Investigation of fluid types in shale oil reservoirs

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Abstract: Lacustrine shale oil resources are essential for the maintenance of energy supply. Fluid types and contents play important roles in estimating resource potential and oil recovery from organic-rich shales. Precise identification of fluid types hosted in shale oil reservoir successions that are characterized by marked lithological heterogeneity from only a single well is a significant challenge. Although previous research has proposed a large number of methods for determining both porosity and fluid saturation, many can only be applied in limited situations, and several have limited accuracy. In this study, an advanced logging technique, Combinable Magnetic Resonance logging (CMR-NG), is used to evaluate fluid types. Two-dimensional Nuclear Magnetic Resonance (2D-NMR) experiments on reservoir rocks subject to different conditions (as received, after being dried at 105℃, and kerosene imbibed) were carried out to define the fluid types and classification criteria. Then, with the corresponding Rock-Eval pyrolysis parameters and various mineral content from X-ray Diffraction, the contribution of organic matter and mineral compositions were investigated. Subsequently, the content of different fluid types is calculated by CMR-NG (Combinable Magnetic Resonance logging viz 2D NMR logging). According to the fluid classification criteria under experimental conditions and the production data, the most favorable model and optimal solution for logging evaluation was selected. Finally, fluid saturations of the Cretaceous Qingshankou Formation in the Gulong Sag were calculated for a single well. Results show that six fluid types (kerogen-bitumen-Group OH, irreducible oil, movable oil, clay-bound water, irreducible water, and movable water) can be recognized through the applied 2D NMR test. The kerogen-bitumen-Group OH was mostly affected by pyrolysis hydrocarbon (S_2) and irreducible oil by soluble hydrocarbon (S_1). However, kerogen-bitumen-Group OH and clay-bound water cannot be detected by CMR-NG due to the effects of underground environmental conditions on the instruments. Strata Q8 and Q9 of the Qing 2 Member of the Cretaceous Qingshankou Formation are the most favorable layers of shale oil. This research provides insights into the factors controlling fluid types and contents; it provides guidance in the exploration and development of unconventional resources, for example for geothermal and carbon capture, utilization, and storage (CCUS) reservoirs.

Key words: Fluid identification; shale oil; 2D NMR tests; CMR-NG; factors controlling

Highlights:

(1) Six types of fluid are identified by 2D NMR experiments; only four of these types are identified by CMR-NG logs.

(2) The content of different fluid types is calculated using T_1-T_2 maps according to the classification criteria.

(3) The kerogen-bitumen-Group OH was mostly affected by pyrolysis hydrocarbon (S_2) and irreducible oil by soluble hydrocarbon (S_1).

1. Introduction

Unconventional resources make up a substantial part of global oil and gas exploration, development and production. Reservoirs in relatively low permeability and low porosity rocks (so-called tight and shale reservoirs) that host shale oil and gas are important targets and assets for energy companies (EIA., 2023; Fang et al., 2019; Li et al., 2019). Existing unconventional reservoirs are mainly distributed in North America, followed by Eastern Europe and Asia (EIA., 2023; Fang et al., 2019). There are more than 100 petroliferous basins all over the world (EIA., 2023; Fang et al., 2019). Of these, the largest shale oil enrichment basin is the West Sibrian Basin in Russia, accounting for 16.5% of the global shale oil resources. The second and third largest are the Permian Basin and Western Gulf Mexico Basin of the United States, respectively. The Junggar and Songliao Basins of China rank 10^{th} and 11^{th}, respectively (Fang et al., 2019). The Gulong shale located in the Gulong Sag of the central depression of the Songliao Basin is the focus of this study due to its favorable resource potential.

