

[Quantitative Characterization for Pore](https://www.frontiersin.org/articles/10.3389/feart.2022.864189/full) [Connectivity, Pore Wettability, and](https://www.frontiersin.org/articles/10.3389/feart.2022.864189/full) [Shale Oil Mobility of Terrestrial Shale](https://www.frontiersin.org/articles/10.3389/feart.2022.864189/full) [With Different Lithofacies](https://www.frontiersin.org/articles/10.3389/feart.2022.864189/full)—A Case [Study of the Jurassic Lianggaoshan](https://www.frontiersin.org/articles/10.3389/feart.2022.864189/full) [Formation in the Southeast Sichuan](https://www.frontiersin.org/articles/10.3389/feart.2022.864189/full) [Basin of the Upper Yangtze Region in](https://www.frontiersin.org/articles/10.3389/feart.2022.864189/full) [Southern China](https://www.frontiersin.org/articles/10.3389/feart.2022.864189/full)

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Some major hydrocarbon-bearing basins are rich in shale with terrestrial facies in China, which may provide abundant terrestrial shale oil and gas resources. This work studied the Jurassic Lianggaoshan Formation in the Southeast Sichuan Basin of the upper Yangtze Region. Core samples were chosen for the total organic carbon content and mineral composition analyses to classify shale lithofacies. Afterward, pore connectivity, pore wettability, and shale oil mobility with different lithofacies were characterized by spontaneous imbibition, nuclear resonance, and centrifugation. Conclusions are as follows: the pore connectivity of organic-rich clay shale was mostly between moderate to good with oil-prone wettability and high mobile oil saturation. The organic-rich mixed shale has moderate to good pore connectivity, water-prone wettability, and the highest mobile oil saturation. Organic matter–bearing clay shale has bad to moderate pore connectivity. Meanwhile, its pore wettability covers oil wetting, mixed wetting, oil-prone wetting, and water-prone wetting. Its mobile oil saturation was moderate. Regarding organic matter–bearing mixed shale, the pore connectivity was bad to moderate with mixed-wetting pore wettability and moderate mobile oil saturation.

Keywords: terrestrial shale, different lithofacies types, pore connectivity, pore wettability, shale oil mobility

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1 INTRODUCTION

Huge achievements have been underscored in geological theories about unconventional oil and gas, horizontal drilling, fracturing technologies, and decreasing single-well drilling costs. In North America, the huge success in shale oil and gas exploration has reshaped the world energy landscape [\(Clarkson et al., 2013](#page-9-0); [Bazilian](#page-9-1) [et al., 2014;](#page-9-1) [Zhang et al., 2014;](#page-11-0) [Geng et al., 2016;](#page-10-0) [Hackley and](#page-10-1) [Cardott, 2016](#page-10-1)). At the same time, China has achieved major progress in marine shale gas exploration in the Sichuan Basin and its surrounding areas, with shale gas fields built in Jiaoshiba, Weiyuan, Changning, Zhaotong, Luzhou, and other regions [\(Tan](#page-11-1) [et al., 2014](#page-11-1); [Liu et al., 2018](#page-10-2); [Long et al., 2018;](#page-10-3) [Xu et al., 2019](#page-11-2); [Yi et al.,](#page-11-3) [2019\)](#page-11-3). Some oil and gas-bearing basins in China, including Sichuan, Junggar, Bohai Bay, Songliao, and Ordos, are home to both marine and terrestrial strata, where terrestrial shale contains a large amount of potential shale oil and gas resources due to the moderate burial depth. At the same time, its thickness and total organic carbon (TOC) content are high with plentiful layers, moderate thermal evolution, and great Kerogen types. The convenience of exploration and high profits has attracted PetroChina and Sinopec to invest more in terrestrial light shale oil and to launch large-scale exploration activities ([Li et al., 2018](#page-10-4); [Yang et al., 2019](#page-11-4); [Zhou](#page-12-0) [et al., 2019](#page-12-0); [Zou et al., 2020;](#page-12-1) [Li and Chen, 2021\)](#page-10-5).

