Research Article

Slope Belts of Paleouplifts Control the Pore Structure of Organic Matter of Marine Shale: A Comparative Study of Lower Cambrian Rocks in the Sichuan Basin

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Extensive exploration of the marine shale of the Niutitang Formation in south China has been conducted. However, exploration and development results have varied considerably in different areas. For example, the Niutitang shale in Chongqing City (south China) has only a short stable production of gas, low gas volumes, or even no gas production at all in some wells, and hence no successful development [3, 4]. However, successful exploration and development of the Niutitang shale has been made in Jingyan City in Sichuan Province (south China), where commercial gas has been successfully produced from the JinYe1 well [1]. The gas produced from shale wells can reach 2.3 m³/t, with the daily gas output of 6.0 × 10⁶ m³/d [5]. Successful exploration and development of the Niutitang shale has been made in Yichang City of Hubei Province (south China) by the China Geological Survey, where commercial gas has been successfully produced from the wells.

1. Introduction

The Lower Cambrian Niutitang Formation contains highly to overly mature organic-rich black marine shales with high TOC content and type I kerogen that is widely distributed in south China [1, 2]. However, their exploration and development potential vary significantly between different areas. For example, the Niutitang shale in Chongqing City (south China) has only a short stable production of gas, low gas volumes, or even no gas production at all in some wells, and hence no successful development [3, 4]. However, successful exploration and development of the Niutitang shale has been made in Jingyan City in Sichuan Province (south China), where commercial gas has been successfully produced from the JinYe1 well [1]. The gas produced from shale wells can reach 2.3 m³/t, with the daily gas output of 6.0 × 10⁶ m³/d [5].
EYY1 and ZD2 [6, 7]. In the EYY1 well, for example, the thickness of the Niutitang shale with gas total hydrocarbon exceeding 1.0% is 77 m [7, 8]. The gas from shale wells reaches 2.2 m$^3$/t with a daily gas output of 7.8 × 10$^4$ m$^3$/d [5]. Furthermore, the Niutitang shale in Jingyan also showed commercial gas with a daily gas output of 6.0 × 10$^4$ m$^3$/d [4].

Successful exploration and development of the Niutitang shale has also been made in Southern Guizhou (south China), where commercial gas has been successfully produced from the HY1 well [9, 10]. The HY1 well has a gas content of 1.3 m$^3$/t [11]. Brittle minerals, such as silica, can relieve the pressure from the overlying rock on OM pores and can provide support for the preservation of OM pores [14, 15]. Kerogen, bitumen, and pyrobitumen have distinct physical and chemical properties and can cause different styles of OM pore development [19]. Too high or too low thermal maturity is also not conducive for the development of OM pores [16].

Many studies have suggested that a number of marine shales in southern China generally have high TOC contents on average (>2.0%), high siliceous mineral content, and the same kerogen type (type I), but the thermal maturity of different formations of marine shales varies considerably [18–21]. For example, several researchers have compared shales of the Lower Silurian Longmaxi Formation (2.0% < $R_o$ < 3.0%) with those of the Lower Cambrian Niutitang Formation in Chongqing, the former of which have produced commercial gas [22, 23]. They concluded that high thermal maturity is the key reason resulting in the low gas content and low production in the Niutitang shale in Chongqing [1, 13, 14, 24–26].

Burial history and hence thermal maturity have a strong control over OM porosity development and petroleum generation [27, 28]. The OM pore evolution and petroleum generation are also closely related to the tectonic evolution [29, 30].

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Figure 1: Position of the study area and Niutitang shale well distribution in Chongqing, Sichuan Basin, Hubei Province, and Guizhou Province.
This study used samples of the Niutitang shale from wells in the Chongqing and Jingyan areas (southwestern Sichuan Basin) as research objects (Figures 1 and 2). Combined with the regional structural characteristics, the shales of Niutitang Formation in the Huangping area in southern Guizhou and Yichang area in western Hubei are compared. The OM pore characteristics of Niutitang shale in different exploration and development blocks in southern China were clarified, and...
finally, the special structural characteristics were proposed as exploration targets for the efficient exploration and development of ancient marine shale in south of China.

