OM pores, including pore shape, pore size distribution, pore quantity, and organic porosity. FE-SEM images show that the OM pore sizes range between 2 and 900 nm. The predominant shape of the OM pores in the Wufeng shale is an irregular polygon, while elliptical and subrounded OM pores were observed in the Longmaxi Formation's first member shale. Average cross-section area ratios of the OM pores to corresponding single-particle organic matter (ØOMP/OM) for shale samples obtained via statistical analysis of FE-SEM images range between 10% and 30%. We propose a new combination approach for the estimation of organic porosity, which gives organic porosities for the Wufeng shale and Longmaxi Formation's first member shale of 0.56%-4.47% and 4.06%-4.21%, respectively. To calculate the organic porosity for the OM pores with diameters of 10-900 nm, the formula derived from the mass-volume-density-organic matter relationships and ØOMP/OM of shale was used, while the organic porosity for pores with diameters of 0.3-10 nm was estimated using carbon dioxide and nitrogen adsorption data. In addition, the reasons for the differences in the OM pore structures in the Wufeng shale and Longmaxi Formation's first member shale were examined for the study area and found to be the result of variations in total organic carbon (TOC) content and tectonic compression of the Wufeng shale.

Keywords
Jiaoshiba Block, Longmaxi Formation, OM pores, organic porosity, Sichuan Basin
1 | INTRODUCTION

The organic matter-hosted pores (OM pores) in shale gas reservoirs are considered to be of particular relevance for the development of unconventional shale resources, and the structural characteristics of OM pores in shale pore systems have been extensively studied over the last decade. A significant amount of nanoscale OM pores develop in organic matter (OM) in shale, especially in marine shales with high thermal maturity, which are among the most important shale intervals for shale gas production. Given the particularity of shale pore systems, to visually observe the OM pore structure, investigations to date have mainly relied on advanced imaging techniques, such as field emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM), with Ar-ion-beam milling being the most common sample preparation method.

High-resolution SEM investigations of OM pores in Barnett shales show that they exhibit irregular, bubblelike, and elliptical cross-sections and generally range between 5 and 750 nm in length. OM pores commonly appear isolated in two dimensions but display connectivity in three dimensions, which has been illustrated using SEM-focused ion beam (FIB) analysis. In recent years, investigations of OM pores have mainly focused on: (a) where OM pores develop (kerogen, bitumen, or pyrobitumen); (b) when they develop with regard to hydrocarbon generation and cracking (within the oil window or beyond); (c) their mode of formation (inherited or authigenic); (d) the influences of the organic carbon content and OM type; and; (e) the importance of the organic porosity with regard to hydrocarbon storage and production.

The porosity of the OM in a shale reservoir is the ratio of the total OM pore volume to the bulk volume of the shale reservoir. The calculation of the organic porosity in shale gas reservoirs is an important part of shale reservoir evaluation. Scholars indicate that the OM pore volume strongly correlates with the shale composition and have derived an equation to calculate organic porosity. Considering that the development of the organic porosity is the result of a chemical reaction, can be evaluated using chemical reaction kinetics. Other researchers focused on quantitative statistics of FE-SEM images of OM pores to obtain the average cross-section area pore ratio of the OM and calculate the organic porosity using the total organic carbon (TOC) content.

Shales are typically dominated by micropores and mesopores that make up approximately 70%-80% of the total pore volume, with various amounts of macropores (pore classification based on Rouquerol et al, 1994). Because the TOC content and thermal maturity of shales are the primary controlling factors for pores <50 nm in diameter and as highly mature organic-rich shale, the OM pores are dominated by micropores and mesopores, OM pore volumes with pore sizes <50 nm cannot be ignored for the estimation of the organic porosity. However, to date, high magnification methods are not capable of resolving the full range of OM pores (ie, smaller pores cannot be observed at high magnification), and a combination of different techniques is required, including gas physisorption and mercury intrusion.

For the organic-rich shales of Upper Ordovician Wufeng Formation (O3w) and the Lower Silurian Longmaxi Formation’s first member (S1l1) in the Jiaoshiba Block of the Fuling gas field, OM pores are the most important pore type. The OM pore system in these shales provides not only important storage space for hydrocarbon gases but also acts as the main channel of gas seepage. The structural characteristics of the OM pores in these shale reservoirs have been previously studied, but are still not well understood.

Based on petrological analysis, FE-SEM, and energy spectrum analysis, the major objectives of this study were to: (a) investigate the development and distribution of OM pores; (b) analyze the relationship between OM and mineral particles; (c) reveal the structural characteristics of the OM pores; and; (d) estimate the organic porosities using statistical data and gas adsorption experimental data in combination with the relationships between mass, volume, density, and OM content for the O3w-S1l1 shales in the Jiaoshiba Block of the Fuling gas field.

2 | GEOLOGICAL BACKGROUND

The Jiaoshiba Block, located in the eastern part of the Fuling District of Chongqing city, is situated to the west of the Qiuyueshan fault, the eastern boundary of the Sichuan Basin, China. It is a junction of multiple structural units, such as the Shizhu synclinorium, the Fangdoushan anticlinorium, and the Wanxian synclinorium, in the eastern Sichuan fold belt (Figure 1A). The Jiaoshiba Block is related to the Jiaoshiba anticline, which is a broad box-shaped anticline with north-east axial trending. The stratum at the top of the anticline has a gentle dip angle and weak deformation and is relatively stable, with few faults in the middle part. However, the edge of the Jiaoshiba anticline is controlled by two groups of faults trending in a northeast/southwest and a nearly north-south direction, respectively (Figure 1B), and the formation dip of the two flanks is approximately 30°. The strata were revealed by drilling of the Jiaoshiba Block range from the Upper Ordovician Jiaocagou Formation to the Lower Triassic Jialingjiang Formation; the Devonian is absent and the Carboniferous partially missing. The total thickness of the stratigraphic sequence from the Sinian to the Jurassic in the Jiaoshiba Block is approximately 4000 m (Figure 1C).

