The characteristics of nano-pore of the 3rd member shale from Dongying Formation of Paleogene in Liaozhong Sag

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Abstract. The reservoirs structure and pore characteristics of shale have greatly influenced the reservoir, adsorption and migration of oil and gas, and have become one of the important contents of reservoir physical characteristics evaluation. The organic-rich shales in the 3rd member shale of the Dongying Formation in Paleogene, Liaozhong sag, Bohai bay basin were studied in this paper. Firstly, the surface of shale sample was etched by argon ion polishing technology, and then the microscopic pore morphology was observed by field emission environment scanning electron microscope. The results show that: 1) the pores of shale reservoir in 3rd member shale of the Dongying Formation from Liaozhong sag are divided into inorganic pores (including intergranular pores, intragranular pores, interstratal pores and intercrystalline pores), organic pores and microfractures; 2) the intergranular pores are largely developed, intragranular pores and interlaminar pores are relatively developed, micro-cracks are less developed, and intergranular pores and organic pores are rarely developed.

1. Introduction
In recent years, with the rapid development of shale gas exploration and development at home and abroad, shale as an unconventional oil and gas reservoir has attracted more and more scholars' attention and research [1-4]. Compared with conventional reservoirs, fine-grained shale reservoirs have a higher content of clay minerals and a smaller pore size and denser structure [5]. Therefore, the structure and pore characteristics of shale reservoir greatly affect the adsorption and migration of oil and gas, which has become one of the important contents of reservoir physical property evaluation.

The Bohai Bay Basin has good shale gas exploration potential, but a few studies and reports have been done before [6, 7]. The study on the shale gas of the Liaozhong Sag is still in the blank stage. Therefore, the organic-rich shales in the 3rd member shale from Dongying Formation of Paleogene, Liaozhong sag, Bohai Bay Basin were studied in this paper. Firstly, the surface of shale sample was etched by argon ion polishing technology, and then the microscopic pore morphology was observed by field emission environment scanning electron microscope. Finally, the characteristics and development of different pore types were studied.

2. Geological background
The Liaodong Bay area refers to the north-eastern part of Bohai Sea, which is a natural extension of the lower Liaohhe Basin to Bohai Sea, and is a secondary tectonic unit of Bohai Bay Basin. The
Paleogene in the Liaodong Bay area is divided into three sags and two uplifts, and there are five secondary structural units in total. From west to east, there are Liaoxi Sag, Liaoxi Low Uplift, Liaozhong Sag, Liaodong Uplift and Liaodong Sag, and all the structural units are distributed in the direction of northeast to southwest. Liaozhong Sag is the largest of the three sags, and its paleogene strata have the largest thickness and deepest burial. Long-term geological research and exploration practice have found that there are three sets of source rocks in Liaozhong sag, which are the 3rd member of the Shahejie Formation (E2s1), 1st member of the Shahejie Formation (E3s1) and 3rd member of the Dongying Formation (E3d3) [8]. The 3rd member shale of the Dongying Formation is the research object in this paper.

3. Samples and experiments

3.1. Samples and instruments
The samples were taken from the 3rd member of the Dongying Formation in the well JX1-1-1 and LD32-2N-1 in Liaozhong area. The instruments used in this experiment include PECS II 685 type precision etch coating system (GATAN, USA), Quanta 250 FEG type Field Emission Environmental Scanning Electron Microscope (FEI, USA) and Quantax200 Xflash type X-Ray Spectrometer (Bruker, Germany).

3.2. Experimental Process
Firstly, fresh rock samples with smooth surface were selected, and then argon ion polishing was carried out in PECS II 685 precision etch coating system. The high-speed ion beam bombardment can eliminate the roughness of shale sample surface and obtain high-quality surface. Finally, Quanta250 FEG field emission environment sem was used to observe high-quality samples. The accelerated voltage of SEM is 10.00kv, and the detector is Backscattered Electron (BE), which is used for image of morphology and composition contrast.

4. Result and discussion
Although scholars at home and abroad have different classification schemes for shale pores [9], overall, the main basis of pore classification is the two objective parameters of pore size and porosity, which correspond to structure classification and occurrence classification respectively. The structure classification focuses on quantitative analysis and evaluation, while occurrence classification focuses on qualitative description. The occurrence classification method of Louck et al. [10] was adopted in this paper, and the pores in shale were divided into inorganic pores, organic pores and micro-fractures.

