In-situ SEM characteristics of dispersed organic matter in continental shale with its implication for dessert evaluation--A case study of Paleogene shale in the Cangdong Sag, Bohai Bay Basin, China

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Abstract. Organic matter (OM) in continental shale serves as both the source of oil and gas and the storage space in unconventional petroleum systems. However, directly identifying the types of organic matter under SEM is challenging when simultaneously observing minerals and pores. Kong2 Member(E2k2) of Paleogene in Cangdong sag of Bohai Bay basin is a typical continental shale oil layer in China. Based on the positioning observation technology combining field emission scanning electron microscope (FE-SEM) and fluorescence microscope, the in-situ SEM identification and observation of macerals were carried out, and the identification methods and characteristics of organic macerals were summarized. The results show that: (1) Organic macerals in E2k2 shale are divided into vitrinite, inertinite, liptinite and solid bitumen by external morphology, hardness, brightness, color, protrusion, pore and fracture development of organic matter, and further subdivided into multiple subcategories. Based on the SEM charging effect of the remaining oil, it is further confirmed that the shale movable oil and oil generation potential developed by lipoid group is the largest, while the shale movable oil and oil generation potential developed by vitrinite group and inertinite group is the worst; (2) The organic pores include primary pores and secondary pores. The pores of primary organic matter are derived from the biological structure of primary organic matter, and the secondary organic pores are developed during the thermal maturation of oily organic matter. Clay mineral catalysis, difference of hydrocarbon generation potential and residual pores of primary organic matter control the development of organic pores; (3) Calcareous-dolomitic shale and felsic shale are typical lithology formed in relatively dry and humid climate respectively, and the types of organic macerals are significantly different. Although the former has weak total hydrocarbon generation, it has stronger oil generation potential and is worthy of attention in dessert prediction and exploration.

Keywords. In-situ SEM characteristics; Dispersed organic matter; Maceral group; Shale oil; Bohai Bay Basin

1. Introduction

Dispersed organic matter (DOM) plays an important role in determining hydrocarbon generation potential of continental shale(Ko et al., 2018; Vandenbroucke and Largeau, 2006). Different from marine shale, continental shale oil reservoirs in China are mixed with extensive terrigenous organic debris, which is new in age and low in maturity, resulting in a variety of macerals of organic matter coexists in continental shale. Therefore, the strong heterogeneity and complexity of DOM in continental shale hinder the correct understanding and evaluation on shale reservoir (Curtis et al., 2012; Loucks et al., 2012). Since that the continental shale has the characteristics of mixed fine particle deposition at the micro-scale, high-resolution field emission scanning electron microscopy (FE-SEM) observations are an indispensable tool for identifying shale minerals and pores(Guo et al., 2018; Milliken et al., 2013). However, the DOM displays in black because of low density, directly identifying the types of organic matter under SEM is challenging when simultaneously observing minerals and pores. The SEM backscatter cross section secondary imaging method based on charging effect of electron beam is used to identify the residual oil (i. e. crude oil not lost in the experimental vacuum) in the micro-nano pore of the reservoir, owing to the charging effect of non-conductive

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samples in the SEM imaging process (Jin et al., 2021; Wang et al., 2015).

Bohai Bay Basin is an important petroliferous rift basin in eastern China, taking the lead in realizing the industry discovery of shale oil (Fig. 1). The shale of the second member of the Paleogene Kongdian Formation (E2k2) in Cangdong sag develops a semi-deep lake deep lake facies shale oil reservoir system formed in a semi-arid humid climate, with felsic shale (FS), calcareous dolomitic shale (CDS) and mixed shale (MS) alternately deposited spatially. A lot of researches have been done in the aspects of mineral composition, geochemical characteristics of organic matter, automatic mineral identification by AMICS and high-resolution SEM imaging at an early stage. High TOC, felsic, high-frequency lamina and low clay minerals are important component characteristics of E2k2 shale, and the laminar FS was regarded as the main exploration lithology of Cangdong Sag (Zhao et al., 2017, 2019). What are the similarities and differences in the in-situ characteristics of the DOM between the CDS formed in a relatively arid climate and the FS formed in a humid climate? How to identify the types and characteristics of DOM in-situ by high-resolution SEM? What impact will it have on the evaluation of shale oil desserts? The above issues, on the one hand, are seriously restricting the evaluation of shale oil resource potential and the dessert prediction in the Bohai Bay Basin; on the other hand, are also the weak field of basic research of continental shale.

