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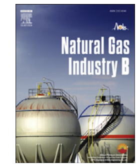

Research article

Characteristics and controlling factors of micropore structures of the Longmaxi Shale in the Jiaoshiba area, Sichuan Basin

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Abstract

Pore structures in shales are a main factor affecting the storage capacity and production performance of shale gas reservoirs. Taking Longmaxi Shales in the Jiaoshiba area of the Sichuan Basin as a study object, we systematically study the microscopic pore structures of shales by using Argon-ion polishing Scanning Electron Microscope (SEM), high-pressure mercury injection and low-temperature nitrogen adsorption and desorption experiments. The study results show that: the Longmaxi Shale in this area are dominated by nano-scale pores which can be classified into organic pores, inorganic pores (intergranular pores, intragranular pores, inter-crystalline pores and dissolution pores), micro-fractures (intragranular structure fractures, interlayer sliding fractures, diagenetic shrinkage joints and abnormal-pressure fractures from organic evolution), among which organic pores and clay mineral pores are predominant and organic pores are the most common; a *TOC* value shows an obvious positive correlation with the content of organic pores, which account for up to 50% in the lower-quality shales with a *TOC* of over 2% where they are most developed; microscopic pore structures are very complex and open, with pores being mainly in cylinder shape with two ends open, or in parallel tabular shape with four sides open and 2–30 nm in diameter, being mostly medium pores. On this basis, factors affecting the micropore structures of shales in this area are studied. It is concluded that organic matter abundance and thermal maturity are the major factors controlling the microscopic pore structures of shales, while the effects of clay mineral content are relatively insignificant.

Keywords: Sichuan Basin; Jiaoshiba area; Early Silurian; Longmaxi Shale; Microscopic pore structure; Controlling factor; Organic pore; Thermal evolution degree; Shale gas

The Lower Silurian Longmaxi Gas Field in the Fuling area of the Sichuan Basin, being the first shale gas field with a daily gas flow rate of 1 MMCM in China, has attracted wide attention from researchers both at home and abroad. Experts and scholars point out that the pore structures of shales are the major factor influencing reservoir capacity and the efficiency of shale gas exploitation [1–3]. For the purpose of studying the microscopic pore structure features of the Longmaxi Shale in this area, we conduct comprehensive studies by using liquid nitrogen adsorption and desorption method and high-pressure mercury injection test, quantitatively characterize the proportions of various types of pores by using the petro-physical model and three types of pore computation models and probe into the major factors controlling the microscopic pore structures of reservoirs in order to evaluate the microscopic pore structures of shales, reveal the shale gas enrichment mechanisms in this area and guide the further exploration and development of marine shale gas in South China.

1. Overview of the study area

Located in Jiaoshiba Town, Fuling District, Chongqing, the Fuling Shale Gas Field is a special positive structure in Wanzhou Synclinorium in the ejective fold belt of the eastern Sichuan Basin, west of Qiyueshan (also called Qiyaoshan)

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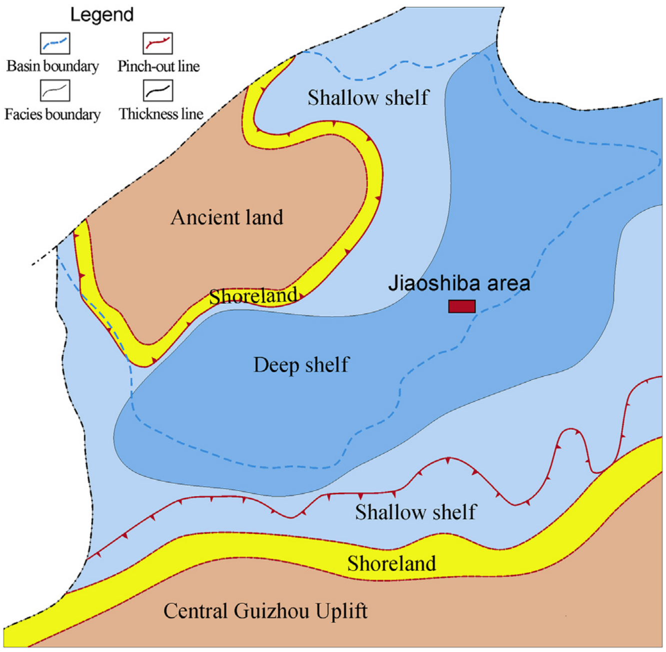


Fig. 1. Sedimentary facies of Upper Ordovician Wufeng Fm. — Lower Silurian Longmaxi Fm. in the Sichuan Basin.

fault, the basin boundary fault. Different from the NE or near SN narrow, high and steep anticlines on its both sides, the main body of Jiaoshiba Structure is a box-like gentle faulted anticline with weak deformation and undeveloped faults; held by two groups of NE and near SN reverse faults, it is diamond-shape and separated from the Qiyueshan Fault by faulted uplifts and faulted sags [4].

