1. INTRODUCTION

Shale is a fine-grained sedimentary rock with fissility, as well as a broader classification of mudstone.\(^1\) Shale having TOC > 2% refers to organic-rich shale,\(^2\) which acts as an important hydrocarbon source, shale oil and gas reservoir, and seal rock in conventional reservoirs.\(^3,4\) Organic-rich shale has generated oil resources, which have been stored in the organic-rich intervals or transported into adjacent, continuous organic-poor intervals.\(^5\) Organic-rich lacustrine shales account for nearly 20% of the conventional oil resources but also turn out to be even more important unconventional oil and gas reservoirs worldwide.\(^6-8\) Organic-rich lacustrine shales are widely distributed all over China, for example, the Eocene Shahejie Formation in Bohai Bay Basin in eastern China,\(^9,10\) the Upper Cretaceous Qingshankou Formation and Nenjiang Formation in Songliao Basin in northeast China,\(^11\) the Upper Jurassic Yanchang Formation in Ordos Basin in central China,\(^12\) and the Middle Permian Lucaogou Formation in Junggar Basin and Santanghu Basin in northwestern China.\(^13\) Lacustrine mudstones have restricted geographical distributions, high stratigraphic variability, mixed kerogen types, and vast total organic carbon (TOC) variations when compared to their marine counterparts. This heterogeneity demands rigorous characterization of lacustrine mudstones to better understand the reservoir characteristics and guide hydrocarbon exploration and production.

The reservoir properties and storage mechanism in the shale are quite different and complex than conventional reservoirs, and it may contain free compressed gas, dissolved gas, and adsorbed gas.\(^15-20\) After successful horizontal drilling and hydrofracking, shale gas reservoirs gain huge attention. Different types of pores within the shale gas reservoirs are micro- to nanoscales in size with random distribution.\(^21,22\) The effective evaluation of shale gas reservoirs is quite difficult because of their

1. Microscopic Reservoir Characteristics of the Lacustrine Calcareous Shale: An Example from the Es\(^4\) Shale of the Paleogene Shahejie Formation in Boxing Sag, Dongying Depression

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ABSTRACT: The Es\(^4\) shale is taken as the main research object to understand and describe the reservoir characteristics in Boxing Sag, Dongying Depression. Through core observation, X-ray diffraction, thin section observation, field-emission scanning electron microscopy analysis, and low-pressure nitrogen gas adsorption experiment, the reservoir space of the Es\(^4\) shale including pore types, pore size, and pore structure characteristics was elucidated. The study shows that the shale in the Boxing Sag has the following characteristics: (1) the reservoir space in the study area is diverse, with the development of inorganic pores, organic pores, and microfractures. The higher the content of calcite and organic matter, the more favorable the development of intergranular pores, dissolution pores, and organic matter pores and (2) complex pore structure. The average Barrett–Joyner–Halenda pore size of calcareous shale is 6.5–22.8 nm, and the Brunauer–Emmett–Teller cumulative specific surface area is 0.7588–4.744 m\(^2\)/g. According to the morphological analysis of the adsorption and desorption curve, it is found that the shale samples in the target interval are mainly ink-bottle-shaped, cylindrical, and slit-shaped pores. The shale samples with relatively well-laminated intervals are mainly composed of ink-bottle-shaped and cylindrical pores, while the samples with relatively unlaminated intervals and high clay mineral content are mainly composed of slit-shaped pores. The contents of clay minerals and calcite are correlated with pore volume, specific surface area, and pore size, which further indicates the controlling effect of clay minerals and carbonate mineral components on pore structure parameters. This study not only helps us to understand the distribution of various micropores/nanopores in the lacustrine shale but also acts as a guiding note for the characterization of the shale oil reservoir, which ultimately offers a theoretical foundation for lacustrine shale oil exploration and development.
higher level of heterogeneity. The shale oil reservoirs can be characterized and evaluated comprehensively with the help of lithofacies analysis that is directly correlated to the mineralogical composition, geochemistry, petrology, and petrophysical parameters of rocks. Different lithofacies have different pore types and each pore type has diverse physio-chemical properties (structure and pore surface properties) that cause significant variations in the reservoir properties of shale.