In view of this, a combination of optical microscopy and FE-SEM were used to systematically analyze the types and characteristics of DOM and the distribution of movable oil in E2k2 shale, particularly the CDS and FS formed in arid and humid climate respectively, with discussion on implication of macerals on the hydrocarbon distribution, migration and and dessert evaluation. This study is expected to provide a theoretical reference for shale oil resource evaluation and exploration and development in sedimentary basins with similar geological background.

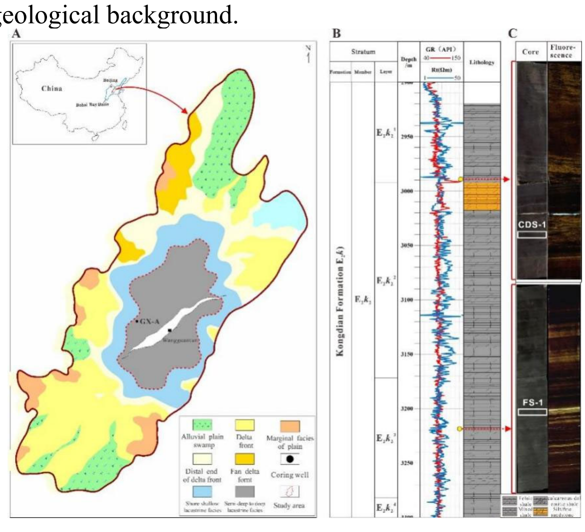

Fig. 1. (A) Sedimentary facies distribution of semi-deep to deep lacustrine shale of the Paleogene E2k2 in Cangdong Sag, Bohai Bay Basin (Zhao et al., 2017); (B) Generalized stratigraphy of Paleogene E2k2 in Cangdong Sag, showing that three main fine-grained sedimentary shales developed in frequent interbedding way including felsic shale (FS), calcareous-dolomitic shale (CDS) and mixed shale(MS).

2. Geological background

Bohai Bay basin is a Cenozoic inland lake basin developed under tensile tectonic background. Cangdong sag in the study area is sandwiched between Cangxian uplift, Xuhei uplift and Kongdian uplift (Fig.1). During the sedimentary period of Paleogene E2k2, Cangdong sag was an inland closed lake basin, which was in a subtropical semi-arid to humid climate. A set of sedimentary formation with a thickness of 400 ~ 600m, mainly dark shale, mixed with thin to medium siltstone, medium-fine sandstone and argillaceous dolomite. The sedimentary facies change regularly from the edge of the lake basin to the cente, showing a delta front subfacies in the edge of the lake basin with mainly developed conventional medium-fine sandstones dominated, a pre-delta subfacies and semi-deep lake subfacies in the middle of the lake basin, which is the main source rock composed of shale and argillaceous dolomite, and a far end of the delta front and gravity flow sedimentation in the transitional zone of both above.

Preliminary research results reveal that (Han et al., 2021): (1) X-ray diffraction (XRD) shows that E2k2 shales are mainly composed of quartz, feldspar, dolomite, calcite, analcite, pyrite, clay and other minerals and lithology mainly includes FS, CDS and mixed shale (MS) (Fig.1B); (2) CDS, formed in a relatively dry climate, has relatively lower TOC (average = 2.23%), S1 (average = 3.01 mg/g) and S2 (average = 10.25 mg/g), but higher OSI(TOC/S1*100) (average = 125.38 mg/g TOC). However, FS formed in a relatively humid climate, has relatively higher TOC (average = 5.61%), S1 (average = 3.89 mg/g) and S2 (average = 31.87 mg/g), but lower OSI (average = 68.80 mg/g TOC); (3) The analysis of organic matter types showed that Tmax and hydrogen index (average = 442.95℃ and average = 418.61 mg/g respectively) of CDS were slightly lower than those of FS (average = 442.95 ℃ and average = 418.61 mg/g respectively). The main type of organic matter in both shales were kerogen type I and II1, but the organic matter type of FS was better; (4) In-situ imaging under SEM showed that the laminar CDS and FS had a high abundance of organic matter along the laminar distribution, with complex and indistinguishable organic macerals.