The O3w-S1l1 shales are the main gas-producing units in the Jiaoshiba Block, and mainly consist of continental shelf sediments. Regional studies show that the O3w-S1l1
shales are widely distributed and vary in thickness from 80-120 m. The current burial depth of the bottom of the O$_3^w$ shale mainly varies from 2250-3500 m. According to drilling data of the Jiaoshiba block, the lithology of the main gas-producing formation is dominated by black shale with abundant graptolite and high silicon and organic carbon contents. Based on the quality of the reservoir, the shale gas-producing formation is divided into a high-quality section and a sub-quality section (the upper part of the S$_1^{l1}$). The high-quality shale section, the main gas-rich shale, is made up of O$_3^w$ and the lower part of S$_1^{l1}$ is mainly composed of deep-water continental shelf sediments with organic-rich and siliceous-rich black shales. The TOC content, porosity, and thermal maturity ($R_o$) are 3.0%-6.0%, 2.8%-7.1%, and 2.5%-3.0% $R_o$, respectively. The layers of the upper part of the S$_1^{l1}$ shale were mainly deposited in shallow-water continental shelf sedimentary environments, developing dark gray and gray mudrock.

According to the lithology, geochemical characteristics, and gas-bearing characteristics, the O$_3^w$-S$_1^{l1}$ shales in the Jiaoshiba Block are divided into nine layers. The first and second layers belong to O$_3^w$ and the third to fifth layers to the lower part of S$_1^{l1}$, while the remaining layers make up the upper part of S$_1^{l1}$, as shown in Figure 2.
SAMPLES AND METHODS

3.1 | Samples

For this study, 14 samples were taken from the nine layers of the O₃w-S₁l shales, eight samples from the JY-A Well and six samples from JY-B Well, to cover a variety of TOC contents and mineral compositions. Details regarding the wells, including burial depth, lithology, TOC content, mineral composition, and sampling location are listed in Table 1. The sampling depths ranged from 2273.55–2618.18 m, with a broad range of TOC values from approximately 0.63%-5.4%.
3.2 | Methods

3.2.1 | Ar-ion-beam milling and FE-SEM

To investigate the nanoscale OM pore structure in the shale samples, a flat surface on each sample was prepared by Ar-ion-beam milling using a Leica EM TIC 3X Triple Ion Beam Miller. Each sample was cut into a small (10 mm × 7 mm × 3 mm) rectangle and milled until an ultra-smooth rectangular surface was achieved. An FEI Helios NanoLab 660 FE-SEM at the Yangtze University laboratory was used to image the tiny pores in shale reservoirs samples using an accelerating voltage of 2 kV. At 20 000-30 000× magnification, pores with diameters >10 nm can be observed and imaged. At magnifications of 30 000-100 000×, some OM pores with diameters of 5-10 nm and a few OM pores varying in size from 2 to 5 nm can be visualized.

3.2.2 | Statistical analysis of OM pores on FE-SEM images

By conducting statistical analyses using a combination of ImageJ software and manual identification of track pore traces in OM particles, we investigated the structural characteristics of OM pores, such as pore shape, pore quantity, and pore size distribution, and calculated the cross-sectional area ratio of the OM pores to the corresponding single-particle OM in the SEM images (ØOMP/OM). The OM pore size refers to the pore diameter of the equivalent circle and is calculated by equating the cross-sectional area of the OM pores in the FE-SEM image of the shale sample to the area of a circle; ØOMP/OM is the ratio of the total cross-sectional area of all OM pores to the cross-sectional area of the corresponding OM particles in every FE-SEM image of each shale sample.

ImageJ software was used for image processing. Binary images of the high-resolution SEM images of the OM pores were created, which were then used to identify and count the OM pores in the images. Determining the thresholds of the grayscale range, which can have a significant influence on the calculation of ØOMP/OM, is difficult, and the gray value range of the OM pores was examined repeatedly to achieve the most comprehensive and correct identification. For each examination, the appropriate gray value was selected and slight adjustments to the gray threshold were made. For areas in images that were not recognized by the processing software, manual adjustments and tracking were required to identify the OM pores.

The organic porosity in shale is an important parameter for shale reservoir evaluations that can provide decisive evidence for the selection of favorable shale gas drilling targets. Because organic porosity cannot be directly determined, the relationships of mass-volume-density-OM content are used to derive the calculation equation for calculating the volume fraction of the total OM in a shale reservoir. In addition, the OM pore volume ratio (ie, the ratio of the total OM pore volume to the total OM volume) is used to obtain the organic porosity. In this study, the OM pore volume ratio (%) with sizes >10 nm was represented by ØOMP/OM (%), which was obtained via statistical analysis of the FE-SEM images, while the OM pore volume ratio with sizes <10 nm was estimated by gas physisorption.

3.2.3 | Low-pressure gas isothermal adsorption

Gas adsorption is usually used to quantify micropores and mesopores in shale. In this study, low-pressure nitrogen isothermal sorption was performed on crushed samples (60-80 mesh) using a Quantachrome Autosorb iQ Surface Area and Porosity Analyzer at the Key Laboratory of Tectonics and Petroleum Resources, Ministry of Education, China University of Geosciences (Wuhan). Prior to analysis, the samples were dried in a vacuum oven at 383.15 K for 10 hours to remove all volatile substances and free water and were then tested at liquid nitrogen temperature (77.3 K) and relative pressures (P/Po) of 0.0002-0.995 (39 pressure points) and 0.995-0.10 (24 pressure points) for the adsorption and desorption processes, respectively. Nitrogen data were processed using the Brunauer-Emmett-Teller (BET) method for specific surface area, the Barrett-Joyner-Halenda (BJH) method for pore volume and pore size distribution of the mesopores and macropores, and the density functional theory (DFT) method for micropores and mesopores. In this work, the quenched solid density functional theory (QSDFT) and BJH methods were used to describe the pore size distribution and pore volume, respectively.