Currently, the research on reservoir properties in the Jiyang Sub-basin is a prime focus of many researchers. In this study, we conducted a detailed analysis of whole-rock mineral composition, thin section petrography, field-emission scanning electron microscopy (FE-SEM) analysis, and low-pressure N\textsubscript{2} adsorption analysis to describe the reservoir characteristics of the Es\textsubscript{4} shale of the Boxing Sag in the Dongying Depression. The main objectives of this research work are (i) microscopic characteristics of lacustrine shale pore types, (ii) the role of

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Figure 1. Regional geological background and location map of the study wells. (A) Geographic map of China showing the location of Bohai Bay Basin, (B) geological map of the Bohai Bay Basin showing the location of Dongying Depression, and (C) geological map showing the location of the Boxing Sag in the Dongying Depression and the study well (modified after Khan et al.\textsuperscript{10}).
mineralogical heterogeneity in pore evolution, and (iii) the effect of TOC contents in pore evolution. This study can be useful to provide a simple approach to characterize pore types for lacustrine sediments and to examine the effect of mineralogy and organic matter on the development of different pore types in the study area. This study not only helps to understand the

Figure 2. Stratigraphic column of the Boxing Sag, Dongying Depression, Bohai Bay Basin. The strata in the red box show the interval of investigation (modified after Khan et al. [10]).
distribution of various micropores/nanopores in the lacustrine shale but also acts as a guiding note for the characterization of the shale oil reservoir, which ultimately offers a theoretical foundation for lacustrine shale oil exploration and development.

2. GEOLOGICAL SETTING

Bohai Bay Basin (BBB) is a Cenozoic rift basin with an area of 200,000 km$^2$ located on the eastern coast of China (Figure 1). From northeast to southwest, it is divided into seven sub-basins, namely, the Linqing, Jizhong, Jiyang, Changwei, Huanghua, Bozhong, and Liaohe, sub-basins (Figure 1). Among these sub-basins, Jiyang represents a typical rift basin of the Mesozoic to Cenozoic age in the southeast of BBB. The Jiyang sub-basin is composed of four secondary depressions: Dongying, Huimen, Zhanhua, and Chezhen, which are separated by Chengdong Uplift, Chenjiazhuang Uplift, and Yihezhuang Uplift (Figure 1). The Dongying Depression is located in the southeast of the