3. Materials and methods

The main workflow involved in this study as followed:

Firstly, macerals are identified under an optical microscope with reflected white light and oil immersion. The surfaces of petrographic pellets are subsequently cleaned to remove immersion oil, and then the pellets are cut for SEM sample preparation. The same surfaces as observed under reflected-light microscopy are Ar ion-milled for SEM observations, and the same fields of view need to be located under the SEM. Microfractures, pyrite framboids, or fossils can be used as markers for easier identification. Alternatively, OM can be first examined on ion-milled surfaces under SEM, and then samples can be moved to the reflected-light microscope for maceral identification. In this case, additional sample preparation is unnecessary.

For the identification method of shale movable oil based on charging effect, seen in Jin et al., 2021; Wang et al., 2015. ImageJ is mainly used for the BSD image processing, and the proportion of shale movable oil is obtained through the statistics of 100 field of view.

Microscope observation, Ar-ion polishing and FE-SEM experiment were carried out at theKey Laboratory of Deep Oil and Gas, China University of Petroleum. In addition, the basic research data of 23 CDS and 35 FS were provided by PetroChina Dagang Oilfield Research Institute.

4. Results

The petrographic classification of DOM in continental shale is mainly based on that of coal rock. In general, there are four macerals developed in E2k2 lacustrine shale, among which vitrinite, inertinite and liptinite are primary organic matter, while the secondary organic matter is mainly solid bitumen(SB) and movable oil, which is the result of the transformation of oil prone macerals during thermal maturation. Different from the coal rocks, there is almost no zooclasts in E2k2 shale.

4.1 Petrographic classification of DOM

4.1.1 Vitrinite

Vitrinite is formed by the gelation of roots, stems and bark of plants. The different degree of gel leads to the different preservation of primitive plant cell structure. It has low oil generation potential and high methane generation potential. OM rich in vitrinite belongs to a typical type-III kerogen. It is usually dispersed in the mineral matrix of shale in the form of small particles (Fig. 2A-A’).

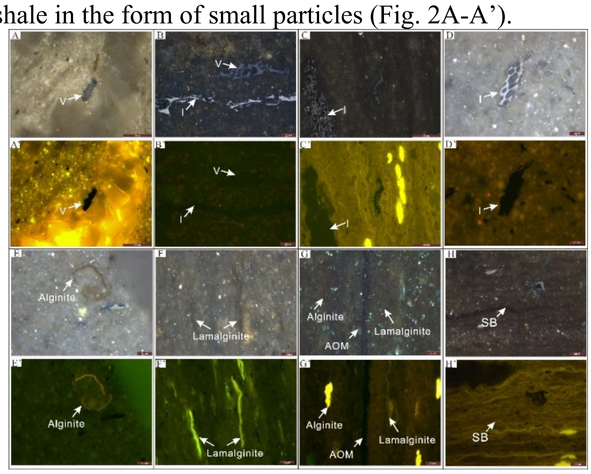

Fig. 2. Photomicrographs of DOM in Paleogene E2k2 shales in reflected white light and oil immersion(A-H) and in fluorescence mode (A’-H’), including primary OM of vitrinite(A-A’), inertinite (B-B’, C-C’), zooclasts(D-D’) and liptinite(E-G, E’-G’), and secondary OM of SB(H-H’). V=vitrinite; I=inertinite; Z=zooclasts; AOM=amorphous organic matter; SB=solid bitumen; OM= organic matter.

4.1.2 Inertinite

Inertinite is a kind of macerals formed from wood microstructure through filament carbonization produced by fire or oxidation before deposition. It has little hydrocarbon generation potential and is usually classified as type IV kerogen. Even in the early mature stage, inertinite has high reflectivity. Under the optical microscope, it is best to distinguish between inertinite and vitrinite by the color of reflected light (Fig. 2B-3D’).

4.1.3 Liptinite

Lipinite is one of maceral that is generally easy to produce oil, with high hydrocarbon generation potential and usually classified as type I/II kerogen. The lipinite macerals in E2k2 shale are mainly including alginite (such as telalginite and lamalginite) and amorphous organic matter(AOM). Alginite appears as slender rods. Under the blue light irradiation, the Alginite at the early mature stage showed strong yellow-green fluorescence. AOM refers to unstructured OM in shale, which is derived from microbial degradation of phytoplankton, zooplankton and bacteria (Fig. 2E-G’).