Both principle and application of the low-temperature carbon dioxide adsorption test are similar to that of nitrogen adsorption, except that carbon dioxide is used as an adsorbent to measure the adsorption capacity of carbon dioxide at 273.15 K (ice-water bath) at different relative pressures. The relative pressure range during adsorption was 0-0.03 (40 pressure points). The distribution of micropores (pore sizes of 0.3-1.47 nm) was calculated by the DFT model using carbon dioxide test data.

4 | RESULTS

4.1 | Microscopic observation

The FE-SEM images of the 14 shale samples from wells JY-A and JY-B in the Jiaoshiba Block show that various types of nanopores developed in the shale, including clay mineral pores, intergranular pores of brittle minerals, dissolution pores and OM pores. In organic-rich shales, the OM pores are the most important and abundant pore type and were
found in OM particles, organic-clay mineral complexes, and the OM within pyrite framboids (Figure 3). The shape of the OM pores varies from nearly spherical (Figure 3A) to irregularly polygonal (Figure 3B), with slightly irregular ellipsoids being the most common shape (Figure 3A). The distribution and development of the OM pores in the OM particles are highly heterogeneous. The OM pores in organic-rich shale samples are mostly compound pores and sponge pores with pore diameters ranging from 2 to 900 nm. In the same shale samples, some OM particles have fewer pores (Figure 3F). Few of the OM particles in some samples developed sharp angular pores (Figure 3E). These OM particles cross-section usually had a large area, clear boundaries, and few clay mineral fragments or secondary mineral crystals at their center.

Clay minerals are important for the storage and evolution of OM in shales. The O3w-S1l1 shales in the Jiaoshiba Block have a high clay minerals content, with fine particle sizes, a large specific surface area, a high adsorption capacity, and a strong surface activity, allowing adsorption of a large number of organic substances. The OM can be stored in micropores of the clay minerals or preserved by chemical bonding with clay, which may have a catalytic effect on OM.

**FIGURE 3** Typical SEM images of OM pore structure of the O3w-S1l1 shales in the Jiaoshiba Block C, E. Well JY-A, 2288.06 m, the upper part of S1l1, TOC is 1.64%; H. Well JY-A, 2306.92 m, the upper part of S1l1, TOC is 1.97%; G. Well JY-B, 2356.00 m, the upper part of S1l1, TOC is 2.11%; I. Well JY-A, 2325.13 m, the lower part of S1l1, TOC is 3.13%; D, F. J. Well JY-A, 2349.17 m, the lower part of S1l1, TOC is 4.98%; B, Well JY-B, 2606.65 m, the lower part of S1l1, TOC is 4.07%; A, L. Well JY-A, 2358.35 m, O3w, TOC is 4.57%; K. Well JY-B, 2617.58 m, O3w, TOC is 4.53%
The OM-hosted pores in clay minerals, that is, elongated strip-like pores, are oriented in the extension direction of the clay minerals (Figure 3B). The clay mineral fragments located in the OM particles indicate that the OM has been transported into this pore, as shown in Figure 3L. Additionally, the pyrite framboids in the shale samples can not only provide reservoir space but can also be related to the origin of the OM. The pyrite framboids are secondary sedimentary minerals, while the OM in the pyrite intergranular material had migrated. Due to the limitations of the original pores (pyrite intergranular pores) in size, these OM pores are generally small, with pore sizes ranging from 10 to 30 nm (Figure 3A). The SEM images show that some of the OM within the many mineral crystals have OM pores with a regular edge, and these pores are pseudo-OM pores left by exfoliation of the mineral particles (Figure 3C).

The SEM images show notable differences in OM pore shapes and OM pore development for the three sets of shales, that is, the O3w and the lower and upper parts of S1l1. The OM pores in the O3w shale are well-developed, with the majority being irregular polygons with angular shapes. The number of nanopores within the grains of OM is large, and the pore sizes are relatively small with a uniform distribution (Figure 3K,L). Similarly, the high-quality shale in the lower part of S1l1 also contains an abundance of OM pores; however, the pore shapes are mostly elliptical and subrounded. The pore sizes in the lower part of S1l1 are larger and vary more widely, with a large number of macropores and a few micron-sized OM pores (Figure 3I,J). Compared with these two shale intervals, the OM pore development of the upper part of the S1l1 shale is poor, with low pore densities. The pore morphology is mostly elliptical and subrounded, with a few irregular shapes (Figure 3G,H). Compared to the shale samples of O3w, the OM pore sizes of the S1l1 shales are generally larger.

4.2 Statistical analysis of the shale pore structure

In total, 194 SEM images were selected for statistical analysis (10–20 images per sample), and a total of 179 281 OM pores were counted (Figure 4; Table 2).

4.2.1 Statistical analysis of OM pores

Of the count data, mainly those with pore sizes >10 nm were selected for analysis, as shown in Tables 2 and 4 and Figures 5 and 6.

Figure 5 shows the pore size distribution histograms of the more than 110 000 OM pores observed and counted in
the selected shale samples. Regarding the distribution of the OM pores with diameters from 10 to 900 nm in the three sets of shales, in O3\textsubscript{w} and the lower and upper parts of S\textsubscript{1l}, an increase with a decrease in pore size is seen (Figure 5A). The number of OM pores in the size range of 10-30 nm accounted for 80%-90% of the statistical data for each layer.

The pore size distribution in the lower part of the S\textsubscript{1l} shale is more heterogeneous than that of the other two sets of shales, with the number of OM pores with pore sizes of 10-15 nm accounting for as much as 50%. In addition, the proportion of OM pores with pore sizes >100 nm is the highest out of the three sets of shales.