### Table 1. Mineral Composition of Es$_4^*$ Shale in the Boxing Sag, Dongying Depression

| depth (m) | quartz (%) | plagioclase (%) | calcite (%) | pyrite (%) | Fe-dolomite (%) | clay (%) | siderite (%) | TOC (%) |
|-----------|------------|-----------------|-------------|-----------|----------------|---------|--------------|--------|
| 3450.7    | 17.6       | 2.3             | 42.4        | 2.4       | 10.8           | 24.5    | 0            | 1.21   |
| 3450.75   | 15.9       | 0               | 54.5        | 0.4       | 10.3           | 18.9    | 0            | 1.21   |
| 3453.85   | 6.4        | 1               | 67.9        | 2.3       | 17             | 5.4     | 0            | 0.97   |
| 3454.4    | 10         | 0               | 71          | 3.7       | 9.7            | 5.4     | 0.2          | 1.09   |
| 3456.7    | 10.5       | 4.4             | 72.7        | 1.3       | 5.4            | 5.7     | 0            | 1.04   |
| 3455.8    | 17.5       | 3               | 40.7        | 5         | 8.8            | 22.8    | 2.2          | 1.51   |
| 3456.0    | 9.8        | 1               | 78.2        | 0.7       | 4.5            | 5.6     | 0.2          | 1.13   |
| 3456.75   | 5          | 0.6             | 89.3        | 0.4       | 2.5            | 2.2     | 0            | 1.14   |
| 3456.14   | 16.6       | 0               | 61.3        | 1.6       | 5.7            | 14.8    | 0            | 2.6    |
| 3456.55   | 16.7       | 1.5             | 58.8        | 1.8       | 16.5           | 4.7     | 0            | 1.47   |
| 3456.95   | 29.2       | 0               | 35.7        | 10        | 14.3           | 10.8    | 0            | 4.01   |
| 3456.26   | 12.3       | 1               | 52.7        | 1.8       | 28.4           | 3.8     | 0            | 1.05   |
| 3456.9    | 13.8       | 0               | 56.4        | 2.2       | 24.3           | 3.3     | 0            | 1.84   |
| 3456.38   | 9.5        | 0               | 75.2        | 0.9       | 6.6            | 7.8     | 0            | 0.88   |
| 3456.35   | 10.1       | 1.2             | 75.8        | 1         | 8.3            | 3.6     | 0            | 1.11   |
| 3456.45   | 32.5       | 12.6            | 7           | 8.8       | 9.8            | 28.8    | 0.5          | 5.54   |
| 3456.6    | 12.4       | 2.6             | 66.4        | 1.4       | 8.2            | 9       | 0            | 2.06   |
| 3456.67   | 15.9       | 1.9             | 62.8        | 1.8       | 7.5            | 10.1    | 0            | 1.39   |
| 3456.86   | 16.9       | 1.5             | 65.6        | 1.3       | 9.7            | 5       | 0            | 1.39   |
| 3456.9    | 7.9        | 0.1             | 81          | 1         | 5.8            | 4.2     | 0            | 1.25   |
| 3456.97   | 14.8       | 1.7             | 68.3        | 1.2       | 8.4            | 5.6     | 0            | 2.22   |
| 3456.95   | 9.2        | 2.5             | 74.8        | 2.4       | 2.8            | 8.3     | 0            | 2.73   |
| 3457.18   | 10.5       | 2.6             | 73          | 1.7       | 2.2            | 10      | 0            | 2.73   |
| 3457.87   | 27.3       | 2.6             | 43.4        | 3.4       | 5.7            | 17.6    | 0            | 2.79   |
| 3457.77   | 8.3        | 1               | 78.4        | 1.4       | 4.6            | 6.3     | 0            | 1.48   |
| 3457.52   | 22.9       | 5.8             | 11.3        | 4.6       | 35.2           | 19.9    | 0.3          | 4.35   |

Figure 3. Compositional characteristics and distribution of different mineral types of the Es$_4^*$ shale from FY1 well in the Boxing Sag.
Bohai Bay Basin, eastern China. It covers an area of 5700 km² and is bounded by the Luxi and Guangrao uplifts in the south, the Tuoshengyong fault, Chenjiazhuang uplift and Binxian uplift in the north, the Qingtuozi uplift in the east, and the Linfanjia uplift in the west. The Dongying Depression is bounded by the Luxi and Guangrao uplifts in the south, the Tuoshengyong fault, Chenjiazhuang uplift and Binxian uplift in the north, the Qingtuozi uplift in the east, and the Linfanjia uplift in the west. The Dongying Depression is composed of several secondary structural units including the Boxing Sag, Lijin Sag, Niuzhuang Sag, Minfeng Sag, northern steep slope belt, central anticline belt, and southern slope belt. The Boxing Sag is located in the southwestern part of the Dongying Depression in the Bohai Bay Basin, eastern China. It covers an area of 1320 km² approximately and is bounded by Sichun Fault in the east, the Gaoqing Fault in the west, the Luxi Uplift in the south, and the Chunhua-Caoqiao Fault in the north.

Stratigraphically, it is composed of Cenozoic to Quaternary strata including Kongdian Formation, Shahejie Formation, Dongying Formation, Guantao Formation, Minghuazhen Formation, and Pingyuan Formation (Figure 2). The Eocene Shahejie Formation is further divided into four members (Figure 2), that is, first member, second member (upper and lower), third member (upper, middle, and lower), and fourth member (upper and lower). The organic-rich shale from the Es1 shale is chosen as a research object in this study. The study area is bounded by the Luxi and Guangrao uplifts in the south, the Tuoshengyong fault, Chenjiazhuang uplift and Binxian uplift in the north, the Qingtuozi uplift in the east, and the Linfanjia uplift in the west.