4.1.4 Solid bitumen

SB is a kind of secondary organic matter formed during kerogen transformation and hydrocarbon generation, and its shape is limited by the pore shape it fills (Fig. 2H-H').

4.2 In-situ characteristics and petrography of DOM under SEM

Based on the identification and verification by optical microscopy, the microscopic features of in organic macerals of the argon ion-polished shale were obtained under FE-SEM observation. Specifically, the types of microcomponents can be identified according to comprehensive factors such as the external morphology of organic matter, contact relationship with surrounding minerals, organic matter color, brightness, protrusions, organic pore development characteristics and fracture development characteristics.

4.2.1 Vitrinite

Vitrinite can be further divided into provitrinite, collinite and vitrodetrinite (Fig.3 A1-A4).

(1) Provitrinite, a kind of vitrinite with relatively well-preserved microstructure of primitive plants, shows various styles of crisscross network, with obvious cell cavities and walls (Fig.3 A1). The cell cavities of provitrinite are usually round, oval and regularly arranged. Under the formation pressure, the cell cavity is often deformed and irregular. The typical in-situ SEM characteristics of provitrinite are high protuberance, dense and uniform texture, dark color, dark brightness, clean and smooth surface, and generally undeveloped pores.

(2) Collinite is one of vitrinite whose original plant cell structure can no longer be seen through strong gel. The color shade of unstructured vitrinite is similar to that of

structural vitrinite, but the protuberance is slightly lower than that of provitrinite. Transverse cracks are often seen inside the collinite, and the protrusion is significantly higher than that of SB, which is an effective sign to distinguish the collinite from the SB (Fig.3 A2).

(3) Vitrodetrinite, was clastic particle of vitrinite that cannot identify the primitive cell structure, formed by mechanical crushing of structural vitrinite and unstructured vitrinite. It is irregular in shape, similar to poorly sorted sedimentary clastic particles, with dark brightness, flat and smooth contact line with minerals, high protuberance, and generally undeveloped pores and fractures (Fig.3 A3-A4).

4.2.2 Inertinite

Inertinite can be further divided into fusinite and semifusinite, sclerotinite, and inertodetrinite (Fig.3 B1-B4).

(1)Fusinite and semifusinite, similar to provitrinite and also derived from plant microstructures, such as roots, stems and bark of plants, are a type of microscopic component with the best-preserved cell structure among inertinite. On the whole, the protrusions of fusinite are higher than that of the structural vitrinite, and the color is slightly lighter than that of the vitrinite, with undeveloped pores and fine stomata distribution on cell walls(Fig.3 B1).

(2)Sclerotinite, darker in color, 70% black, are usually in a regular round shape, with high protrusions, dense edges, no pores, and regular circles of different sizes often developed in the middle (Fig.3 B2).

(3)Inertodetrinite, similar to vitrodetrinite, the clastic particles of inertinite, whose cell structure can no longer be identified under the scanning electron microscope, are all irregular in shape, uniform and dense in texture, smooth and glossy, dark gray in color, slightly lighter than the vitrodetrinite, and slightly higher in brightness than the vitrodetrinite. The contact lines between minerals and inertodetrinite are straight and smooth, with high protrusions, and generally no pores and no cracks are found (Fig.3 B3-B4).

4.2.3 Liptinite

Under the scanning electron microscope, algae can be identified according to the different shapes of the alginite (Fig.3 C1-C3). Mixed with clay-sized mineral particles and others, AOM has an uneven surface (Fig.3 C4). At the early maturity stage, it may be difficult to distinguish between AOM and SB, but SB usually has a uniform surface filling pores.

4.2.4 Secondary organic matter

According to the order of appearance of SB in the evolution stage of organic matter, it can be divided into pre-SB, post-SB and pyrobitumen.

The E2k2 shale SB of Well GX-A is dominated by the former two, and the pyrobitumen increases with the increase of burial(Fig.3 D1-D4). The most notable feature of SB is that it has no specific particle shape, and is often filled in the space composed of inorganic minerals in the form of cement, which is greatly affected by the distribution of surrounding materials. Under SEM, the SB is 40% to 50% black, lighter in color than vitrinite and intertinite, and has lower protrusions, with some even lower than the protrusions of surrounding minerals. The pores of pre- SB and post- SB are not developed (Fig.3 D1-D2). Pyrobitumen, as a type of insoluble organic matter, is the solid residual bitumen after the pyrolysis of crude oil. Thus, a large number of sponge-like or honeycomb-like micro-pores often developed in the pyrobitumen, and the pores are fine and evenly distributed, irregular in shape, developed in network, with the phenomenon of small pores appearing in large pores, which is the main difference between pyrobitumen and other SB.