Since Sinian, the Sichuan Basin has experienced multi-stage structural movements and thus the present shape come into being ultimately [5,6]. In Late Ordovician, Central Guizhou Uplift in the south, Sichuan Uplift in the west and Xuefeng Palaeo-high in the east turned the area from open sea in the Lower-Middle Ordovician into restricted sea surrounded by uplifts [7,8] (Fig. 1), bringing about the extensive low-energy, undercompensated and anoxybiotic depositional environments. Two global transgressions happened at the end of Ordovician and at the beginning of Silurian and gave rise to the deposition of Longmaxi Fm. Organic-rich shales [9]. The drilling results of the four wells in the Jiaoshiba Structure reveal that the thickness of Longmaxi Fm. Organic-rich shales is 80—120 m; quality shale at the bottom is stably distributed, with 38—42 m in thickness, high *TOC*, and high gas content on site.

2. Pore space types and features

As a kind of low porosity and ultra-low permeability tight reservoirs, shales have much smaller pore diameters (micro-nm scale), complex origins and diverse types. A number of researchers abroad have conducted corresponding studies on them and proposed classification schemes for pore types [10,11]. However, different geologic movements in different areas would result in different pore features. With the help of argon ion polishing SEM technique, we conduct a series of studies on the samples taken from the Longmaxi Shale in Wells JY1, JY2 and JY4 in the Jiaoshiba area, based on the origin, size and shape of pores, we classify the pore space into the types listed in Table 1.

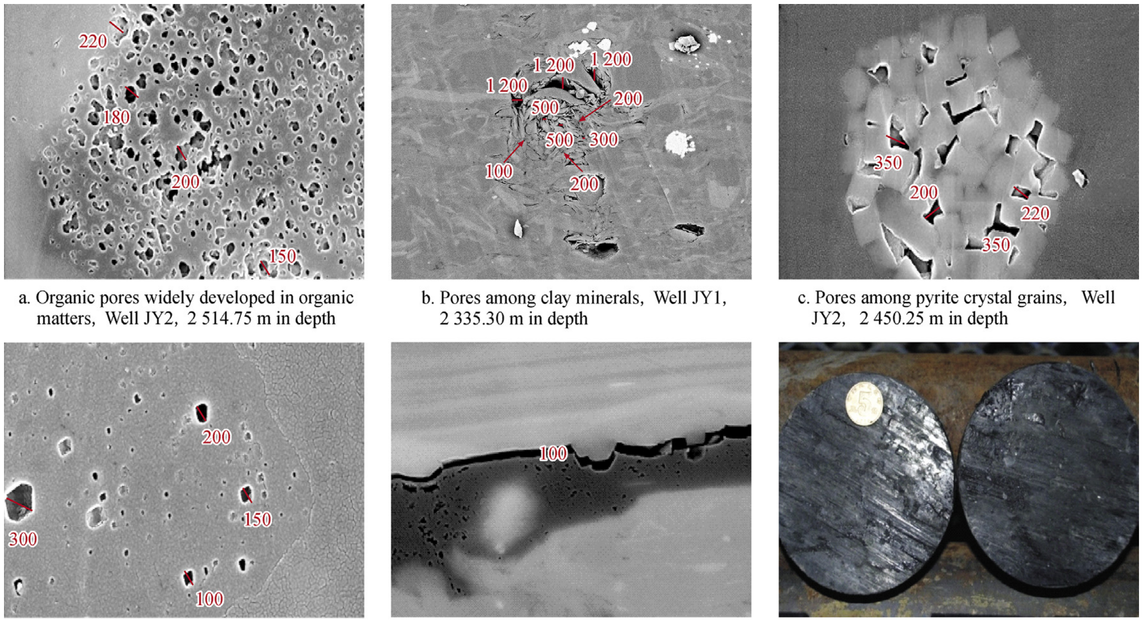
2.1. Pores

2.1.1. Organic pores

Organic pores in shales, formed during the thermal cracking hydrocarbon generation of organic matters, mainly occur between and inside the organic matter grains. Organic nano-pores, one of the most common pore types in shales, are of great significance to the generation and accumulation of shale gas. Observed under microscope, they mainly take the shapes of subsphaeroidal, ellipsoid, gneissic, concave, meniscus and slit (Fig. 2-a), 2—1000 nm in diameter,

Table 1
Features of various types of pores in the Longmaxi Shale of the Jiaoshiba area.