TABLE 2 FE-SEM images and quantity of OM pores in the selected shale samples from O\textsubscript{3w}-S\textsubscript{1l}, Jiaoshiba Block

| Layers   | Average TOC (%) | Samples | Photos | Statistics quantity of OM pores |
|----------|-----------------|---------|--------|--------------------------------|
|          |                 |         |        | pore sizes of 10-30 nm | pore sizes of >10 nm | All data |
| 9        | 0.74            | 2       | 20     | 9302                     | 10 006               | 10 971   |
| 8        | 1.87            | 2       | 33     | 9933                     | 11 987               | 18 564   |
| 7        | 1.97            | 1       | 20     | 8805                     | 12 449               | 15 153   |
| 6        | 1.40            | 1       | 15     | 6192                     | 8416                 | 8629     |
| The upper part of S\textsubscript{1l} | 1.43            | 6       | 88     | 34 232                   | 42 858               | 53 317   |
| 5        | 3.13            | 1       | 17     | 6753                     | 7626                 | 10 084   |
| 4        | 2.77            | 1       | 10     | 6217                     | 6889                 | 9345     |
| 3        | 4.54            | 3       | 39     | 34 562                   | 28 152               | 40 014   |
| The lower part of S\textsubscript{1l} | 3.90            | 5       | 66     | 47 532                   | 42 667               | 59 443   |
| 1 ( O\textsubscript{3w} ) | 4.56            | 3       | 40     | 41 162                   | 54 212               | 66 521   |
| Total    | /               | 14      | 194    | 122 926                  | 139 737              | 179 281  |

FIGURE 5 Histogram of the OM pore diameters of the sampled shales from O\textsubscript{3w}-S\textsubscript{1l}, Jiaoshiba Block
As shown in Figure 5B, the pore sizes of the OM pores of each layer are concentrated at 30 nm. In the high-quality section (TOC > 3%), the proportion of the OM pore sizes <15 nm is larger, including the 1st, 3rd, 4th, 5th, and 8th layers. Although the 6th, 7th, 8th, and 9th layers were classified into one group due to the relatively low TOC and lower OM pore numbers, the characteristics of the OM pores in each layer vary considerably. The OM pores of the 8th and 9th layers are concentrated in mesopores, and the number of macropores is small. The 6th and 7th layers also mainly contain mesopores; however, there are a certain number of macropores and the fraction of OM pores with pore sizes >200 nm, in particular, reach 3.7% in the 7th layer. The pore size distributions of the

**FIGURE 6** Relationship between the average ØOMP/OM and TOC content

**FIGURE 7** Relationships of gas isothermal sorption data with clay, quartz and TOC content
OM pores in the 3rd, 4th, and 5th layers have similar features. The proportion of OM pores with sizes <30 nm is >90% and >1.5% for pores >100 nm, of which the 3rd layer accounts for 3%. In contrast, the number of OM pores in the 1st layer decreases with an increasing average pore size; however, the total amount of the decrease is relatively small, and the average pore size distribution is more uniform; however, there are fewer macropores, and the proportion of pores with sizes >60 nm is <1%, and the number of pores with sizes >200 nm is extremely small (0.03%).

4.2.2 | Gas isothermal sorption data

Figure 7 shows that the pore specific surface area and pore volume of the shale samples have no obvious relationship with the clay content. Both show a rough positive correlation with quartz content and a good linear correlation with TOC content, which indicate that the pore structure of the shales is closely related to the OM. Additionally, SEM images show that abundant pores are developed in OM, while relatively few inorganic pores were observed. Therefore, the pore size distributions calculated from the experimental results of gas adsorption can be used to derive the pore size distributions of the OM pores in the shale samples.

The pore sizes of OM pores in Jiaoshiba Block shales are mainly concentrated in the range of 4-30 nm, and the micropores (<2 nm) are well-developed (Figure 8). The pore volume of the O3w shale is largely composed of both micropores and mesopores, while the number of pores >50 nm decreases sharply, which is consistent with the observations and statistical results of the SEM image analysis of OM pores. Overall, the increase in pore volume (ie, the number of pores) is positively correlated with the TOC content, especially in the size range of 4-30 nm. The specific surface area of the pores with sizes <30 nm calculated by the DFT method occupies >90% of the surface area calculated by the BET method, and the proportion of their respective volume reaches 60%-70% (Figure 9), which is consistent with the results indicating that the small pores (especially OM pores) provide the main storage space.

4.3 | Organic porosity estimation

The organic porosity of a shale reservoir is the ratio of the total OM pore volume to the bulk volume of the shale reservoir. Currently, organic porosity cannot be directly measured by instruments. In this study, we used the relationships between the mass, volume, and density of the OM in the shale, combined with the carbon dioxide and nitrogen adsorption data, to estimate the organic porosity.

4.3.1 | Organic porosity calculation using ØOMP/OM

Organic porosity (ψs) can be estimated using the relationships between related parameters of shale reservoirs, as shown in Equation (1). We used the ØOMP/OM instead of the ratio of the OM pore volume to the OM volume, and, therefore, the resulting estimate will have a certain error. First, we calculated the ratio of the OM volume to the total volume of the shale (Nb) using the TOC content and then obtained ØOMP/OM (Ks) (Equation 2). Finally, the equation for calculating the organic porosity (ψs) was obtained as follows:

\[
\phi_s = \frac{V_{op}}{V_r} = \frac{V_{op}}{V_o} \cdot \frac{V_o}{V_r} \approx K_s \cdot N_b = K_s \cdot C \cdot \text{TOC} \cdot \frac{\rho_r}{\rho_o} \cdot 100\%, \tag{1}
\]

\[
N_b = \frac{V_o}{V_r} = \frac{m_o}{\rho_o} \cdot \frac{\rho_r}{m_r} = \frac{m_o}{m_r} \cdot \frac{\rho_r}{\rho_o} = C \cdot \text{TOC} \cdot \frac{\rho_r}{\rho_o} \cdot 100\%, \tag{2}
\]

\[
K_s = \frac{S_o}{A_o} \cdot 100\%, \tag{3}
\]