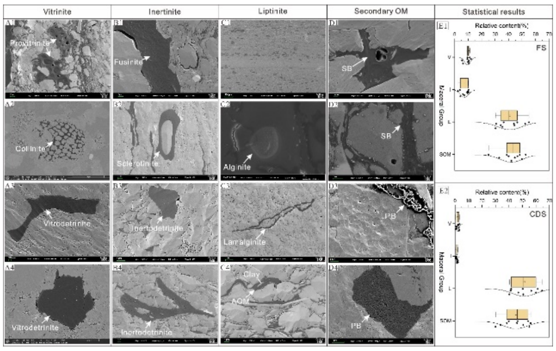

Fig. 3. Photomicrographs of DOM in Paleogene Kong2 Member shales under SEM, including vitrinite (A1-A4), inertinite (B1-B4), liptinite (C1-C4) and secondary OM (D1-D4). AOM= amorphous organic matter; SB= Solid bitumen; PB=pyrobitumen; OM=organic matter.

4.3 Quantitative results of DOM under SEM

Quantitative analysis of E2k2 shale maceral group based under SEM, shows that FS has higher vitrinite (average =9.56%), inertinite (average = 7.41%), and lower liptinite (average =40.91%) and secondary organic matter (average = 42.12%)(Fig.3 E1). However, in calcareous-dolomitic shale (CDS), there are lower vitrinite (average =2.11%), inertinite (average =1.46%), and higher liptinite(average =51.36%) and secondary organic matter (average = 45.07%)(Fig.3 E2).

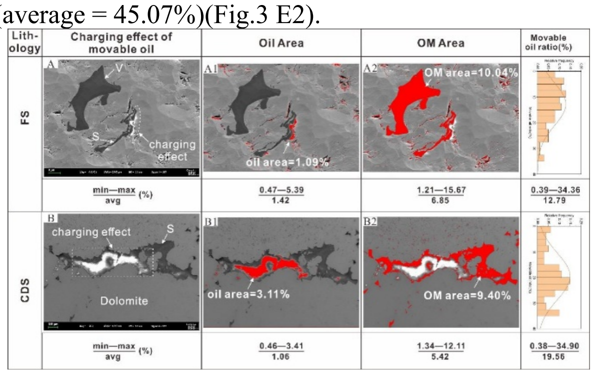

Fig. 4. Distribution of movable oil in FS (A-A2) and CDS (B-B2) under SEM based on charging effect. Image processing was completed in the software of ImageJ. V=vitrinite; S=sporinite; FS= felsic shale; CDS= calc areous- dolomitic shale; OM= organic matter. Movable oil ratio=oil area/OM area(%).

4.4 SEM petrography of Movable oil

The charging effect of SEM can be used to identify the residual movable oil in shale (Fig.4). BSD imaging shows that charging effect on the surface of vitrinite and inertinite was hardly difficult to observe under SEM (Fig.4A), while that of alginite and AOM is the most significant (Fig.4B), which is consistent with the hydrocarbon generation tendency of the macerals. Through ImageJ processing of SEM images with charging effect, it is found that, CDS has a higher distribution of movable oil (average =19.56%) than that of FS (average =12.79%).

5. Discussion

5.1 Controls on organic pore development

The controls of shale organic macerals on organic pores are manifested in three aspects:

(1) Residual pores of primary organic matter: the organic matter in shale includes primary organic matter and secondary organic matter, and organic matter pores can also be divided into primary organic matter pores and secondary organic matter pores (Gao et al., 2021, 2019; Hu et al., 2021; Teng et al., 2022). The pores of primary organic matter are mainly distributed in the provitrinite, fusinite and semifusinite. The common feature of these microscopic components is that the lignocellulosic structure of the original higher plants is well preserved, with visible cell walls and cell cavities. Under the action of vertical compaction or lateral extrusion, the pores of primary organic matter are easily deformed.