2. Geological Setting

2.1. Chongqing Area. The Chongqing area is located in the southeastern margin of the Sichuan Basin, south China, and structurally, it is located in the southeastern part of the Yangtze Platform (Figure 3). This area has experienced three main tectonic stages since the Late Mesoproterozoic: continental margin oceanic crust changing to continental crust during the Late Proterozoic to Silurian, continental expansion during the Devonian to Middle-Late Triassic, and Pacific plate subduction and Indian plate collision during the Mesozoic to Cenozoic [31, 32].

During the Early and Late Paleozoic, the Yangtze Platform experienced many large-scale transgression events, forming four sets of black shales during the Cambrian, the Upper Ordovician to the Lower Silurian, the Lower Permian, and the Upper Permian (Figure 2). The Niutitang shale in Chongqing was deposited during the Early Cambrian, when extensional activities resulted in the disintegration of southern Pangea and the expansion of the oceanic crust, leading to large-scale transgression [34]. The exploration target in the Chongqing area is the base section of the Niutitang Formation, which is mainly thick, black carbonaceous shale with high TOC content and siliceous minerals [35]. There is also some silty shale and a small amount of siliceous shale. These
sediments were deposited on a deep-water shelf under mainly reducing conditions [28–30].

2.2. Jingyan Area. The Jingyan area is located at the southwestern margin of the Sichuan Basin, between the large Weiyuan and Tieshan anticlines (Figure 4). In the Early Cambrian, medium-deep-water shelf deposition developed in the Jingyan area, and the water column gradually deepened from NW to SE [28].

The Niutitang Formation deposited in the Jingyan area is characterized by unequal interbedding of gray-dark gray shale, silty shale, and gray-dark gray calcareous litharenite, gray siltstone, and argillaceous siltstone [3]. From bottom to top, the black shale mainly changes into an interlayer between black shale and dark gray and between gray argillaceous siltstone and gray siltstone, and the interlayer is increased [4]. In the middle and upper part, there is a small amount of lenticular gray or siliceous nodules and a small amount of pyrite. The mineralogy is mainly quartz and clay, with minor carbonate minerals [3, 4, 20, 21].

3. Focused Ion Beam Scanning Electron Microscopy (FIB-SEM)

FIB-SEM analysis for the observation of OM pores was conducted using a FEI HELIOS NanoLab 650 (FEI Corporation, United States). Before that, samples were ground, argon ion polished, and carbon sprayed to increase electrical conductivity (Table 1). The secondary electron emission technology of FIB-SEM was also used to further identify OM pore characteristics. This technology can create bright lights around the pores, highlighting the surface.
4. Results

There are no OM pores in the pyrobitumen of the Chongqing samples, as indicated by SEM imaging (Figure 5). The secondary electronic imaging was used to highlight the morphology of any smaller-scale inner structure in these shale samples; however, none was observed in the pyrobitumen of the Chongqing samples (Figure 6). This further proves that there are no OM pores in the Chongqing samples. However, under the FIB-SEM, the pyrobitumen in the Niutitang shale in Jingyan shows a large number of OM pores (Figures 7 and 8), which are characterized by round and oval shapes, uniform distribution, and a large pore size.

5. Discussion

5.1. Comparison with Jingyan and Chongqing Areas. A large number of OM pores can not only increase the adsorption capacity for gas but also be conducive to the effective flow of gas in a shale reservoir by enhancing the connectivity [2, 17, 26]. This may be one reason why the Niutitang shale produces commercial gas in Jingyan but not in Chongqing (Figures 5–9).

Rock parameters of the Niutitang shale from Jingyan were compared with those from Chongqing. As shown in Table 2, these shale samples share very similar reservoir characteristics such as average TOC content, mineral composition, and kerogen type, but they have very different Eq-R_o. The Niutitang shale in Jingyan with abundant OM pores has an Eq-R_o of less than 3.0%, while the Niutitang shale in Chongqing with almost no OM pores has an Eq-R_o larger than 3.0%. It reveals that the formation and preservation of OM pores are likely controlled by thermal maturity [1, 26, 27].
The thermal evolution and the associated OM pore generation and hydrocarbon potential in shale are closely related to the tectonic evolution of the study areas [37, 38]. For the Chongqing study area, the maximum paleoburial depth of the Niutitang shale is 8000 m, corresponding to an Eq-\( R_o \) exceeding 3.5% (Figure 10). A large number of OM pores in the pyrobitumen were destroyed due to excessive thermal evolution, resulting in the loss of hydrocarbon gases in shale.