3. RESULTS AND DISCUSSION

3.1. Characteristics of Mineral Composition and TOC Contents. The mineral composition and TOC content play an important role to classify shale lithofacies and characterize of shale reservoirs. The mineral composition and TOC contents of 27 bulk samples from the Es1 shale member of the Eocene Shahejie Formation from well FYP1 in the Dongying Depression are shown in Table 1. The vertical distribution of minerals and TOC content in Es1 shale is very variable and complicated. The Es1 shale is mainly composed of quartz, plagioclase, calcite, Fe-dolomite, clay minerals, and pyrite (Figure 3).

In Es1 shale, quartz ranges from 5 to 32.5% (average 14.71%) (Table 1). Quartz is mainly clay to a silt-sized mineral that is distributed randomly throughout the shale interval (Figure 4A,B). The concentration of quartz in Es1 shale increases from top to bottom in the study area (Figure 3). The content of plagioclase ranges from 0 to 12.6% with an average of 2.22% (Figures 3, 4B, Table 1). Calcite ranges from 7 to 89.3% (avg 59.6%) in the Es1 shale with three different morphologies including micrite, sparite (recrystallized calcite), and fibrous calcite (Figure 3, Table 1). Various laminations are composed of micrite, sparite, and fibrous calcite in the study area (Figure 4), while other laminations are composed of OM and mixed (clay, quartz, and OM) laminae (Figure 4C).

The TOC content ranges from 0.88 to 5.54 wt % (avg. 1.98 wt %) (Table 1). The TOC content in the Es1 shale increases with the increasing burial depth. The TOC content is directly proportional to quartz and inversely proportional to calcite with the increasing burial depth (Figure 3). The OM is distributed as OM laminae in the study area (Figure 4A,E,F). Some algae and other biogenic fragments are also observed during thin section petrography (Figure 4C).

3.2. Characteristics of Pore Types Based on FE-SEM Analysis. FE-SEM analysis revealed that the Es1 shale mostly contains inorganic InterP pores, IntraP pores, intercrystalline pores, dissolution pores with fewer OM pores, and microfractures.
InterP pores are mostly formed among quartz, calcite, and dolomite minerals. However, these pores are mostly encountered between the calcite crystals in the Es4 shale. InterP pores of calcite are mostly filled with authigenic quartz, clay, and dolomite, while intraP pores are formed within calcite and dolomite, 3462.6 m, well FYP1. (B) Calcite dissolution pores filled with organic matter, authigenic quartz, and clay, 3462.6 m, well FYP1. (C) InterP pores formed among different minerals (e.g., calcite, quartz, and dolomite) and intercrystalline pores of pyrite, 3462.6 m, well FYP1. (D) Calcite interP pores are filled with authigenic quartz, dolomite, and clay, 3466.7 m, well FYP1. (E) Calcite interP pores are filled with authigenic quartz; dissolution pores are formed in the bioclastic material, 3466.7 m, well FYP1. (F) Pyrite intercrystalline pores, authigenic quartz filling in calcite interP pores, 3466.7 m, well FYP1. (G) Distribution of organic matter pores, 3469.75 m, well FYP1. (H) Dolomite and authigenic quartz fill the calcite interP pores, while organic matter pores are formed in clay, 3469.75 m, well FYP1. (I) Intercrystalline pores of clay are filled with organic matter, pyrite, authigenic quartz, and euhedral dolomite, while interP pores are formed between authigenic quartz, calcite, and dolomite, 3472.87 m, well FYP1. (J) Distribution of intercrystalline pores (interC pores) in the sparry calcite laminae and horizontal microfractures filled with fibrous calcite, 3469.75 m, well FYP1. (K) Distribution of horizontal and oblique microfractures filled with calcite and organic matter, 3472.87 m, well FYP1. Abbreviations: interP = inter-particle, intraP = intra-particle, intraC = intercrystalline, and OM = organic matter.