Pore type		Origin	Pore diameter	Common distribution characteristics
	Organic pore	Organic matters maturated and generated hydrocarbon	2—1000 nm	Occur in organic matters with higher thermal evolution degree in subsphaeroidal, oval, concave or gneissic shapes
Inorganic pore	Intergranular pore	Accumulation of mineral grains	5—1200 nm	Often found at the contact of soft and hard particles and in the clay mineral aggregates
	Intragranular pore	Mineral diagenetic transformation	8—100 nm	Often found between laminated or flaky clay mineral grain layers
	Intercrystal pore	Incompact accumulation and erosion during the crystal growth	5—200 nm	Often seen at the contact of framework grains or cement crystals
	Dissolved pore	Denudation	200—1200 nm	Occur in chemically unstable minerals like quartz, feldspar and calcite
Microfracture	Interlayer lamellation fracture	Sedimentation diagenesis and tectonism	10 nm—60 μm	Most filled completely, Member I connects with a high-angle tension crack
	Bed gliding fracture	Sedimentation diagenesis and tectonism	10 nm—40 μm	Smooth bedding plane or bedding plane with scratches or steps, unclosed underground
	Diagenetic shrinkage joint	Diagenesis	5 nm—100 μm	Good connectivity, large opening variation, filled partially
	Organic evolution abnormal pressure fracture	Local abnormal pressure by organic evolution	5 nm—100 μm	Irregular fracture plane, does not occur in group, mostly filled by organic matters



a. Organic pores widely developed in organic matters, Well JY2, 2 514.75 m in depth

b. Pores among clay minerals, Well JY1, 2 335.30 m in depth

c. Pores among pyrite crystal grains, Well JY2, 2 450.25 m in depth

d. Dissolved pores inside calcite grains, Well JY4, 2 570.71 m in depth

e. Microfracture between organic matter and clay, Well JY1, 2 381.91 m in depth

f. Interlayer sliding fractures, Well JY2, 2 521.99 m in depth

Fig. 2. Pore space types of the Longmaxi Shale in the Jiaoshiba area.

representing medium pores. The formation of organic pores is mainly affected by kerogen type, organic abundance and thermal evolution degree, with a surface porosity of 10%–50%, 30% on average. Organic pores can also be affected by burial compaction, it is discovered by scanning of SEM that due to compaction, organic matters at the bottom of Longmaxi Fm. have various irregular shapes, and the organic pores are not isolated but connected to some extent (Fig. 2-a). Organic pores, being lipophilic, are more favorable for the adsorption and accumulation of shale gas. Therefore, substantial organic pores with good connectivity can form favorable micro-channels and thus improve the permeability of shales.

2.1.2. Inorganic pores

2.1.2.1. Intergranular pores. Usually occurring at the contact of mineral grains, intergranular pores are formed due to the incomplete cementation of grains or late diagenetic transformation; Most of them are in polygon and elongation shapes, with a diameter of 5–1200 nm. There are substantial intergranular pores in clay minerals. The clay content of Longmaxi Fm. in this area is 16.6%–62.8%, 40.9% on average; the clay minerals [especially illites (Fig. 2-b)] can form schistose granular aggregates during the deposition and formation of shales, and the particles are connected through their edges [12] and thus form the pores. Observations on samples taken from Well JY1 under microscope show there are substantial schistose granular clay aggregates and pores in the samples, which can serve as storage space for the methane molecules (0.38 nm) and good channels for the percolation of shale gas.

2.1.2.2. Intragranular pores. Intragranular pores are mostly 8–100 nm in diameter. Substantial intragranular pores could be generated during the transformation of chemically unstable minerals like montmorillonite into illite-montmorillonite mixed layers or illites under depositional and burial environments [13], and these intragranular micropores largely increase the space for shale gas storage. Substantial intergranular pores and a few intragranular pores developed during the course of shallow burial of shales have very good connectivity, forming a very effective pore network; however, observation under microscope show that this type of pores only take a small portion in this area.