**Figure 6:** No OM pore in the pyrobitumen of Chongqing samples (CQ2 well, 1149 m). The secondary electron technique can highlight the pores in a circular layer, making them easy to identify. If no highlight is found in the pyrobitumen, there is no OM pore. The secondary electron technique proves that there is no OM pore in the pyrobitumen of the Chongqing sample.
In contrast, for the Jingyan study area, the maximum paleoburial depth of the Niutitang shale is 6200 m [4], corresponding to an Eq-$R_o$ of 2.2% to 2.8% (Figure 11).

The maximum paleoburial depth of the Niutitang shale seems to be closely related to the paleostructure of the Jingyan area, which is structurally located on the wing of the Chuanzhong anticline (Figure 4). Due to the presence of the Chuanzhong paleouplift, the maximum ancient burial depth of the Niutitang shale in Jingyan is much shallower than its equivalents in south China. In contrast, the maximum paleoburial depth of the Niutitang shale in Chongqing reaches 8000 m due to a lack of such a paleouplift, and the Eq-$R_o$ of the Niutitang shale thus reaches a stage of overmaturity due to metamorphism [26, 39].

5.2. Comparison of the Niutitang Formation in Other Areas

5.2.1. Niutitang Shale in Yichang, Western Hubei. The Yichang area is located in the center of the Middle Yangtze Platform, and it is mainly comprised of the Huangling
Figure 9: Gas adsorption models in different OM pore structures. (a) Based on the large number of OM pores, abundant gas was adsorbed in Jingyan shale samples. (b) No gas was adsorbed in Chongqing shale samples because there were no OM pores.

Table 2: Geological and reservoir development parameters with different gas contents and production of the Niutitang shale in Chongqing, Jingyan, Yichang, and Southern Guizhou. Parts of this table are revised from Refs. [2, 5, 7].

| Formation | Area   | Well | TOC (%) | Eq-\(R_o\) (%) | Kerogen type | Porosity (%) | Pressure | Gas content (m³/t) | Gas test production (10⁴ m³/d) |
|-----------|--------|------|---------|----------------|--------------|--------------|----------|--------------------|-------------------------------|
| Niutitang | Chongqing | CQ2 | 2.82 | 3.74 | I | 0.16 | 0.7 | 0.18 | 0 |
|           | Jingyan | JinYe1 | 2.36 | 2.79 | I | 3.94 | 1.38 | 2.02 | 10.5 (HF) |
|           | Yichang | EYY1 | 2.34 | 2.79 | I | 3.68 | 0.78 | 1.98 | 7.83 |
|           | Huangping | HY1 | 5.81 | 2.51 | I | 0.6 | / | 1.3 | / |

Figure 10: Burial history of the Niutitang shale in Yichang is based on the EYY1 well (revised from Ref. [6]). Due to the existence of the Huangling paleouplift, the ancient burial depth of the Niutitang shale distributed in its periphery is only 6500 m.
paleouplift, the Yichang Slope Belt, the Zigui Depression, and many other tectonic units (Figure 12). At present, shale gas exploration has focused mainly on the Sinian Doushantuo Formation and the Niutitang Formation by the China Geological Survey, in the southeastern margin of the Huangling paleouplift [33, 39]. The Huangling paleouplift was formed during the Late Jurassic to Early Cretaceous with a monoclinal structure trending SE [40].

The Early Cambrian facies in the study area was developed as a result of the structuration in the Late Sinian period. Tectonic movement at the end of the Sinian led to the area as a whole forming a low section in the southwest and a high section in the northeast [5]. As a result, the Niutitang Formation thickens from the northeast to the southwest. The lithofacies of the Niutitang Formation in Yichang is gray-black and black carbonaceous shale [2]. The sediments were deposited in shallow-deep and deep shelves with a thickness of 150-240 m [6, 7]. The Niutitang Formation can be roughly divided into three sections: (i) the lower section is black shale with a thin layer of silty limestone, which is a nontypical carbonate deposit at the early stage of transgression; (ii) the middle section is gray-black and black carbonaceous shale,
deposited on the outer shelf as a result of extensive transgression; and (iii) the upper section is micritic limestone which is deposited on the open platform at the high stage of transgression [2, 5, 6]. The shale with $\text{TOC} \geq 2\%$ is mainly developed in the middle section of the Niutitang Formation, but with thickness variations from 9 m to 140 m [23].