InterP pores are mostly formed among quartz, calcite, and dolomite minerals. However, these pores are mostly encountered between the calcite crystals in the Es4 shale. InterP pores of calcite are mostly filled with dolomite, authigenic quartz, and clay minerals in the study area (Figure 5A,D). These pores generally have good connectivity and an effective pore

**Figure 5.** FE-SEM images show the morphology and characteristics of pore types in the Es4 shales. (A) InterP pores of calcite are filled with authigenic quartz, clay, and dolomite, while intraP pores are formed within calcite and dolomite, 3462.6 m, well FYP1. (B) Calcite dissolution pores filled with organic matter, authigenic quartz, and clay, 3462.6 m, well FYP1. (C) InterP pores formed among different minerals (e.g., calcite, quartz, and dolomite) and intercrystalline pores of pyrite, 3462.6 m, well FYP1. (D) Calcite interP pores are filled with authigenic quartz, dolomite, and clay, 3466.7 m, well FYP1. (E) Calcite interP pores are filled with authigenic quartz; dissolution pores are formed in the bioclastic material, 3466.7 m, well FYP1. (F) Pyrite intercrystalline pores, authigenic quartz filling in calcite interP pores, 3466.7 m, well FYP1. (G) Distribution of organic matter pores, 3469.75 m, well FYP1. (H) Dolomite and authigenic quartz fill the calcite interP pores, while organic matter pores are formed in clay, 3469.75 m, well FYP1. (I) Intercrystalline pores of clay are filled with organic matter, pyrite, authigenic quartz, and euhedral dolomite, while interP pores are formed between authigenic quartz, calcite, and dolomite, 3472.87 m, well FYP1. (J) Distribution of intercrystalline pores (interC pores) in the sparry calcite laminae and horizontal microfractures filled with fibrous calcite, 3469.75 m, well FYP1. (K) Distribution of horizontal and oblique microfractures filled with calcite and organic matter, 3472.87 m, well FYP1. Abbreviations: interP = inter-particle, intraP = intra-particle, intraC = intercrystalline, and OM = organic matter.
IntraP particles formed are observed within calcite and dolomite grains (Figure 5A). Dissolution pores are mostly associated with calcite grains and are mostly filled with OM, clays, and authigenic quartz (Figure 5B-E), while the intercrystalline and intracrystalline pores are mostly developed in clay, pyrite, and sparry calcite crystals (Figure 5C,F,I,J). These pores can provide significant pore spaces for hydrocarbon accumulation.

The OM pores are observed, but their quantity is limited in the study area. These pores are associated with calcite and clay (Figure 5G,H). In this study, the OM pores are not quite obvious. The formation mechanism of OM pores remains unclear. The lack of OM pores in the study area may result due to low organic maturity. The maturity of Es₄ shale is 0.88% in the study area.

Microfractures play an important role in the shale oil system because they offer effective reservoir spaces and enhance the hydrocarbon migration system. Two types of microfractures are observed in the studied shale including horizontal (bedding) and oblique (structural) microfractures, but their quantity is limited. The bedding microfractures are developed parallel to the bedding planes and formed due to low stress. They are usually filled with OM and fibrous calcite (Figure 5J). Structural microfractures are also known as tectonic fractures because these are formed due to tectonic stress. These microfractures are usually filled with calcite (Figure 5K). They can be easily observed under a polarizing microscope because they are very wide. These two types of microfractures serve as migration channels for hydrocarbons in shales. Thus, inorganic pores and microfractures played an important role in the shale oil storage and migration in the Boxing Sag, Jiyang sub-basin.