Shale pores serve as the primary reservoir space and seepage channel of light shale gas, and their connectivity as well as wettability are important research objects in studying a shale reservoir's characteristics because they affect the difficulty of oil and gas transportation in shale. Additionally, mobility is crucial to the evaluation of shale oil. The quantitative characterizations of connectivity, wettability, and shale oil mobility are essential. According to [Huang et al. \(2017\)](#page-10-6), shale reservoirs have poorer pore connectivity than conventional oil and gas reservoirs; spontaneous imbibition is closely related to shale mineral composition; and foliation direction can affect spontaneous imbibition. Normally, the curve of spontaneous imbibition along the layers has a higher slope, but samples show that the curve through the layers has the same slope, which is probably because the higher hydrophilic mineral content in shale reservoirs weakens the directional dependence of water transportation in shale reservoirs. By analyzing the difference in pore space through nuclear magnetic resonance and high-pressure mercury intrusion porosimetry, [Ning et al. \(2017b\)](#page-10-7) proposed a method for evaluating the pore connectivity of tight reservoirs based on experimental data and verified its correctness by spontaneous imbibition experiments. [Jiang et al. \(2020\)](#page-10-8) studied terrestrial shale experimental tools including scanning electron microscope, Soxhlet extraction, gas adsorption, nuclear magnetic resonance, and centrifugation. They pointed out that pore structure characteristics and mineral composition of shale reservoirs jointly control the mobility of shale oil. Mobile oil is mainly reserved in large pores (>50 nm) and bound oil in small pores (<50 nm). Large pores offer space for shale oil reservoirs and contribute to shale oil flow, while small pores go against the move of shale oil owing to their larger pore-specific surface area, stronger adsorption capacity, and worse connectivity. The mineral composition has a significant impact on shale oil

mobility. In addition, the bedding structure, such as the bedding fracture, is conducive to the development of reservoir space, which improves the shale pore connectivity and further promotes shale oil mobility.

This study explored pore structure characteristics of terrestrial shale with different lithofacies types, with the key exploration well TY1 being taken as the research object in the case analysis of terrestrial shale from the Jurassic Lianggaoshan Formation in the southeast Sichuan Basin of the upper Yangtze Region in southern China ([Figure 1](#page-2-0)). This article is the first to classify lithofacies of shale in accordance with the TOC content and mineral composition. Afterward, experiments were conducted on shale samples: spontaneous imbibition was adopted to clarify the pore connectivity and wettability of shale with different lithofacies types; nuclear resonance and centrifugation were used to study the mobility of shale oil with different lithofacies types.

2 GEOLOGICAL SETTINGS

2.1 Sedimentary and Stratum **Characteristics**

The study area Lianggaoshan Fm. was divided into Liang Members I, II, and III, among which Liang Members I and II could be further divided into two sub-members, the upper and the lower ([Figure 2](#page-3-0)). A set of gray siltstone, silty shale, black graygray black shale, and fine gray siltstone were found in the Lianggaoshan Formation. From the upper Liang Member I to the lower Liang Member II [\(Li et al., 2010](#page-10-9); [Li et al., 2017](#page-10-10); [Wang](#page-11-5) [et al., 2018](#page-11-5); [Qing et al., 2019](#page-11-6); [Li X. et al., 2021](#page-10-11)), these sections were mainly deposited by gray-black shale deposits due to their favorable conditions for developing organic-rich shales. During the sedimentary period, the Lianggaoshan Formation went through a complete lake transgression and regression cycle. In the early period of Liang Member I, the resources were relatively sufficient before the lake transgression. Maximal flooding took place in the early period of Liang Member II, where the lake deposits were shallow to moderate. Afterward, the lake regression brought a favorable zone for the growth of organic-rich shale. In Liang Member III, coarse-grained clastic materials from the Delta Front were mainly deposited. Dark mud shales mainly existed in the upper Liang Member I and lower Liang Member II sections [\(Pang et al., 2019](#page-11-7); [Liu et al., 2020](#page-10-12); [Li C. et al., 2021](#page-10-13)).