The shale oil reservoir of the Qingshankou Formation, in the Gulong Sag, Songliao Basin is characterized by high clay content, diverse mineral composition, pronounced lamination within many beds, and strong lithological heterogeneity and anisotropy. It is important to accurately evaluate the shale oil saturation parameters in reservoir assessment and evaluation. Previous studies have introduced numerous methods with which to calculate resource potential parameters (Ballinas et al., 2023; Li et al., 2023; Zhao et al., 2023). The traditional saturation calculation method is the Archie Formula or advanced Archie Formula (Han et al., 2019; Li et al., 2022; Zhu et al., 2023). However, these methods are not suitable for the Gulong Shale due to the complex pore structure and the presence of micro- and nano-scale pores, for which electrical experiments to determine porosity and saturation are not suitable. In addition, conventional well-log suites – for example, resistivity logs, gamma ray logs, and three porosity logs – are not able to detect the variation of the reservoir space, fluid properties, and lithology clearly. Nuclear Magnetic Resonance (NMR) has developed as a commonly applied technique to evaluate physical properties (porosity, permeability, and pore distribution) qualitatively and quantitatively both in well-log and core experiments (Liu et al., 2022; Ramia and Martin., 2016). For one-dimensional NMR, only transverse relaxation time (T_2) can be detected, but this is insufficient to separate different fluid types, because of the overlapping of different fluid T_2 signals (Zhang et al., 2020; Mukhametdinova et al., 2021). For saturation calculation using the NMR test, centrifugation measurements required to obtain the oil saturation of samples are necessary for the calibration of the NMR logs (Liu et al., 2019). However, centrifugation tests are difficult to conduct in reservoirs characterized by lamination and lamellation fractures. Thus, a new method to estimate the oil content and saturation, and fluid types is urgently required.

Compared with one-dimensional NMR technology, two-dimensional NMR (2D NMR) technology can simultaneously detect and record more parameters, such as transverse relaxation time T_2, longitudinal relaxation time T_1, diffusion coefficient D, and internal magnetic field gradient G. This improves the accuracy of fluid type identification and fluid saturation calculation (Khatibi et al., 2019; Yan et al., 2021). This technique is now widely used in the evaluation of physical properties, oil saturation, and mobility of unconventional oil and gas reservoirs (Qin et al., 2022). Additionally, 2D NMR can also be used to elucidate wettability (Liang et al., 2019). However, to detect the 2D NMR signal characteristics and condition of in-situ preserved rocks, there are very specific requirements for how core samples must be obtained. Moreover, the 2D NMR experiment is costly and the length and quantity of cores that can be measured are limited. To make up for these shortcomings, 2D NMR logs (CMR-NG) are usually applied to measure and record the 2D NMR signal from only a single well.

The aim of this study is present a workflow for the application of 2D NMR as the key technique to determine the saturation and proportion of solid organics, water, and oil behaviors in shales of the Qingshankou Formation, Gulong Sag, Songliao Basin, China. Specific research objectives are as follows: (i) to divide the fluid types in shales and determine the classification criteria according to T_1 and T_2; (ii) to elucidate factors controlling various fluid types; (iii) to evaluate the oil potential of different layers of the Qingshankou Formation using 2D NMR logs.

2. Geological setting

As a well-known petroliferous basin located in northeastern China, the Songliao Basin is present in the Heilongjiang, Jilin, and Liaoning provinces of China; the basin covers are area of ~26×10^4 km^2, with a length of 820 km (north-south) and a width of 350 km (east-west) (Fig