where \(m_r, V_r, \rho_r\) refer to the rock mass (g), volume (cm\(^3\)), and density (g/cm\(^3\)) of the shale reservoir, respectively; \(m_o, V_o, \rho_o\) are the OM mass (g), volume (cm\(^3\)), and density (g/cm\(^3\)) in the shale reservoir, respectively; and, \(C\) is the conversion coefficient of the organic carbon in the OM in the shale. According to the analysis of the purified OM elements in the shale, the conversion
coefficient \( C \) in the high-evolution stage of the \( O_{3w-S1} \) shales ranges from 1.05 to 1.15. In this study, a conversion coefficient \( C \) of 1.1 was adopted to calculate the organic porosity.\(^46\) Based on relevant research on kerogen density,\(^47\) the range of the density of the OM (mainly pyrobitumen) with well-developed nanopores in the high-evolution \( O_{3w-S1} \) shales is 1.0-1.2, with a calculated value of 1.1 \( \text{g/cm}^3 \). In the formula, TOC is the organic carbon content of the shale (%), \( S_O \) is the OM pore area (\( \text{nm}^2 \)) in the cross-section of the OM particles in the shale, and \( A_o \) is the cross-sectional area of the OM particles (\( \text{nm}^2 \)). Values for \( S_O \) and \( A_o \) were obtained from the FE-SEM images via statistical analysis.

The results of the organic porosity calculations and \( \bar{\phi}_{OMP/OM} \) of the single-particle sections derived from the multiple SEM images via statistical analysis are listed in Table 4. Average \( \bar{\phi}_{OMP/OM} \) values range from 19.02% to 22.87% in the \( O_{3w} \) shale, from 17.84% to 23.34% in the lower part of the \( S_1 \) shale, and from 12.62% to 18.45% in the upper part of the \( S_1 \) shale. According to the results from Equation (1), the average organic porosity of the \( O_{3w} \) shale is approximately 2.15%-2.61%, that in the lower part of the \( S_1 \) shale is 1.30%-2.91%, and that in the upper part of the \( S_1 \) shale is 0.27%-0.95%.

### 4.3.2 Organic porosity calculated using gas adsorption

Through statistical comparative analysis, we found that the FE-SEM images can show the OM pores of the shale clearly at a magnification of \( >30,000 \times \). In this study, we imaged OM pores with pore sizes of 2-900 nm. The very low amount of OM pores with pore diameters of 2-5 nm could be observed even with extremely high-resolution FE-SEM observation, while the number of OM pores with pore diameters ranging from 5 to 10 nm was often not resolved completely. OM pores with pore sizes of 10-900 nm, however, were clearly resolved in all FE-SEM images, and, therefore, the following statistical results include only OM pores with pore diameters >10 nm.

The FE-SEM images and gas adsorption results show that a large number of OM pores are <10 nm and play an important role along with unobserved micropores (pore diameter <2 nm). These micropores are abundant and have a large specific surface area, which can control the abundance of shale-adsorbed gas. The micropores are mainly present in the highly mature OM, which, in turn, can be used to estimate the number of micropores in the high-maturation to over-maturation stages. Therefore, we combined the gas adsorption and TOC data to calculate the number of OM pores <10 nm.

The linear relationship between the pore volume of pores with sizes <10 nm (calculated by the DFT method), and the TOC of the samples was established, and their functional relation was obtained, as shown in Figure 10. In areas or strata with similar geological conditions (such as sedimentary environments, petrology, geochemistry, and diagenesis), it can be assumed that the volume of the inorganic pores with pore sizes <10 nm is a fixed value regardless of whether the TOC changes, that is, 0.0067 cc/g in Figure 10 \( (y = 0.0018x + 0.0067) \). Then, the volume of the OM pores <10 nm and the ratio of the OM pores volume to the total pore volume can be calculated, as summarized in Table 3. The results show that the OM pore volume...
| Sample  | TOC (%) | Total porosity (%) | Total pore volume (cc/g) | Pore size <10 nm | Calculated OM pore volume (cc/g) | Proportion of OM pore volume (%) | Ratio of OM pore volume to total pore volume (%) | Ratio of OM pore volume to OM volume (%) |
|---------|---------|--------------------|--------------------------|-----------------|----------------------------------|---------------------------------|---------------------------------------------|------------------------------------------|
| A-9-8   | 0.84    | 2.92               | 0.0196748                | 0.096867        | 0.0029867                        | 30.83                           | 15.18                                       | 19.77                                    |
| B-9-6   | 0.63    | 2.80               | 0.0210791                | 0.0089129       | 0.0022129                        | 24.83                           | 10.50                                       | 17.34                                    |
| A-8-7   | 1.64    | 3.65               | 0.0209693                | 0.0091862       | 0.0024862                        | 27.06                           | 11.86                                       | 10.02                                    |
| B-8-5   | 2.11    | 4.70               | 0.0200000                | 0.0113843       | 0.0046843                        | 41.15                           | 23.42                                       | 19.55                                    |
| A-7-6   | 1.97    | 4.73               | 0.0204667                | 0.0091198       | 0.0024198                        | 26.53                           | 11.82                                       | 10.81                                    |
| B-6-4   | 1.40    | 4.33               | 0.0242978                | 0.0081659       | 0.0014659                        | 17.95                           | 6.03                                        | 7.05                                     |
| A-5-5   | 3.13    | 4.73               | 0.0311335                | 0.0125745       | 0.0068745                        | 46.72                           | 18.87                                       | 10.89                                    |
| A-4-4   | 2.77    | 3.31               | 0.0235949                | 0.0092974       | 0.0025974                        | 27.94                           | 11.01                                       | 5.01                                     |
| A-3-3   | 4.98    | 5.30               | 0.0310566                | 0.0158816       | 0.0091816                        | 57.81                           | 29.56                                       | 12.59                                    |
| A-3-2   | 4.58    | 6.30               | 0.0289887                | 0.0148907       | 0.0081907                        | 55.01                           | 28.25                                       | 15.30                                    |
| B-3-3   | 4.07    | 5.97               | 0.0246831                | 0.0138257       | 0.0071257                        | 51.54                           | 28.87                                       | 16.82                                    |
| A-1-1   | 4.57    | 6.02               | 0.0305749                | 0.0140479       | 0.0073479                        | 52.31                           | 24.03                                       | 12.66                                    |
| B-1-2   | 4.53    | 6.37               | 0.0299693                | 0.0159453       | 0.0092543                        | 57.98                           | 30.85                                       | 17.36                                    |
| B-1-1   | 4.59    | 6.39               | 0.0316812                | 0.0158862       | 0.0091862                        | 57.83                           | 29.00                                       | 16.21                                    |
of the pores with sizes <10 nm accounts for 20%-60% of the pore volume <10 nm and 6%-30% of the total pore volume.