2.2 Tectonic Characteristics

Located within the fold belt of the high-steep fault in eastern Sichuan, the study area is bordered by the faults of Huaying Mountain in the west, which is adjacent to the uplifts of central Sichuan, and by the fracture zone of Qiyue Mountain in the east, which sits between the Sichuan and Hubei provinces. With a series of arc-shaped mountains, the structural belt of the study area has a trending of regional structural lineament turning from NNE into NEE northward. The mountains within the structural belt form a high-steep anticline with the Permian–Triassic system at its core. The slow and steep sides are not asymmetrical. In general, the former has a stratum dip of 20° ~30°, while that of the latter is 40°~70° or with an upside-down stratum. The wide valleys

between the mountains are a wide and slow syncline composed of the Jurassic system as a typical barrier in structure and topography [\(Liu et al., 2010;](#page-10-14) [Gao et al., 2017](#page-10-15)).

3 SAMPLES, EXPERIMENTS, AND DATA **SOURCE**

3.1 Total Organic Carbon Content and Mineral Component Analysis

A Sievers 860 TOC analyzer was used in the TOC content analysis for the same-depth samples, and a YST-I mineral analyzer was employed in the X-ray mineral-wide and clay mineral analysis. The results of the mentioned experiments were availed to classify the shale petrographic types.

3.2 Spontaneous Imbibition Experiment

Spontaneous imbibition refers to the process where a wetting fluid (water or oil) replaces a nonwetting fluid (air) under capillary force. Shale samples were selected in 15 depths of the TY1 well in the Lianggaoshan Formation. As shown in [Figure 3](#page-4-0), in each depth, two samples as a pair, respectively being horizontal and vertical to the bedding, were chosen to conduct the spontaneous imbibition experiment via water and oil (oil was replaced by decane). The numbering of experiment samples is as shown in [Table 1](#page-4-1), and experiment methods of spontaneous imbibition were as follows [\(Gao and Hu, 2015](#page-10-16); [Ning et al.,](#page-11-8) [2017a](#page-11-8); [Wei et al., 2019](#page-11-9); [Zuo et al., 2019\)](#page-12-2):

- 1) Shale samples of each depth were cut along the beddings into four 1 cm \times 1 cm \times 1 cm cubes, namely, A, B, C, and D. The top and the bottom of the cubes A and C were parallel, while those of B and D were vertical to the bedding. The other four sides of the samples were covered with epoxy resin ([Zhang](#page-11-10) [et al., 2019d;](#page-11-10) [Wang G. et al., 2020](#page-11-11); [Gao et al., 2020](#page-10-17)).
- 2) The spontaneous imbibition experiment was conducted for 24 h. A and B used oil samples (decane), while water samples (deionized water) were adopted by C and D, as shown in the schematic diagram in [Figure 3](#page-4-0). The absorption content of oil and water was monitored by using an electronic balance.

[et al., 2021;](#page-10-20) [Wei et al., 2021\)](#page-11-12).

When the reading stabilized, the experiment ended. [Figure 4](#page-5-0) shows the schematic diagram of the experiment devices ([Liu](#page-10-21) [and Zhang, 2019;](#page-10-21) [Zhang et al., 2020a;](#page-11-13) [Zhang et al., 2020b\)](#page-11-14).

3) After the experiment, all data were presented into a spontaneous imbibition curve, where the axis x referred to time, and the axis y meant the water absorption height. Then, the slope of the spontaneous imbibition curve was calculated.

3.3 Experiment Analysis of Shale Oil Mobility

This study chose shale samples of 10 depths from the TY1 well in Lianggaoshan Fm. and conducted experiments on shale oil mobility with the combined methods of nuclear magnetic resonance (NMR) and centrifugation. The detailed steps are as follows, with the numbering of samples shown in [Table 2](#page-5-1).

Column samples were achieved by drilling holes with a diameter of 25 mm and a length below 60 mm. Samples were weighed before reaching the T_2 spectral line of original samples by NMR imaging (the first measurement of the T_2 spectral line) ([Xie et al., 2019](#page-11-15); [Li et al., 2020;](#page-10-22) [Zhang et al., 2022](#page-11-16)).