3.3. Microscopic Characteristics of Pore Types Based on LPNA Analysis. According to the IUPAC, pores in shale are divided into micropores (<2 nm), mesopores (2−50 nm), and macropores (>50 nm). Based on the IUPAC classification, isothermal adsorption curves of the Es₄ shale samples are type-II (Figure 6). These samples are marked with a type-H2 hysteresis loop except for one sample (3462.6 m) that shows a type-H4 loop. The gas adsorption curve is slightly concave when the relative pressure (P/P₀) was <0.5, and there is a gas monolayer adsorption process at this stage. An inflection stage appeared when the P/P₀ value was almost 0.5, and this stage showed the discrimination point of gas monolayer and multilayer adsorption.
adsorptions, and gas adsorption capacity began to increase at this stage. The gas adsorption curve showed strong convexity when \( P/P_0 > 0.5 \). The strong convexity of the adsorption curve indicates the presence of mesopores and macropores in this shale. At this stage, the process of \( N_2 \) gas multilayer adsorption occurred and adsorption capacity quickly rose. When the \( P/P_0 \) value was 0.5, there was a sudden declination in the desorption curve, which indicates the shape of the pores with large spaces.\(^{37}\) The H3 loop is narrow, and the adsorption curve is almost parallel to the adsorption curve (Figure 6A).\(^{38}\) H3 only elevates abruptly when reaching the saturated vapor pressure because of clear capillary condensation.\(^{39}\) On the other hand, the wide H2 hysteresis loop indicates the slow changes in the adsorption curve, while the desorption curve is steeper than the adsorption curve and drops vertically at moderate pressure (Figure 6B–D). The types of isothermal curves and hysteresis loops show that the Es\(_4\) shale is composed of slit-shaped and ink-bottle-shaped pores (Figure 6). A single pore structure is very limited due to the complexity of pore structures in shale. The loops are usually the combination of two or more types.

The adsorption volume of all the studied samples of the Es\(_4\) shale ranges from 0.000194 to 0.968141 cm\(^3\)/g with an average volume of 0.322882 cm\(^3\)/g (Table 2). The Brunauer–Emmett–Teller ( BET) specific surface area of the Es\(_4\) shale samples ranges from 0.030 to 1.194 m\(^2\)/g (avg. 0.454 m\(^2\)/g) (Table 2). The pore volume and the BET specific surface area mostly account for mesopores and macropores in the study area (Figure 7). The low-pressure nitrogen gas adsorption (LPNA) analysis indicates that the pore size varies from 1.1 to 204.9 nm (Figure 7). The average Barrett–Joyner–Halenda (BJH) pore volume is 0.322 cm\(^3\)/g in the studied shale intervals, which corresponds to the mesopores (Figure 7), while the average BET specific surface area of the Es\(_4\) shale samples is 0.454 m\(^2\)/g and it corresponds to micropores and mesopores (Figure 7). The pore volume of micropores and mesopores increased with the increasing specific surface area in the studied shale samples.

### 3.4. Role of Mineralogical Heterogeneity on Pore Evolution

A variety of different minerals possess variable chemical and mechanical stabilities during diagenesis.\(^{40}\) These variable properties play an important role in the development and preservation of different types of pores. Additionally, the sedimentary structures, for example, laminations, also play an important in this context. Laminated calcareous shale, vein-like fibrous calcite, and dolomitic shale intervals have a higher number of pores that are larger than 10 nm. The non-laminated and poorly laminated shale intervals are dominated by small-sized pores. The adsorption–desorption isotherms indicate that the pore morphology varied among different shale intervals containing variable mineral compositions. The siliceous minerals are dominated by the pores having <100 nm size with higher adsorption quantity. The siliceous minerals are followed by clay minerals and pyrite in this framework. Based on LPNA analysis, the siliceous minerals (quartz and plagioclase), clay minerals, and pyrite are positively correlated to the average pore volume and specific surface area (Figures 8A–C, 9A–C). It can be suggested that the hydrocarbon accumulation capacity and surface area increased with the increasing content of these minerals. Although clay minerals hosted intercrystalline pores, these pores are so small with strong surface adsorption capacity and area, which hinders the storage of movable liquid hydrocarbons. The relationship of carbonate minerals (calcite and Fe-dolomite) with pore volume and specific surface area is the opposite (Figure 8D,E, 9D,E). This relation suggested that these minerals do not provide storage spaces and are not favorable to free shale oil richness. The reason behind this negative relationship can be the action of calcite cementation and recrystallization that can reduce the space and area of the primary microcrystalline calcite. This action can destroy the primary intercrystalline pores and dramatically reduce their numbers in the study area.