### 4.3.3 | Total organic porosity

Since $\Phi_{OMP}$ was only calculated for pore diameters >10 nm, the total organic porosity of the shale gas reservoir in the study area was estimated using the OM pore volume ratio of the pores with sizes <10 nm, as calculated in the previous section. Based on the organic porosity calculated for $\Phi_{OMP}$, plus the estimates of the OM pore volume of the pores with sizes <10 nm using gas adsorption data, the average organic porosity of the O$_3$w shale is approximately 4.13%, which is approximately 66% of the total porosity. That of the lower part of the S$_1$1 shale ranges between 1.67% and 4.47%, accounting for 50%-84% of the total porosity, and that of the upper part of the S$_1$1 shale ranges between 0.56% and 1.81%, accounting for 19%-38% of the total porosity (Table 4; Figure 11).

### 5 | DISCUSSION

#### 5.1 | OM pore development and origin

The OM pores in the O$_3$w-S$_1$1 shales of the Jiaoshiba Block are mainly mesopores and micropores. The main reason may be that with the increase in thermal evolution, especially in the stages when large amounts of gas are generated, aromatization is enhanced and the order degree of benzene ring accumulation increases, resulting in intense micropore development. Previous studies on nitrogen adsorption on immature shale show that when the maturity increases the number of micropores increases gradually, indicating that micropores are mainly formed during the maturation of OM. The degree of thermal evolution ($R_o$) of the shales in the Jiaoshiba Block is 2.5%-3.0%, indicating over-maturation within the dry gas generation window.

According to the gas adsorption data, the OM pore volume of <10 nm pores occupies 6%-20% of the OM volume, and the maximum $\Phi_{OMP}$ of the pores with size >10 nm, based on statistical analysis, vary from 25% to 45%, with averages of 12%-23%. According to the carbon/hydrogen

| Samples | Layer | Depth (m) | TOC (%) | Apparent density (g/cm$^3$) | Total porosity (%) | The range of $\Phi_{OMP}$ | The average of $\Phi_{OMP}$ (%) |
|---------|-------|-----------|---------|-----------------|-------------------|------------------------|--------------------------|
| A-9-8   | 9     | 2273.55   | 0.84    | 2.66            | 2.92              | 2.49-25.36             | 14.03                    |
| B-9-6   | 9     | 2533.99   | 0.63    | 2.70            | 2.80              | 5.87-24.87             | 15.93                    |
| A-8-7   | 8     | 2288.06   | 1.64    | 2.64            | 3.65              | 4.82-31.04             | 17.73                    |
| B-8-5   | 8     | 2556.00   | 2.11    | 2.67            | 4.70              | 2.88-25.62             | 12.62                    |
| A-7-6   | 7     | 2306.92   | 1.97    | 2.62            | 4.73              | 6.85-36.49             | 18.45                    |
| B-6-4   | 6     | 2572.30   | 1.40    | 2.65            | 4.33              | 2.81-34.44             | 15.67                    |
| A-5-5   | 5     | 2325.13   | 3.13    | 2.62            | 4.73              | 5.00-30.82             | 18.26                    |
| A-4-4   | 4     | 2338.55   | 2.77    | 2.63            | 3.31              | 8.08-27.88             | 17.91                    |
| A-3-3   | 3     | 2349.17   | 4.98    | 2.50            | 5.30              | 4.82-45.00             | 23.34                    |
| A-3-2   | 3     | 2353.92   | 4.58    | 2.54            | 6.30              | 2.22-36.08             | 19.56                    |
| B-3-3   | 3     | 2606.65   | 4.07    | 2.52            | 5.97              | 3.16-30.43             | 17.84                    |
| A-1-1   | 1     | 2358.35   | 4.57    | 2.50            | 6.02              | 4.81-34.59             | 22.87                    |
| B-1-2   | 1     | 2617.58   | 4.53    | 2.50            | 6.37              | 4.03-28.36             | 19.02                    |
| B-1-1   | 1     | 2618.18   | 4.59    | 2.49            | 6.39              | 12.79-33.74            | 20.58                    |

Note: $\Phi_{OMP}$ refers the cross-section area ratio of the OM pores to the corresponding single-particle OM.
ratio, the conversion rate of bitumen or oil-generated gas is 50% at most.\textsuperscript{46} It needs to be clarified whether the OM of the O$_3$W-S$_1$\textsuperscript{l1} shales in the Jiaoshiba Block is mainly bitumen or crude oil. During the stage of the O$_3$W-S$_1$\textsuperscript{l1} shales formation in which the OM was transformed into petroleum, large amounts of crude oil and bitumen were retained in the shale, and shale gas is thought to have been generated by thermal cracking in the later high-evolution stage.\textsuperscript{12,52} Subsequently, OM pores developed in the pyrolyzed bitumen.