Vacuum pumping devices were used to obtain saturated water (-0.1Mpa). After about 6 h, the saturated samples were taken out and wiped with a moisturized tissue. Next, processed samples were weighed before measuring the T_2 spectral line in the NMR device (the second measurement of the T_2 spectral line; the general oil and water signal).

After processing the samples in the saturated manganese (Mn) for 72 h (to eliminate the water signal in the samples), we took them out and wiped the sample surface with wet tissues before weighing the saturated samples. Afterward, the T_2 spectral line of Mn-saturated samples was measured via NMR devices (the third measurement of the T_2 spectral line; oil signal). Oil saturation could be calculated based on the second and third measurement results.

Samples were put into the centrifuge. After an 8-hour 200psi centrifugation, the T_2 spectral line was measured again (the fourth measurement of the T_2 spectral line).

TABLE 1 | The depth and member of samples in the spontaneous imbibition experiment

Number	Sample depth in the spontaneous imbibition experiment (m)	Formation
1	2,403.93	Liang Member III
2	2,513.07	Lower Liang Member II
З	2,517.83	Lower Liang Member II
4	2,527.04	Lower Liang Member II
5	2.540.45	Lower Liang Member II
6	2.542.24	Lower Liang Member II
7	2,547.35	Lower Liang Member II
8	2,552.98	Lower Liang Member II
9	2,557.73	Lower Liang Member II
10	2,573.32	Upper Liang Member I
11	2,576.98	Upper Liang Member I
12	2,579.45	Upper Liang Member I
13	2,589.7	Upper Liang Member I
14	2,607.23	Upper Liang Member I
15	2,618.06	Upper Liang Member I

Based on the centrifugation results, mobile oil saturation, bound oil saturation, and the T2 cutoff value were obtained.

A MesoMR12-025H core NMR analyzer was employed in the NMR logging. The multi-functional NMR analyzer has been widely used in geologic research and energy exploration. The magnets were permanent with a resonance frequency of 12.403 MHz. The temperature of the magnets was kept at 32.00 ± 0.02 ° C, and the diameter of the sonde body was 25 mm.

4 RESULTS AND DISCUSSION

4.1 Lithofacies Types

Previous studies summarized several classification plans based on the TOC content and mineral constituents ([Ji et al., 2016](#page-10-23); [Wang](#page-11-17) [et al., 2016;](#page-11-17) [Tang et al., 2017](#page-11-18); [Zhang et al., 2017;](#page-11-19) [Zhang et al., 2019a\)](#page-11-20). Based on the TOC content, shales can be divided into three groups, namely, organic-rich (TOC content ≥1.5%), organic-containing (TOC content ranges 1~1.5%), and organic-poor shale (TOC content ranges 0~1%) ([Zhang et al., 2018b](#page-11-21); [Zhang et al., 2020c;](#page-11-22) [Xiao et al., 2020;](#page-11-23) [Guo et al., 2021;](#page-10-24) [Shan et al., 2021](#page-11-24)). In terms of mineral contituents, shales can be classified into four types: calcareous (carbonate minerals ≥50%), grapholith (clay minerals ≥50%), siliceous (siliceous minerals ≥50%), and mixed shale (each mineral accounts for less than 50%) ([Li et al., 2019](#page-10-25); [Huang H. et al.,](#page-10-26) [2020](#page-10-26); [Xia et al., 2020](#page-11-25); [Chen et al., 2021\)](#page-9-2). Combining the two classification standards, we can achieve three times four, that is, twelve lithofacies types based on the TOC content and mineral constituents, as shown in [Tables 3](#page-5-2), [4](#page-5-3).

4.2 Pore Connectivity and Wettability of Shale With Different Lithofacies Types

The curve slope of spontaneous imbibition can be used to evaluate the pore connectivity of shale reservoir properties [\(Zhang et al.,](#page-11-26) [2018a](#page-11-26); [Huang J. et al., 2020](#page-10-27); [Liu et al., 2021a;](#page-10-28) [Liu et al., 2021b\)](#page-10-29). Previous studies have carried out simulations under the pore

TABLE 2 | The depth and members of samples in the shale oil mobility experiment.