TOC content of the Niutitang shale in Yichang mainly ranges between 1.2% and 4.4%, with an average value of 2.3% [2, 6, 7, 40]. The $\text{Eq-R}_o$ of the Niutitang shale in Yichang is generally below 3.0%, with an average value of 2.8% [2, 6, 7]. The Niutitang shale in Yichang is dominated by quartz, clay, and carbonate. The average quartz content is 39.3% for samples from the EYY1 well, with a range of 30.2% to 50.2%. The average clay mineral content is 33.7% and has a range of 2.7% to 61.3% from samples of the YD2 well. The average carbonate mineral content is 26.2% and has a range of 8% to 39% from samples of the ZD1 well [2, 5–7].

There are a lot of OM pores inside the pyrobitumen of the Yichang shale samples. These OM pores are mostly oval and round, with a regular morphology [2, 12, 15]. For Yichang, the maximum paleoburial depth of the Niutitang shale is 6500 m, corresponding to an $\text{Eq-R}_o$ of 2.3% to 2.8% (Figures 13 and 10).

The maximum paleoburial depth of the Niutitang shale seems to be closely related to the paleostructure of the study area, as in the case of Yichang, which is structurally located on the wing of the Huangling paleouplift [6, 40]. Due to the presence of the Huangling paleouplift, the maximum burial depth of the Niutitang shale in the Yichang area is much shallower than its peers. According to Figure 14 which is closer to the Huangling paleouplift, the $\text{Eq-R}_o$ of the Niutitang shale is lower (Figure 15). The $\text{Eq-R}_o$ average of the Niutitang shale in the ZD1 well is correspondingly 1.61% [2, 5, 7, 40].

5.2.2. Niutitang Shale in Southern Guizhou. The Southern Guizhou area is located in the south of China, bordering the Central Guizhou Uplift in the west and adjacent to the Xuefeng Mountain Uplift in the east, the Wuling Depression in the north, and the Qiannan Depression in the south.

Figure 12: Plane and section structure of the Huangling Uplift in Hubei (revised from Refs. [5, 39]).
In the early Paleozoic and late Paleozoic, Southern Guizhou experienced many large-scale transgression events and formed two sets of black shales representing the Cambrian, the Upper Ordovician, and the Lower Silurian [41, 42]. The bottom of the Niutitang Formation is gray siliceous shale, the middle and lower part is gray carbonaceous shale, the upper part is dark gray argillaceous dolomite and mudstone, and the top is dark gray limestone [43, 44].

TOC content of the Niutitang shale in Southern Guizhou mainly ranges between 2.7% and 4.0%, with an average value of 3.41% [45]. The Eq–R_o of the Niutitang shale in Southern Guizhou is generally below 3.0%, with an average value of 2.4% [46, 47]. Minerals of the Niutitang shale in Southern Guizhou are also dominated by quartz, clay, and carbonate [10, 11].

There are a lot of OM pores inside the Southern Guizhou samples (Figure 17). These OM pores are mostly oval and round, with a regular morphology. For the Southern Guizhou, the maximum paleoburial depth of the Niutitang shale is 5600 m, corresponding to an Eq–R_o of 2.2% to 2.7% (Figure 14), similar to the Yichang shale samples. The maximum paleoburial depth of the Niutitang shale seems to be closely related to the paleostructure of the Southern Guizhou area, which is structurally located on the east wing of the Qianzhong paleouplift [42, 45]. Due to the presence of the Qianzhong paleouplift, the maximum ancient burial depth of the Niutitang shale in Southern Guizhou is much shallower than its peers.

5.3. Summary. The Niutitang shale in Yichang and Guizhou has a similar thermal maturity compared with that in Jinyan. A large number of OM pores are developed in the Niutitang shale samples in Yichang and Guizhou, which is similar to the pore structure characteristics of the Niutitang
Figure 14: Burial history of the Niutitang shale in southern Guizhou was based on the HY1 well (revised from Ref. [9]). Due to the existence of Qianzhong paleouplift, the ancient burial depth of the Niutitang shale distributed in its periphery was only 5600 m.