2.1.2.3. Intercrystal pores. Intercrystal pores are intercrystal micropores formed during the crystallization of minerals under stable environments and proper media conditions [12], mostly 5–200 nm in diameter. This type of pores are widespread in this area. Most pores among framboidal pyrite crystal grains are formed in anaerobic environments (Fig. 2-c). With neat edges, this type of pores are connected with each other to some extent.

2.1.2.4. Dissolved pores. Shales often contain unstable minerals like quartz, carbonate and feldspar, which will dissolve and form dissolved pores due to the effects of air, underground water or de-carbonic acid radical action during the course of pyrolysis of kerogen (Fig. 2-d). Sample observations under microscope show there are relatively fewer dissolved pores in this area and their diameter is 200–1200 nm.

2.2. Fractures

Natural microfractures in shales can largely improve the efficiency of hydraulic fracturing, increase the flow capacity of shales and provide migration channels for shale gas to enter the wellbore from matrix pores [14−17]. The factors affecting the formation of fractures are complex, which can be divided into internal factors and external factors from the perspective of geology. The external factors mainly include regional tectonic stress, tectonic position, sedimentation diagenesis and anomalously high formation pressure generated during the hydrocarbon generation; while the internal factors mainly include rocks, lithofacies and rock mineral composition features. Core observations and argon ion polishing SEM observations show there mainly developed the following types of fracture systems in this area.

2.2.1. Tectoclases inside mineral grains

Tectoclases inside mineral grains refer to the fractures formed by or associated with local tectonic movements. They can be divided into tension cracks and shear cracks based on mechanical properties. Under SEM, tension micro-cracks have different intersection angles with the bed plane and most are nearly perpendicular to the bed plane, which often cut through bedding fractures with larger attitude variation.

2.2.2. Organic evolution abnormal-pressure fractures

Organic evolution abnormal-pressure fractures refer to the fractures formed due to rock failure under local abnormal pressure generated during the organic evolution. They universally developed in carbonaceous shales with higher *TOC*. Irregular in fracture plane, not developed in groups and mostly filled by organic matters, this kind of fractures often occur at the edge of organic matters and other minerals (Fig. 2-e).

2.2.3. Diagenetic shrinkage joints

Diagenetic shrinkage joints refer to the fractures formed due to the shrinkage and volume reduction of rocks in diagenetic process. The causes include drying shrinkage effects, dehydrolysis, mineral facies change effects or thermal contraction, which have nothing to do with tectonic movements [18,19]. Commonly seen under SEM, the connectivities of this type of fractures are good and of great variation in opening. Diagenetic shrinkage joints occur widely in the quality interval with high silica content at the bottom of the Longmaxi shales because chemical changes in diagenetic process led to rock contraction.

2.2.4. Interlayer lamellation fractures

Interlayer lamellation fractures are primarily pores and fractures between horizontal bedding lamina planes with parting lineation, which are formed by deposition and composed of a series of thin shales. The inter-shale lamellation is the interface with weak mechanical properties and can be stripped very easily. Interlayer lamellation fractures are the most fundamental crack type in shales [20]. Very common in this area, this type of fractures usually have sandy interstratal lamellation planes and smaller opening degree.

2.2.5. Interlayer sliding fractures

Interlayer sliding fractures are fractures parallel to bed plane with apparent slip traces. The bed plane is generally smooth or characterized by scratches and steps (Fig. 2-f). Stratal configuration is the most fundamental rock texture of shales, and the bed plane is the weakest mechanical structural plane; whether in extensional basins or compressional basins, the interlayer sliding fracture is one of the most fundamental crack patterns in shales [20]. Affected by tectonic movements, this kind of fracture is very common in this area, but mainly developed at the bottom of Wufeng Fm.