1A). During its evolution, the basin experienced four major structural episodes: upper mantle uplift in the Middle-Late Jurassic; continental rifting from the late Jurassic to Early Cretaceous; rapid subsidence (the so-called depression stage) in the late Cretaceous; and inversion from late Cretaceous to the Quaternary (Ge et al., 2010; Wang et a., 2021). The structure of the Songliao Basin is characterized by seven tectonic zones: the Central Depression, Northern Plunge, Western Slope, Southwestern Uplift, Kailu Depression, Southeastern Uplift, and Northeastern Uplift (Fig 1B). The Central Depression can be further divided into 11 secondary tectonic units and the Gulong Sag is one of these (Fig 1C). The Gulong Sag is bounded by the Honggang Terrace to the northwest, the Qijia Sag to the north, the Changling Sag to the south, and the Daqing Placanticline to the east (Fig 1C). Within the Gulong Sag, three formations accumulated during the episode of active faulting: the Huoshiling, Shahezi, and Yingcheng formations (Ge et al., 2010; Cheng 2019; Wang et al., 2021). Active volcanism occurred during the emplacement of both the Huoshiling and Yingcheng formations (Cheng 2019; Wang et al., 2021). Five formations accumulated during the depression stage: the Kuloudeng, Quantou, Qingshankou, Yaojia, and Nenjiang formations (Ge et al., 2010; Wang et al., 2021; Pang et al., 2023). Of these, the Qingshankou Fm became a focus of hydrocarbon production due to its medium-high thermal maturity of organic matter (Liu et al., 2018, 2019; Liu et al., 2020). A counter map of vitrinite reflectance and the locations of key wells are shown in Fig 1D. The Qingshankou Formation was characterized by semi-deep to deep lacustrine depositional environment and experienced a major transgression (Liu et al., 2019; Liu et al., 2020). The lithofacies are dominated by laminated siliceous shales and laminated clay shales interbedded with siltstones, shelly limestones, and massive dolomite, implying a depositional environment that was subject to several transgressive and regressive cycles (Liu et al., 2019). The Cretaceous Qingshankou Formation is divided into three members, form bottom to top, the first member (K_2qn_1), second member (K_2qn_2), and third member (K_2qn_3). The first member and the lowermost part of the second member have been a focus of hydrocarbon exploration and development because they exhibit high TOC (Liu et al., 2019; Pang et al., 2023). According to the Gamma-Ray log data, the first member and the lowermost part of the second member are further subdivided into nine stratal units (Fig 1E).

3. Samples and Methods

Core, SEM, thin section, and X-ray diffraction (XRD) were used to analyze the petrology characteristics of the reservoir. Rock-Eval pyrolysis analysis was carried out on 16 samples from four key Wells (Well SY1, Well SY2, Well SY3, and Well A34) using the OGE-VI oil and gas evaluation workstation (Table 1). Then, pyrolysis properties including soluble hydrocarbon (S_1), pyrolysis hydrocarbon (S_2), organic carbon dioxide (S_3), residual carbon (S_4), total organic carbon (TOC), hydrogen index (I_H), oxygen index (I_O), total hydrocarbon (S_1+S_2 or Pg), and the maximum temperature of the pyrolysis peak (T_{max}) were obtained. S_1 was tested at 300°C for 3 minutes to provide an indication of the quantity of free petroleum present in the sample. S_2 was measured by increasing the temperature from 300°C to 550°C to provide an indication of the kerogen content present in the sample at the time of analysis. S_3 was determined as the yield of carbon dioxide during the pyrolysis of kerogen. S_4 was tested after 600°C. T_{max} was determined as the maximum temperature corresponding to the highest peak of S_2 pyrolysis peak. I_H and I_O are expressed as S_2/TOC×100 and S_3/TOC×100, respectively.