Figure 15: The Eq-$R_0$ distribution of the Niutitang shale around the Huangling paleouplift. Due to the existence of the Huangling paleouplift, the ancient burial of the Niutitang shale is lower than those of other areas in south China. The closer to the uplift, the lower the thermal maturity of the shale.
shale in the Jingyan area. However, from the perspective of the Niutitang shale in Chongqing, its thermal maturity is much higher than that in the Jingyan, Yichang, and Guizhou regions, while there are no OM pores of the Niutitang shale in Chongqing. However, other parameters such as TOC content, mineral composition, and kerogen type of the Niutitang shale reservoirs in the four regions (Jingyan, Chongqing, Yichang, and Guizhou) have similar characteristics [2, 6, 7, 40, 42, 48–51]. Therefore, thermal maturity is the key factor to control the OM pore development.

From the perspective of tectonic evolution, the maximum burial depth of a formation controls the thermal maturity of a shale reservoir, while the burial depth is controlled by the regional tectonic pattern [1, 4, 14, 32]. According to the regional structure pattern of Jingyan, Yichang, and Guizhou, the underlying structures of the Niutitang shale are all paleouplift structures, and the target shale is distributed in the slope belt of the paleouplift [2, 4, 26, 33]. Due to the existence of the paleouplift, the maximum ancient burial depth of the Niutitang shale in Jingyan, Yichang, and Guizhou is about 6000 meters [2, 4, 5, 10], and the burial depth of the shale is moderate, avoiding excessive evolution and leaving a large number of OM pores in the shale reservoir [1, 16]. In contrast, in the Chongqing area, due to the absence of the paleouplift structure, the maximum ancient burial depth of the Niutitang shale in this area reached 8000 meters [26], and the shale reservoir experienced excessive thermal evolution, resulting in the disappearance of a large number of OM pores, and a large volume of gas was lost due to the absence of effective storage spaces in the reservoir [49, 52, 53].

According to the successful exploration experiences of the Niutitang shale in Jingyan, Yichang, and Southern Guizhou and the failure experience of the same formation in areas like Chongqing, the exploration of the ancient marine shale reservoirs should focus on areas with moderate thermal maturity. The marine shale sedimentary in the paleouplift region has appropriate thermal maturity, so that a large number of OM pores are retained in the shale reservoir, which provides an effective space for gas occurrence, as well as a large amount of gas enrichment. Therefore, the exploration and development of marine shale with old sedimentary age (especially the lower Cambrian and older formations) should focus on the slope belt of a paleouplift (Figure 18).

Figure 16: Plane and section structure of Qianzhong Uplift in Guizhou (revised from [41]).
Figure 17: A large number of OM pores in pyrobitumen of southern Guizhou samples. (a–e) OM pores in the pyrobitumen in the mineral matrix. (f) The pyrobitumen with OM pores in pyrite particles. This figure was revised from Refs. [10, 11, 44, 46].

Figure 18: The target area for the exploration of ancient marine shale gas reservoirs. The exploration and development of ancient marine shales should focus on areas around paleouplifts.
6. Conclusions

Scanning electron microscope (SEM) and geotectonic characteristics were used to illustrate organic matter (OM) pore structure and evolution in the Lower Cambrian Niutitang shale samples with different thermal maturities in northeastern Chongqing and Jingyan of the southwestern Sichuan Basin in south China. Conclusions of this study are as follows:

(1) The Niutitang shales in both Jingyan and Chongqing cities (south China) have similar TOC content (ranging from 1.5% to 3.0%), mineral composition (dominated by silica and clays), and kerogen type (dominated by type I) but different thermal maturity. The Niutitang shale in Jingyan has a lower thermal maturity (2.0%-3.0%) than that (3.0%-4.0%) in Chongqing.

(2) OM pore development characteristics of the Niutitang shale in Jingyan and Chongqing are quite different. The Niutitang shale in Jingyan developed a lot of large-sized OM pores. In contrast, almost no OM pores were found in the Niutitang shale in Chongqing. The OM pores in the Niutitang shale in Jingyan act as an effective storage of gas, contributing to gas enrichment.

(3) OM pore development is mainly controlled by thermal maturity. A lot of OM pores can develop in shale reservoirs when the thermal maturity (Eq-Ro) of shale is lower than 3.0%. A significant amount of OM pores will disappear when the thermal maturity exceeds 3.0%.

(4) Ancient marine shales have a relatively shallow maximum burial depth when they were deposited on the slope belt of paleouplifts, which can ensure that shale reservoirs are likely not overly mature.

Data Availability

All data, models, and code generated or used during the study appear in the submitted article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Authors’ Contributions

Pengfei Wang and Chen Zhang contributed equally to this work.

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