3. Characterization of shale pore shape by nitrogen adsorption and desorption experiments

Shales are mainly composed of clay minerals and organic matter grains, which form pores of different types, sizes and shapes, but pore properties affect the specific surface area of shales and the relative abundance of micropores, which are directly related to the adsorptivity and mobility of shale gas in shales. For the purpose of analyzing the pore shape features, the International Union of Pure and Applied Chemistry (IUPAC) propose four types [21]. The pore shapes of the Longmaxi Shale in the Jiaoshiba area are complex. The adsorption and desorption curves (Fig. 3-a,b) of the two samples taken from Well JY1 have the following features: on the whole, the adsorption curves of the two samples are very steep near the saturated vapor pressure, whereas the desorption curves are very steep at median pressure, indicating that the pores of Longmaxi Shale gas reservoirs are mainly composed of nano-pores, irregular (amorphous) in shape, while the pore structures inside grains are featured by narrow fissure-like pores and comprised of other pores with various shapes. The pores are open, dominated by cylindrical pores with two ends open and parallel sheet-like pores with four sides open.

4. Discussions

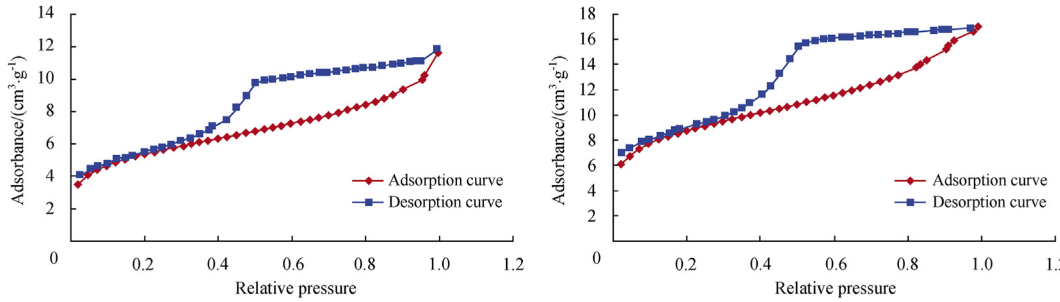
4.1. Main sizes of nano-pores in shale reservoirs

Nitrogen adsorption method is to use the adsorption principle of nitrogen on solid surface to quantitatively characterize the micropores and mesopores, while high pressure mercury injection technique can be used to accurately evaluate the mesopores and micropores [22,23], they can better describe the diameter range and features of pores in reservoirs together.

The test results of the nine samples taken from Well JY1 (Fig. 4) show that the pores with 2−30 nm in diameter in shales account for about 70% of the total; according to the classification criterion of IUPAC [21], this means the pores are mainly mesopores, followed by micropores and macropores.

4.2. Pore composition

The shale matrix pores can be classified into brittle mineral micropores, organic pores and inter-clay-mineral pores according to organic matters and mineral types, which is the important basis for quantitative characterization of the reservoir space of marine shales [24,25]. Wang Daofu et al. proposed the petrophysical model for porosity calculation and the



a. Adsorption and desorption curves of the Longmaxi Shale in Well JY1 (2 394.9 m in depth)

b. Adsorption and desorption curves of the Longmaxi Shale in Well JY1 (2 405.8 m in depth)

Fig. 3. Adsorption and desorption curves of the Longmaxi Shale.

mathematical model for matrix porosity calculation [24]. The porosity calculation formula of the model is as follows:

$$\varphi = \rho A_{\mathrm{Bri}} V_{\mathrm{Bri}} + \rho A_{\mathrm{Clay}} V_{\mathrm{Clay}} + \rho A_{\mathrm{Toc}} V_{\mathrm{Toc}}$$

where, φ is shale porosity; ρ is shale rock density, t/m^3; A_{Bri}, A_{Clay} and A_{Toc} are mass percentage of brittle minerals, clay and organic matters respectively; and V_{Bri}, V_{Clay} and V_{Toc} are micropore volume in unit mass of brittle minerals, clay and organic matters respectively, m^3/t, i.e., pore volume in the three types of minerals, reflecting the pore density.

This model is used to calculate the unit mass pore volume in brittle minerals, clays and organic matters of the Longmaxi Shale in Well JY1, the results are 0.0017 m^3/t, 0.0270 m^3/t and 0.2688 m^3/t respectively. Then, the results above are used to further compute the proportion occupied by the three types of pores in different *TOC* intervals, and it is found that *TOC* is clear in positive correlation with the organic pore content, e.g., the organic pore content is up to 50% in the quality shale interval at bottom (*TOC* > 2%), which has vital effects on the porosity of the interval Table 2.