TABLE 4 | Lithofacies types of shale samples in the shale oil mobility experiment.

TABLE 3 | Lithofacies types of shale in the spontaneous imbibition experiment.

network model and found that high pore connectivity (the average possibility of connectivity $p > 0.28$) corresponded to the spontaneous imbibition slope of 0.5. When $p = 0.2488$ (imbibition critical point), the spontaneous imbibition slope was 0.26. A lower slope means worse connectivity of porous media [\(Gao and Hu, 2013;](#page-10-30) [Gao and](#page-10-31) [Hu, 2016;](#page-10-31) [Kang et al., 2019](#page-10-32); [Gao, 2021](#page-10-33); [Yu et al., 2022](#page-11-27)). [Table 5](#page-6-0) shows the connectivity evaluation results. The spontaneous imbibition curve has two-stage features, where the slope in the early stage outnumbers that of the late period. Water absorption in the early stage resulted from the dry surface and rapid absorption of the bedding fracture, while that in the late stage was due to pore absorption characteristics. Imbibition curve slopes in the late stage were taken as the standard values.

Based on previous studies ([Gao et al., 2018;](#page-10-34) [Zhang N. et al., 2019;](#page-12-3) [Wang, 2019;](#page-11-28) [Wang J. et al., 2020](#page-11-29); [Hou et al., 2020](#page-10-35)), the rock wettability index was proposed and expressed in the following formula:

the rock wettability index: $W = (P_{water} - T_{water}) - (P_{oil} T_{oil}$),where P_{water} refers to the spontaneous imbibition slope in

TABLE 5 | Evaluation of reservoir connectivity.

TABLE 7 | Connectivity and wettability evaluation of organic-rich grapholith shales and organic-rich mixed shales.

a parallel direction with water as the fluid, T_{water} refers to the spontaneous imbibition slope in a vertical direction with water as the fluid, P_{oil} refers to the spontaneous imbibition slope in a parallel direction with oil as the fluid, and T_{oil} refers to the spontaneous imbibition slope in a vertical direction with oil as the fluid. [Table 6](#page-6-1) shows the rock wettability based on the index ([Zhang et al., 2019b;](#page-11-30) [Zhang et al., 2019c](#page-11-31)).

The connectivity and the wettability index of samples 1–15 are presented in [Tables 7](#page-6-2), [8](#page-7-0). For organic-rich mixed, organic-rich grapholith, organic-containing grapholith, and mixed shale, the pore connectivity was mostly moderate to good. But most organic-poor shales had bad to moderate pore connectivity.

Organic-rich grapholith shales are mostly oil-prone wetting, and a few are water wetting and mixed wetting, while organic-rich mixed shales are water-prone wetting or water wetting. In terms of organic-containing grapholith shales, their wettability can be more complex, including oil, mixed, oil-prone, and water-prone wettability. The wettability of organic-containing mixed shales is mixed. Organic-poor grapholith shales are water wetting, while organic-poor mixed ones have water-prone or oil-prone wettability.

4.3 Evaluation of the Mobile Shale Oil With Different Lithofacies

4.3.1 Results of the Shale Oil Mobility Experiment

[Tables 9](#page-7-1), [10](#page-7-2) show the findings from the shale oil mobility experiment. For the accumulative total oil porosity, the relaxation time of accumulative bound oil porosity is correlated with the T_2 cutoff value. Therefore, with the T_2 cutoff value, the cutoff diameter r can be calculated. Pores smaller than r represent bound oil reservoir spaces, while those larger represent mobile oil reservoir spaces. The cutoff diameter r can be calculated according to the following equation:

 $R = \rho \times T_2 \times a$, where, based on the data provided by the laboratory, the value of a was set as 2 and shale surface relaxivity ρ as 10.

4.3.2 Evaluation of the Mobile Shale Oil

This chapter is a synthetical analysis of the experiment results. [Figure 5A](#page-8-0) shows the average pore cutoff diameters of shales with different lithofacies types, which vary to quite a large extent. The pore cutoff diameters of organic-rich grapholith, organic-

TABLE 8 | Connectivity and wettability evaluation of organic-rich grapholith shales and organic-rich mixed shales.