4.1. Inorganic pores
The shale samples are rich in inorganic minerals, mainly clay minerals, and a small amount of siderite, pyrite, calcite, dolomite, quartz, plagioclase, potash feldspar, gypsum and apatite. Pores of different sizes occur between different mineral particles, within individual mineral particles, between the same mineral crystal, and between organic matter and minerals. Therefore, inorganic pores can be further divided into intergranular pores, intragranular pores, interlamellar pores and intercrystalline pores according to the occurrence position of pores (figure 1).
Figure 1. Pore characteristics of shale in the 3rd member shale in the Dongying Formation of Liaozhong Sag

a. Pores between granular minerals; b. Pores between granular minerals and clay minerals; c. Pores between clay minerals; d-f. Intragranular pores; g-h. Pores between clay mineral sheets; i. Pyrite intergranular pore; j. Organic matter pore; The arrows in the picture are used to indicate pores. The sample of picture i is from well JX1-1-1, and the samples of other images are from the well LD32-2N-1.

Intergranular pores, usually triangular and crescent in shape, with a length and width of microns, developed between different granular minerals (figure 1a). Irregular linear pores developed between granular minerals and clay minerals, no more than a micron in width and generally within a few microns in length (figure 1b). Intergranular pores are largely developed between different clay minerals, forming irregular linear and curved slits, generally less than 1 micron in width and several microns in length (figure 1c).

Intragranular pores are mainly distributed in the interior of granular minerals such as quartz, feldspar and calcite, which are formed by dissolution. The shape of the pores is generally round and oval, and the aperture is generally less than 1 micron (figure 1d, e, f).
Interlaminar pores are developed between layers of clay minerals such as kaolinite, illite, etc. They are generally wedge-shaped, irregular, reticulated, generally less than 1 micron in width and several microns in length (figure 1 g, h).

Intercrystalline pores also develop between the crystals in strawberry pyrite crystal, with pore sizes ranging from tens to hundreds of nanometers (figure 1 i).

4.2. Organic pores
Organic pores refer to the pores formed in the organic mass or after the organic matter generates hydrocarbon due to the reduction of volume. In this study, organic pores in shale form 3rd member shale of the Dongying Formation in the Liaozhong sag are not well developed. The shape of organic pore is nearly round, elliptic, half-moon and irregular. The pore size is generally no more than a few microns (figure 1 j).

4.3. Micro-fractures
The morphology and size of micro-fractures are quite different from that of organic and inorganic pores, and they are the main channel for adsorbed gas to dissociate from desorption, which is beneficial to the seepage of shale gas. The causes of micro-fractures include dehydration (clay minerals), compaction, dissolution (carbonate minerals), hydrocarbon thermal pressurization and tectonic stress. Microscopic observation showed that there are nanometer to micron micro-cracks in organic matter and clay mineral particles in shale samples, as well as between organic matter and clay mineral, but the number is not many (figure 1 k, l).

Table 1. The pore development degree of shale reservoir in 3rd member shale of the Dongying Formation from the Liaozhong sag

| Pore type          | Inorganic pore | Organic pore | Micro-fractures |
|--------------------|----------------|--------------|-----------------|
|                    | Intergranular  | Intragranular|                 |
|                    | pore           | pore         |                 |
| sketch map         | ★★★★          | ★★           | ★★              |
| development degree | ★★★☆☆☆         | ★☆☆☆         | ★☆              |

★★★★ represents largely developed; ★★★☆ represents relatively developed; ★☆☆ represents less developed; ★☆ represents rarely developed.

According to the observation results of Scanning Electron Microscope, the pore development degree of shale reservoir in 3rd member shale of the Dongying Formation from the Liaozhong sag is listed in table 1. The intergranular pores are largely developed, intragranular pores and interlaminar pores are relatively developed, micro-cracks are less developed, and intergranular pores and organic pores are rarely developed.

5. conclusion
The pores of shale reservoir in 3rd member shale of the Dongying Formation from Liaozhong sag are divided into inorganic pores (including intergranular pores, intragranular pores, interstratal pores and intercrystalline pores), organic pores and microfractures.

The intergranular pores are largely developed, intragranular pores and interlaminar pores are relatively developed, micro-cracks are less developed, and intergranular pores and organic pores are rarely developed.

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