### 3.5. Effect of TOC Contents on Pore Evolution

The content of TOC ranges from 0.88 to 5.54 wt % with an average of 1.98 wt % in the Es\(_4\) shale of the Boxing Sag (Table 1). The relationship of TOC content with pore volume and specific surface area is weakly positive (Figures 8F, 9F). The weak correlation indicates that OM pores are poorly developed in the Es\(_4\) shale. The reason behind this poor growth is the low maturity of the Es\(_4\) shale (0.88\%), which is lower than the threshold maturity (\( R_{o} = 1.2\% \)) to develop OM pores.\(^{41}\) In the Es\(_4\) shale, organic matter is composed of type-II kerogens,\(^{9}\) and so, the oil-prone type-II (liptinitic) kerogen can produce OM pores in large quantities with the consumption of organic matter;\(^{14}\) however, lower maturity of the Es\(_4\) shale does not meet the conditions to produce pores. Therefore, the generation of OM pores is very weak in the studied shale. Under these circumstances, the mineral-hosted pores act as the main contributors to the total pore morphology in the Es\(_4\) shale.

Overall, mineralogical composition plays an important role to control the pore structure parameters of the Es\(_4\) shale. Based on the shreds of this evidence, we can suggest that the inorganic pores are the key contributors to the development of pores that enhance the reservoir characteristics of the Es\(_4\) shale in Boxing Sag, Dongying Depression. The results achieved from the current research work not only help us to understand the distribution of various micropores/nanopores in the lacustrine shale but also act as a guiding note for the characterization of the shale oil reservoir, which ultimately offers a theoretical foundation for lacustrine shale oil exploration and development.

### 4. CONCLUSIONS

This study highlights the reservoir characteristics (pore types and their formation mechanism) of the lacustrine shale at micro- to nanoscales in the rift-related basin (Jiyang sub-basin). Based on these shreds of analyses, the overall research work elucidates the following main conclusions.

(1) In the Es\(_4\) shale, the clay and siliceous minerals are the main contributors to the total system. The quantity of

| depth (m) | average width (nm) | pore volume (cm\(^3\)/g) | pore surface area (m\(^2\)/g) | siliceous minerals (%) | calcite (%) | pyrite (%) | Fe-dolomite (%) | clay (%) | TOC (%) |
|----------|-------------------|--------------------------|-------------------------------|-----------------------|-------------|-----------|----------------|---------|--------|
| 3462.6   | 25.61             | 0.000194                 | 0.0030724                    | 13.3                  | 52.7       | 1.8       | 28.4           | 3.8     | 1.05   |
| 3466.7   | 26.48             | 0.000362                 | 0.185419                     | 17.8                  | 62.8       | 1.8       | 7.5            | 10.1    | 1.39   |
| 3469.75  | 24.33             | 0.000259                 | 0.119625                     | 11.7                  | 74.8       | 2.4       | 2.8            | 8.3     | 2.73   |
| 3472.87  | 25.02             | 0.968141                 | 1.194677                     | 29.9                  | 43.4       | 3.4       | 3.5            | 17.6    | 2.79   |

Table 2. Pore Structure Parameters With Mineral Composition and TOC Contents in the Es\(_4\) Shale
Figure 7. Pore volume (micro-, meso-, and macropore distribution) and pore-specific surface area of the Es4 shale in the Dongying Depression. (A–H) Pore volume and pore surface area with pore width (nm), which ultimately suggests that the studied shale samples are dominated by mesopores.
Figure 8. Relationship between different mineral compositions with pore volume in the Es₄ shale. (A–C) Positive relationship of clay minerals, siliceous minerals, and pyrite with incremental pore volume, (D,E) negative relationship between calcite and Fe-dolomite with incremental pore volume, and (F) weakly positive relationship of TOC contents with incremental pore volume in the study area.
Figure 9. Relationship between different mineral compositions with the pore surface area in the Es₁ shale. (A–C) Positive relationship of clay minerals, siliceous minerals, and pyrite with the incremental pore surface area, (D,E) negative relationship between calcite and Fe-dolomite with the incremental pore surface area, and (F) weakly positive relationship of TOC contents with the incremental pore surface area in the study area.
OM pores is very rare in this shale due to their lower maturity. (2) The relationship of carbonate minerals (calcite and Fe-dolomite) with pore volume and specific surface area is the opposite. This relation suggested that these minerals do not provide storage spaces and are not favorable to free shale oil richness. The reason behind this negative relationship can be the action of calcite cementation and recrystallization that can reduce the space and area of primary microcrystalline calcite. This action can destroy the primary intercrystalline pores and dramatically reduce their numbers in the study area. (3) The average pore diameter is 25.3 nm, which corresponds to the mesopores. The average pore volume and BET specific surface area are 0.322 and 0.454 cm$^2$/g, respectively. (4) The shape of the pores varies from slit to ink-bottle-shaped. (5) The whole pore system after detailed LPNA analysis suggests that the meso- and macropores are also abundant and act as the main contributors to the pore network system in the Es$_4$ shale.