4.3. Controlling factors on pore configuration of shale gas reservoirs

4.3.1. TOC

TOC of shales is not only an important parameter to evaluate the hydrocarbon generation potential of source rocks, but also an important factor controlling the development of organic pores [26,27]. Substantial nano-pores developed in organic matters provide the principal specific surface area and pore volume. Based on the nitrogen adsorption and desorption test results of the samples taken from Well JY1, the pore volume ranges between 0.008 cm^3/g and 0.024 cm^3/g, 0.013 cm^3/g on average; the specific surface area ranges between 8.4 m^2/g and 33.3 m^2/g, 18.9 m^2/g on average. Correlation analyses on *TOC*, specific surface area and pore volume (correlation coefficient is 0.6941 and 0.7204 respectively) (Fig. 5-a,b) shows clearly they have positive correlation, indicating that *TOC* is one of the main factors controlling the microscopic pores of the Longmaxi Shale. Complicated in internal structures, organic pores have rough surfaces and diverse inside shapes (Fig. 2-a), which largely increases their specific surface area and pore volume, thus enhancing the adsorption and accumulation capacity of shales. That is very important for the enrichment of shale gas.

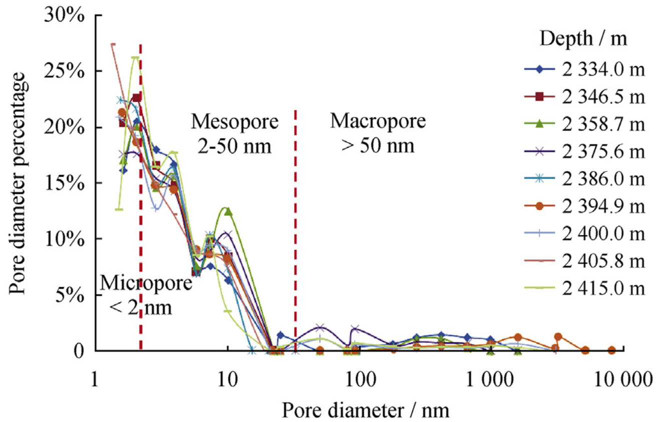


Fig. 4. Pore diameter distribution characteristic of the Longmaxi Shale in Well JY1.

4.3.2. Clay mineral content

Ross and Bustin [28] think that the clay minerals in shale gas reservoirs have higher micropore volume and larger specific surface area. We conducted correlation analyses on the clay minerals, pore volume and specific surface area of the Longmaxi Shale in the Jiaoshiba area (Fig. 6-a,b) and found that they exhibit some negative correlation; therefore, the clay minerals contribute relatively little to the pore volume and pore surface, they possibly provide pore volume and specific surface area of other pore scale.

4.3.3. Thermal evolution degree

Cheng Peng et al. [29] conducted thermal simulation experimental analyses on the relationship of nano-pore configuration and maturity (Fig. 7), the results show that when the thermal evolution degree ranges in between 0.7% and 3.5%, the pore configuration has an apparent positive correlation with the thermal evolution degree.

Zou Cai'neng et al. conducted high power SEM observations of the shale samples (after argon ion polishing) taken from Qiongzhusi Formation (with R_o 3.2%–3.6%) and Longmaxi Fm. (with R_o 2.3%–2.8%) in the Weiyuan area and found that the micropore volume generated by organic matters in Qiongzhusi Fm. shales is much less than that in the Longmaxi Shale [24,30], which also indicates that too high

Table 2
Statistics on matrix pore composition of the Longmaxi Shale in Well JY1 of the Jiaoshiba area.

Well interval/m	Content			Porosity	Proportion of various pores			Remarks
	Brittle minerals	Clay minerals	*TOC*		Brittle minerals	Clay minerals	Organic matters	
2326–2353	57.2%	40.9%	1.96%	4.50%	5%	71%	24%	20 samples
2353–2378	54.8%	42.6%	2.66%	5.04%	6%	64%	30%	26 samples
2378–2415	66.2%	29.5%	4.30%	5.17%	6%	36%	58%	40 samples