The OM pores in highly mature to over-mature organic-rich shales are generally considered to be formed by secondary cracking of crude oil or bitumen,\textsuperscript{12} and some OM pores have been observed in kerogen.\textsuperscript{14} According to the origin of the OM, it can be divided into in situ depositional OM and micro-migrated OM.\textsuperscript{51} The in situ depositional OM is the original hydrocarbon generation material in the shale, mainly including kerogen and its evolvement products (such as solid bitumen and pyrobitumen), which are distributed inside the kerogen without micro-migration. Other types of OM are filling mineral pores or fossil voids which are transported by bitumen or the oil formed by kerogen. As the thermal maturity increases further, the bitumen or oil can evolve into solid bitumen and pyrobitumen, resulting in the generation of natural gas.\textsuperscript{12,54} It is important to identify and distinguish the properties of the OM that hosts OM pores for studying the genesis and evolution of OM pores. However, the type of OM in the organic-rich shale of the Jiaoshiba Block is mainly marine oil-prone kerogen within the dry gas generation window, and the composition and morphological characteristics of kerogen and bitumen are extremely similar and difficult to distinguish.\textsuperscript{53} Therefore, scholars have proposed several petrographic criteria from SEM photomicrographs to separate depositional versus micro-migrated OM. It is generally believed that the secondary mineral particles (eg, quartz and calcite) with perfect crystallinity are formed in OM particles, indicating that the OM is migrated bitumen or the solid residue from crude oil cracking rather than the original sedimentary OM (Figure 3A). This is because the formation of secondary minerals mainly occurs during diagenesis, when crystals grow in the mineral pores, and, subsequently, bitumen or oil migrates into the mineral pores.\textsuperscript{14,53} A well-developed spongy texture containing numerous OM pores was usually observed in the identified transported OM, which can be used as an important sign for identification (Figure 3K).

According to the SEM images, the OM in the O$_3$W-S$_1$\textsuperscript{l1} shales of the Jiaoshiba Block is most likely migrated OM, with OM pores developed in the migrated OM particles. As migrated OM fills the three-dimensional interconnected mineral pores, it can attain better connectivity in three-dimensional space compared to in situ deposited OM.\textsuperscript{7,11}

### Table 4

| Samples | Layer | Statistical results showing the range and average surface pore rate of OM and the organic porosity for shale |
|---------|-------|----------------------------------------------------------------------------------------------------------|
|         |       | The total organic porosity to total porosity (%) |
|          | The range of organic porosity (%) | The average of organic porosity (%) | The organic porosity of pore size <10 nm (%) | The total organic porosity (%) | Proportion of organic porosity to total porosity (%) |
| 0.06-0.57 | 0.31 | 0.42 | 0.74 | 25.22 |
| 0.10-0.42 | 0.27 | 0.29 | 0.56 | 20.14 |
| 0.21-1.34 | 0.77 | 0.43 | 1.20 | 32.85 |
| 0.16-1.44 | 0.71 | 1.10 | 1.81 | 38.54 |
| 0.35-1.89 | 0.95 | 0.56 | 1.51 | 32.00 |
| 0.10-1.28 | 0.58 | 0.26 | 0.84 | 19.44 |
| 0.41-2.53 | 1.50 | 0.89 | 2.39 | 50.53 |
| 0.59-2.03 | 1.30 | 0.34 | 1.67 | 50.37 |
| 0.60-5.60 | 2.91 | 1.57 | 4.47 | 84.36 |
| 0.26-4.20 | 2.28 | 1.78 | 4.06 | 64.38 |
| 0.32-3.12 | 1.83 | 1.72 | 3.55 | 59.50 |
| 0.55-3.95 | 2.61 | 1.45 | 4.06 | 67.43 |
| 0.46-3.21 | 2.15 | 1.96 | 4.12 | 64.63 |
| 1.46-3.86 | 2.35 | 1.85 | 4.21 | 65.81 |

### 5.2 | $\theta_{OMP/OM}$ and organic porosity

The statistical results and microscopic observations suggest an internal connection between microscopic features and macroscopic phenomena, and, therefore, that microscopic characteristics of shale can be used to explain macroscopic
phenomena. At the macroscopic level, Yang et al. report that the methane sorption capacity and gas content of shales is usually positively correlated with the TOC content of Wufeng-Longmaxi shales of the Fuling gas field. At the micro-level, $\Omega_{\text{OMP/OM}}$ and organic porosity have the same relationship with TOC content. The OM pores developed in the O$_{3\text{w}}$-S$_1^{\text{w}}$ shales of the Jiaoshiba Block have a positive correlation with TOC content (Figures 5, 6 and 11), with the higher TOC samples generally having higher $\Omega_{\text{OMP/OM}}$ and organic porosity. This suggests that the better the development of OM pores, the higher the contribution ratio to the shale porosity and the greater the influence on the methane sorption capacity and gas content. In addition, the content of migrated OM within the network of the O$_{3\text{w}}$-S$_1^{\text{w}}$ shales decreases from bottom to top. The interconnected OM pore network is correspondingly reduced, and the shale reservoir quality gradually deteriorates. The decline of reservoir quality also results in a decrease of the shale gas content.

The OM pores in the upper part of the S$_1^{\text{l}}$ shale are less developed and distributed sparsely, compared with the other two sets of shales (Figures 5 and 6), which is most likely due to the presence of thin layers of siltstone and fine sandstone within this shale interval. In addition, the TOC content in the upper part of S$_1^{\text{l}}$ is low overall, contributing to the smaller cross-section area of the OM particles and resulting in a smaller number and sparser density of the OM pores in the OM particles. Another reason for the relatively poor development of the OM pores in the upper part of the S$_1^{\text{l}}$ shale may be the OM composition, which differs from the other two shale intervals.