A total of nine samples from two key wells (Well SY2 and Well SY3) were used for the 2D NMR test. Compared with the low magnetic field, when the hydrogen protons are tested under a high external magnetic field, the fluid signal in the T_1-T_2 map will shift to the left. Under a higher-frequency magnetic field, the magnetic field gradient increases, resulting in a shorter transverse relaxation time, a longer longitudinal relaxation time, and then a larger value of T_1/T_2. Due to the different properties of each fluid type – notably diffusion coefficient, viscosity, and magnetic susceptibility at the solid-liquid interface – the magnetic field strength has different effects on the relaxation time of each type. Taking oil and water as an example, when the magnetic field frequency increases, the transverse relaxation time of oil becomes shorter and more than the water signal, and the longitudinal relaxation time becomes longer and greater than that of the water signal (Qin et al., 2021, 2022). For unconventional oil and gas reservoir analysis, 21 MHz is mostly used in the NMR experiment. Under this circumstance, high-quality echo data can be obtained (Li et al., 2018; Liu et al., 2022). Therefore, the oil and water signals can be separated more specifically. However, experimental data obtained from NMR testing under a 21 MHz magnetic field cannot be directly used to calibrate the NMR logs, because the NMR log detects T_1 and T_2 signals under a lower magnetic field, commonly of 2 MHz. For improved NMR log calibration, a 2D NMR test under a low magnetic field (2 MHz) is carried out in this study. It should be noted that there is a problem of overlapping of different components attributed to the low magnetic field intensity. The identification accuracy of different fluid types is not sufficient. The samples are tested under three different conditions: (i) as received (AS), (ii) after being dried at 105 °C (D), and (iii) kerosene imbibed (KI). The laboratory conditions were at normal temperature and atmospheric pressure. The samples were dried, then wrapped with plastic to prevent moisture in the air from affecting the measurement results, and then placed at normal temperature for measurement.

Schlumberger's Combinable Magnetic Resonance logging tool named CMR-MagniPHI (CMR-NG), was used to detect the Multiple CPMG echo signals with different waiting times (T_W) in the borehole. Then, multi-echo series joint inversion is processed to obtain a T_1-T_2 map. The original measurement data of CMR-NG mainly includes six sets of orthogonal echo channel data. Echo data of each group is stored in order from long to short length of Tw. Therefore, echo data of different T_W can be extracted according to the number of echoes. The echo data processing of multi-T_W CPMG signals mostly adopts the echo series inversion method (Guo et al., 2019; Yarman and Mitchell, 2019), which is processed by Techlog2019 advanced interpretation module. CMR-NG Analysis consists of three steps: Cluster Estimator, Volumes, and Finisher (Fig. 2).

The resonant frequency of the above 2D NMR experimental conditions is 2 MHz, which is consistent with the magnetic field of the CMR-NG instrument. Therefore, the experimental data can be used to calibrate the CMR-NG logging. Thus, the standard of fluid type classification under experimental conditions can be applied to logging. However, in the actual logging process, due to the requirements of the instrument's data acquisition method and aging, it is difficult to achieve the same effect as that of experimental measurement. Moreover, the experimental measurement is not entirely representative of the natural state of underground rock nuclear magnetic response characteristics, so there will be some deviation between these two.

In this study, first, the classification criteria of different fluid types were established according to the energy cluster variation of T_1-T_2 maps under three conditions (as received, after being dried at 105 °C, and kerosene imbibed). Then the content of various fluid types can be calculated through T_1 and T_2 data obtained by 2 MHz 2D NMR experiment. Subsequently, combing the mineral composition, lithology, and pyrolysis parameters, the controlling factors of different fluids were discussed. Consequently, logging evaluation of oil saturation in the Qingshankou Formation in a single well was unraveled using CMR-NG (2 MHz 2D NMR log).

4. Results

4.1. Rock characteristics

4.1.1. Mineral composition and pore space

The reservoir is highly heterogeneous with complex sedimentary components. According to the observation of the SEM, the dominant minerals are quartz, feldspar (albite and orthoclase), clay (mainly illite, mixed layer illite/smectite, and chlorite), ankerite, calcite, and pyrite (Fig 3). Compared with other shale oil and gas reservoirs, for example the Barnett shale in the Fort Worth Basin in the United States, the content of clay minerals is higher and the content of carbonates is lower in the Qingshankou Formation. The SEM images show that the illite is characterized by layered structure, contributing to the development lamellation fractures and beddings under the compaction in the burial history.