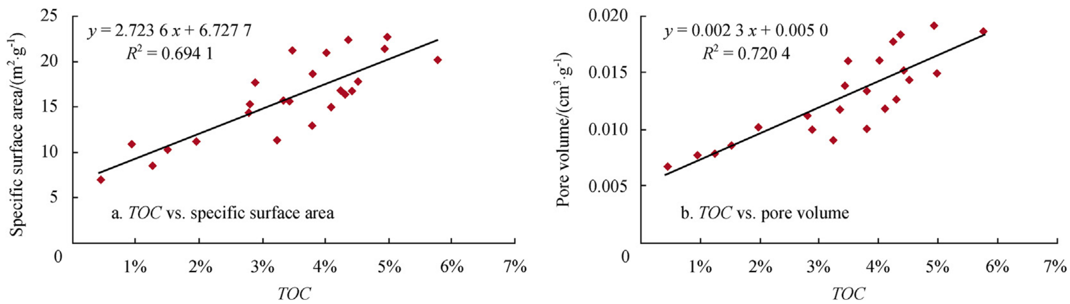


Fig. 5. *TOC* vs. specific surface area and pore volume of the Longmaxi Shale in Well JY1.

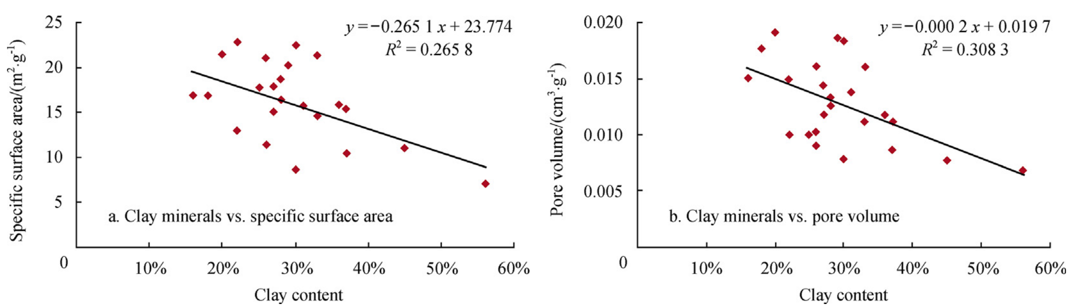


Fig. 6. Clay minerals vs. specific surface area and pore volume of the Longmaxi Shale in Well JY1.

thermal evolution degree can result in the reduction of organic pore development level. The thermal evolution degree R_o of the Longmaxi Shale in the Jiaoshiba area ranges between 2.42% and 3.13%, 2.65% on average, indicating the shales are just at the stage when the nano-pores are generated massively. The moderate evolution degree is an important factor affecting the development of organic pores.

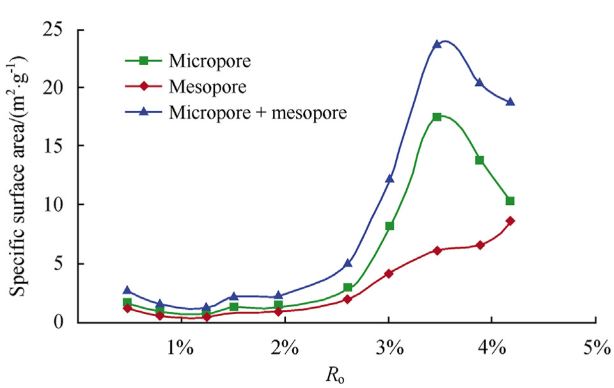


Fig. 7. Variation of nano-pore configuration with maturity of organic-rich shales.

5. Conclusions

1) The reservoir space of the Longmaxi Shale gas reservoirs in the Jiaoshiba area are diverse, including organic pores, inorganic pores (intergranular pores, intragranular pores, intercrystal pores and dissolved pores) and microfractures (tectoclases inside mineral grains, interlayer sliding fractures, diagenetic shrinkage joints and organic evolution abnormal pressure fractures); among which, organic pores and clay mineral pores are the most extensively developed pores in the shales of the area and are of great importance to the occurrence of shale gas, whereas microfractures serve as both reservoir space and main percolation channels for shale gas.
2) The pores of the Longmaxi Shale are in open state in this area, dominated by cylindrical pores with two ends open and parallel sheet-like pores with four sides open; the pores are mainly 2–30 nm in diameter, representing mesopores.
3) The pores are dominated by organic pores and clay mineral pores; *TOC* is in apparent positive correlation with organic pore content; the pores in quality shale interval at bottom (*TOC* > 2%) are dominated by organic pores, with a content of up to 50%.

4) *TOC* and thermal evolution degree (R_o) are the major factors controlling the microscopic pore structures of shale reservoirs; but the clay mineral content has little correlation with pore volume and specific surface area, so it possibly provides pore volume and specific surface area of other pore scale.

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