Overall, the organic porosity and the total porosity are positively correlated, especially in the high-quality O$_{3\text{w}}$ and lower part of S$_1^{\text{w}}$ shales (Table 4), in which the proportion of the organic porosity to the total porosity increases with increasing TOC content; in the sub-quality upper part of S$_1^{\text{l}}$ shale; however, this correlation was not observed. The total porosity is approximately 4% in the 8th-9th layers, with a proportion of organic porosity of only 20%-30%. In contrast, in the 4th-5th layers, the organic porosity is as high as 50% of the total porosity, which is consistent with the observation that a larger number of inorganic pores are present in the sub-quality section (6th-9th layers), with some layers even dominated by inorganic pores. In the 1st and 3rd layers, OM pores occupy the majority of the reservoir space, which is one of the important reasons why they are high-quality gas production reservoirs in the Fuling shale gas field.

5.3 | Factors influencing OM pores shape variations between Wufeng and Longmaxi shales

Figures 3 and 4 show the differences in OM pore shapes of Wufeng and Longmaxi shale pores. Near-circular to elliptical outlines of OM pores are common in the OM grains of the S$_1^{\text{l}}$ shale, and the pore diameter varies from several nanometers to hundreds of nanometers. Some of the OM pores within the OM grains of the lower part of the S$_1^{\text{w}}$ shale exhibit larger, more complex shapes that are possibly the result of the merging of simpler forms during pore development (Figure 3D). The pores with complex shapes appear to be more interconnected than those with simpler shapes.

The majority of the OM pore shapes in the O$_{3\text{w}}$ shale samples are irregularly angular, which is notably different from the elliptical and near-circular shapes of the shale samples along the vertical-bedding or parallel-bedding plane in the overlying Longmaxi Formation. This phenomenon indicates that the vertical load compaction of the Yanshanian-Himalayan tectonic uplift and erosion process may not be the main cause for the OM pore deformation of the O$_{3\text{w}}$ shale. It is worth noting that the observation of the shale cores from several wells in the Jiaoshiba anticline shows that O$_{3\text{w}}$ has more structural fractures and calcite and quartz veins filled in the fractures compared to the Longmaxi Formation. In the middle-lower part of O$_{3\text{w}}$, compressed fractured layers and structural phenomena reflecting the layered compressional decollement (such as secondary detachment surface scratches, microdetachment folds, and imbricate structures) are also developed. This may indicate that against the background of the tectonic stress of the regional compressive nappe, O$_{3\text{w}}$ is located at the bottom of a set of soft formations of the O$_{3\text{w}}$-S$_1^{\text{w}}$ shales, which are located above the underlying hard strata of carbonates, and the bottom of the O$_{3\text{w}}$ shale is the main detachment surface. This results in O$_{3\text{w}}$ having experienced more notable tectonic compression, layered decollement, and reconstruction compared with the S$_1^{\text{l}}$ shale, which caused some degree of overpressure release. Then, the OM pore morphology in the O$_{3\text{w}}$ shale was subjected to lateral stress extrusion deformation and compression. This may be the main reason that the majority of the OM pore shapes in the O$_{3\text{w}}$ shale samples are irregular prismatic and angular, and the $\Omega_{\text{OMP/OM}}$ in the O$_{3\text{w}}$ shale is lower than that in the lower part of S$_1^{\text{l}}$ with a same TOC content.

6 | CONCLUSION

1. Nanoscale OM pores are the dominant pores in the marine organic-rich shales of O$_{3\text{w}}$-S$_1^{\text{l}}$ in the Jiaoshiba Block, Sichuan Basin. Some of the pores in the OM are associated with clay minerals and pyrite frambooids, and the OM-hosted pores are micro-migrated pyrobitumen.

2. The OM pores in the O$_{3\text{w}}$-S$_1^{\text{w}}$ shales of the Jiaoshiba Block are mainly composed of mesopores and micropores, mostly concentrated in the pore size range <30 nm, and the shapes are near-circular, elliptic, or irregularly polygonal. Significant variations exist in the nanoscale
OM pore structures among the three sets of studied shales. The majority of OM pores in the O3 shale are irregular polygons, while elliptical and subrounded OM pores were observed in the S1 shale. Generally, the OM pores in the O3 shale have a smaller pore size and are more abundant; the size of the OM pores in the high-quality shale (TOC > 3.0%) in the lower part of the S1 shale is relatively large overall, and the OM pores in the upper part of the S1 shale are sparsely developed.

The organic porosity of the Jiaoshiba Block shale, calculated using the statistical data and gas adsorption data, accounts for 50%-84% of the total porosity in the high-quality shales of the O3w and the S1l lower part, and 19%-38% of the total porosity in the sub-quality shale of the S1l upper part. Furthermore, in the high-quality section, the organic porosity with pore sizes >10 nm, calculated using the O3/OMP/OM, accounts for 31%-55% of the total porosity, and the organic porosity with pore sizes between 0.3 and 10 nm, calculated using the gas adsorption data, accounts for 9%-21% of the total porosity. In the sub-quality section, the organic porosity with pore sizes >10 nm, and the organic porosity with pore sizes between 0.3 and 10 nm, accounts for 11%-31% and 6%-23% of the total porosity, respectively.

Thus, the structural differences in the OM pores in the sampled shales are derived from the TOC content variations and the special location of O3w, the shales of which are located at the bottom of compressive decollement formations, which have experienced layered decollement reconstruction, in turn resulting in partial gas release under overpressure.

ACKNOWLEDGMENTS
The authors would like to thank the National Key R & D program of China (No. 2017YFE0106300), the National Natural Science Foundation of China (Nos. 41672139 and 41902140), China Geological Survey Project Grant (No. DD20190561-1), China National Science and Technology Major Projects (No. 2016ZX0503-4002-003) for financial assistance to this research.

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**How to cite this article:** He C, He S, Zhang T, Yang R, Shu Z, Han Y. Structural characteristics and porosity estimation of organic matter-hosted pores in gas shales of Jiaoshiba Block, Sichuan Basin, China. *Energy Sci Eng*. 2020;8:4178–4195. [https://doi.org/10.1002/ese3.796](https://doi.org/10.1002/ese3.796)