TABLE 9 | Sample experiment results of theT2 cutoff value and pore cutoff diameter.

TABLE 10 | Experiment results of saturation of shale oil fluids.

containing grapholith, and mixed shales were about 8 nm. Organic-rich mixed shales have a relatively large diameter of about 18 nm. Thus, it can be concluded that shale oil in 8–18 nm pores are mobile.

The pore fluid model used in this study is shown in [Figure 5B](#page-8-0). Water, fluid loss (natural gas, light hydrocarbon, and moisture dissipation), and oil (mobile and bound) exist in the shale pores. [Figure 5C](#page-8-0) shows the average oil saturation of shales with different

with different lithofacies types. (B) Pore fluids model. (C) Oil saturation average of shales with different lithofacies types. (D) Water saturation average of shales with different lithofacies types. (E) Bound oil saturation average of shales with different lithofacies types. (F) Mobile oil saturation average of shales with different lithofacies types.

lithofacies types. Both organic-containing and organic-rich mixed shales have an oil saturation of about 35%, while the value for organic-rich and organic-containing grapholith shales is about 20%. According to [Figure 5D](#page-8-0) presenting the average water saturation of different shales, organic-containing shales have the highest value, up to about 60%, while the water saturation of organic-rich grapholith, organic-rich mixed, and organic-containing mixed shales ranges from 30 to 40%.

Shale oil can be divided into mobile and bound types. According to [Figure 5E](#page-8-0), the bound oil saturation of organicrich and organic-containing mixed shales is about 25%, while that of organic-rich and organic-containing grapholith shales is about 13%. The content of mobile oil determines the productivity of the shale oil well, as shown in [Figure 5F](#page-8-0). Organic-rich mixed shale has a mobile oil saturation of about 12%, and the mobile oil situation of organic-rich grapholith shale is 10%, which doubles that in organic-containing grapholith and organic-containing mixed shale with 6% of mobile oil saturation.

5 CONCLUSION

This study selected cores from the Jurassic Lianggaoshan Formation in the southeast Sichuan Basin of the upper Yangtze Region in southern China to analyze their TOC content and mineral constituents. Then, we divided shales into different lithofacies types and conducted the spontaneous imbibition, NMR, and centrifuge experiments to analyze pore connectivity, wettability, and shale mobility. The conclusions obtained are as follows:

- 1) The light shale oil in pores above 8–18 nm in size is mobile in shales of different lithofacies types.
- 2) The TOC content is the highest in organic-rich grapholith shales, mostly with moderate to good pore connectivity and comparably bad connectivity. Besides, the majority has oilprone wettability, and the rest has water wettability. The mobile oil has high saturation. Overall, the TOC content is relatively high with moderate to good pore connectivity for organic-rich mixed shales, with a few shales having bad connectivity. The pores tend to be water-prone or water wetting with the highest mobile oil saturation.
- 3) The TOC content in organic-containing grapholith and mixed shales is moderate with bad-moderate pore connectivity mostly. A small number of the former type of shales have good connectivity. The wettability of organic-containing grapholith shales is complex, including oil, mixed, oilprone, and water-prone wettability, while the organic-

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containing mixed shales are mixed wetting with moderate mobile oil saturation.

4) The TOC contents for organic-poor grapholith shales and mixed shales are low. Most shales have bad-moderate pore connectivity, with only a few possessing good connectivity. Besides, these shales are characterized by complex wettabilities, which include water wetting, oil-prone wetting, and water-prone wetting.

DATA AVAILABILITY STATEMENT

The raw data supporting the conclusion of this article will be made available by the authors, without undue reservation.

AUTHOR CONTRIBUTIONS

KZ, ZJ, YS, and CJ contributed to the conception and design of the study. KZ organized the database. ZJ performed the statistical analysis. KZ, ZJ, YS, and CJ wrote the first draft of the manuscript. XY, XW, LZ, FH, YY, YZ, PL, LT, XC, and ZZ wrote sections of the manuscript. All authors contributed to manuscript revision, read, and approved the submitted version.

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