(2) Differences in hydrocarbon generation potential. The organic maceral, with sapropel and chitin as the main parent materials, has the greatest potential of hydrocarbon generation, while the vitrinite is mainly the parent materials for gas-generating maceral. The intertinite can generate neither gas nor oil, but it is different from “dead carbon”. As secondary organic matter, SB has the potential of further cracking to produce light oil and natural gas, therefore it has better hydrocarbon generation potential than vitrinite and intertinite.

(3) Catalysis of clay minerals. Organoclay complexes are widespread in shale, and almost all SB therein developed abundant honeycomb pores, especially the SB distributed around illite has significantly more pores than the SB that coexists with other clay minerals. The catalysis of clay minerals can be resolved into two processes (Xie et al., 2021, 2020)- when kerogen is converted into hydrocarbons, organic acids are generated, which promote the conversion of montmorillonite to illite; and the H+ precipitated during the illiteization of clay minerals and H2O can create conditions for the hydrocracking of crude oil, having a certain catalytic effect on the generation of hydrocarbons and developing pores.

5.2 Implications on movable oil and dessert evaluation

Different from marine shale, the organic matter of continental shale is controlled by lake basin area, water properties, climate and structure, especially the influence of climate is significant. In the CDS formed by a relatively arid climate, the input intensity of terrigenous detrital rocks is low, and the content of terrigenous organic matter, such as vitrinite and inertinite, is significantly lower; instead, under strong evaporation conditions, liptinite especially algite, such as telalginite and lamalginite, develop and thus form favorable organic matter types with stronger oil-generating potential. On the contrary, in the FS formed by a relatively humid climate, the input intensity of terrigenous detrital rocks is high, the content of terrigenous organic matter such as vitrinite and inertinite is high, and the total organic matter abundance of shale is significantly higher than that of CDS, but the relatively lower liptinite means that the oil generation potential of FS is not higher than that of CDS(Fig.5）.

Due to the development of high TOC, the FS in Cangdong Sag has always been regarded as the exploration priority zone (Zhao et al., 2019, 2017). However, the above understandings indicate that CDS, despite its weak total hydrocarbon generation, has stronger oil generation potential and a higher proportion of movable oil, which is worthy of attention in sweet spot prediction and exploration.

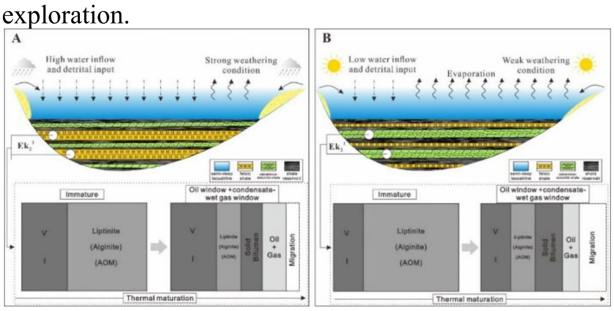

Fig. 5. Evolution model of DOM in typical continental shale oil reservoirs, including FS (A), calcareous-dolomitic shale(B).

6. Conclusions

Taking the E2k2 shale in Cangdong sag of Bohai Bay Basin as an example, the following conclusions are obtained through microscope, fluorescence observation and FE-SEM:

Organic macerals in E2k2 shale are divided into vitrinite, inertinite, liptinite and SB by external morphology, hardness, brightness, color, protrusion, pore and fracture developmentof organic matter, and further subdivided into multiple subcategories. Based on the SEM charging effect of the remaining oil, it is further confirmed that the shale movable oil and oil generation potential developed by lipoid group is the largest, while the shale movable oil and oil generation potential developed by vitrinite group and inertinite group is the worst;

The organic pores include primary pores and secondary pores. The pores of primary organic matter are derived from the biological structure of primary organic matter, and the secondary organic pores are developed during the thermal maturation of oily organic matter. Clay mineral catalysis, difference of hydrocarbon generation potential and residual pores of primary organic matter control the development of organic pores;

Calcareous-dolomitic shale and FS are typical lithology formed in relatively dry and humid climate respectively, and the types of organic macerals are significantly different. Although the former has weak total hydrocarbon generation, it has stronger oil generation potential and is worthy of attention in dessert prediction and exploration.

Acknowledgments

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