5. METHODOLOGY

5.1. Sample Collection. The samples were collected from the Es$_4$ shale of the Eocene Shahejie Formation in the Boxing Sag, Dongying Depression. Approximately 27 core samples were obtained for this research work from FYP1 well in the study area. The study interval of the Es$_4$ shale members ranges from 3450.7 to 3474.52 m (Figure 2). The lithology of the Es$_4$ shale is composed of gray to dark-gray fine calcareous shale.

5.2. Sample Preparation and Thin Section Analysis. Thin sections are made from the research interval’s core samples. Core samples were repeatedly grounded to achieve a standard thickness ranging from 0.03 mm. After a thorough examination of hand specimens that reflect the main features of the Es$_4$ shale of the Eocene Shahejie Formation, 27 thin sections were prepared for thin section observation. The detailed observation of each sample was performed by using a polarizing microscope (Model: Leica DM4).

5.3. X-Ray Diffraction and TOC Analyses. A total of 27 representative samples from the Es$_4$ shale were chosen and analyzed for whole-rock mineralogical composition by a Panalytical X'Pert PRO X-ray diffractometer enabled with a Cu X-ray target (40 kV, 40 mA). Each sample weighing 5 g was oven-dried at 40 °C for 2 days and grounded to less than 40 mm in size to disperse the mineral fractions by using an agate mortar. The angle range for the analysis was 5−10°. Various phases of different minerals and their relative abundances (wt %) were identified by using computer diffractogram analysis. The TOC content is a key factor for the source rock evaluation because it reflects the distribution of organic matter (OM). A total of 27 representative shale samples were chosen for TOC using a carbon and sulfur analyzer (model: LECO CS744) that worked at 1200 °C in a closed system. The shale samples were <100 mesh in size, and then, 0.13−0.14 g of each sample was added to a container and soaked in a diluted HCl solution (volume ratio = 1:7) for 24 h to remove inorganic carbonate contents. The samples were washed with distilled water to eliminate acid impurities and placed in an oven for 2 h at 60 °C. Each sample was placed in the LECO CS744 analyzer for TOC analysis.

5.4. FE-SEM Analysis. Based on thin section observation and mineral composition, representative samples with different mineralogy and textures were cautiously selected for FE-SEM analysis to observe the arrangement and distribution of different minerals, OM, various pores, and microfractures. The representative samples were polished with argon ions and then coated with platinum to enhance conductivity. The analysis was completed on a Zeiss Crossbeam-550 (Gemini-2) scanning electron microscope.
5.5. Low-Pressure Nitrogen Adsorption Analysis. The pore morphology including PSD, pore-volume, and surface area of Es3 shale was observed by using the LPNA technique. A total number of four representative samples of shale were selected, and then, each sample was crushed and grounded to powder form (60–80 mesh size). The required powdered samples (0.5 g from each sample) were degassed for 6 h at 110 °C for pore surface cleansing. The LPNA test was carried out at −195.8 °C temperature to characterize the pore structure.36 The pore surface area is calculated by using the BET model,43 while the BJH model is used to compute PSD and pore volume.43,44 The isotherms and hysteresis loops were analyzed based on previously defined models,45 as shown in Figure 10.

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Notes
The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

This research work was supported by the National Natural Science Foundation of China (no. 41972099) and the project of the “Jiyang depression continental faulted lacustrine Basin shale oil National demonstration zone”. The authors would like to thank Shengli Oil Company Dongying for providing core samples and other necessary geological data from the FYP1 well for this research work. The authors would also like to acknowledge the School of Geosciences, China University of Petroleum East China for financial support.

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