As an unconventional oil reservoir, the succession studied here is characterized by relatively low porosity with an average value of 7.7% and low permeability (horizontal permeability lower than 0.61×10^{-3} μm^2 and vertical permeability lower than 0.056×10^{-3} μm^2 (Gao et al., 2022). The SEM images indicate that the pore types of the Qingshankou Formation can be divided into organic pores and inorganic pores. The inorganic pores can be further classified into dissolution pores and intercrystalline pores (Fig 4). Additionally, natural fractures, and microfractures can be observed from cores and thin sections. The radius of pores mainly ranges from 2 nm to 50 nm (mesopores) (Liu et al., 2018; Bai et al., 2022). Lamellation fractures played an important role in oil migration and accumulation and provided the majority of the reservoir space (Zhang et al., 2021; He et al., 2022; Pang et al., 2023).

4.1.2. Geochemical Characteristics

Parameters commonly used to evaluate the abundance of organic matter include total organic carbon (TOC), chloroform asphalt "A" and total hydrocarbon (HC), as well as hydrocarbon generation potential (Pg) obtained from rock pyrolysis experiment. Total organic carbon (TOC) is an important index to evaluate the abundance of organic matter. It is worth noting that in the process of hydrocarbon generation by the evolution of organic matter, with the increase of maturity, part of the oil and gas migrated to the reservoir, causing part of the carbon to discharge from the source rock. Therefore, it is important to restore the original total organic carbon (both residual and discharged organic carbon) prior to an assessment of the source rock. No migration is recognized in the Qingshankou Formation of the Gulong Sag (Feng et al., 2020). Shales with TOC contents of less than 0.5%, 0.5%~1%, 1%~2%, and greater than 2% represent poor, fair, good, and excellent petroleum potential, respectively (Peters and Cassa, 1994; Hu et al., 2021). Meanwhile, Pg with values of less than 0.2 mg HC/g rock, 0.2 mg HC/g rock ~ 2 mg HC/g rock, 2 mg HC/g rock ~ 6 mg HC/g rock, and greater than 6 mg HC/g rock represent poor, fair, good, and excellent source rocks, respectively. The organic carbon content ranges from 0.75% to 7.98%, with an average value of 3.46%. The cross-plot of the TOC and Pg shows that most core data is located in the favorable area, indicating the shale of the Qingshankou Formation is a high-quality source rock (Fig 5).

According to the pyrolysis analysis of the rocks, the parameters of hydrogen index (I_H), oxygen index (I_O), and maximum pyrolysis peak (T_{max}) are used to determine that the organic matter types of Qingshankou Formation in the Gulong Sag. The results show that the kerogen types are mainly Type I and Type II_1, indicating that the study area is dominated by oil-prone kerogen with an oil generation (Fig 6). Generally, Type I and Type II are attributed to flourishing low aquatic algae (Liang et al., 2015; Martins et al., 2020). Organic matter is mainly derived from layered alginate and telalginite, which is characterized by high conversion rate of hydrocarbon generation and a large area shrinkage rate. In the process of hydrocarbon generation and expulsion by thermal evolution of organic matter, a large number of nano-sized elongated pores (fractures) appeared along layers, which are defined as lamellation fractures (Feng et al., 2021). This type of reservoir space contributed the most storage space and was good as migration pathway.

The reflectivity of vitrinite (Ro) and T_{max} are commonly used to evaluate thermal maturity. During the thermal evolution of organic matter, a series of reactions such as thermal desorption of alkanes and fusion of aromatic ring occurred, and all that matters is this reaction is irreversible. The pyrolysis process of kerogen has a good consistency with the evolution of vitrinite. With the increase of thermal evolution degree, the reflectivity of vitrinite increases and the transmittance decreases. Therefore, R_o can be used to evaluate the maturity of organic matter. The pyrolysis experimental results show that the Ro values of the Qingshankou

Formation range from 0.7 to 1.7, with an average value of 1.3. The T_{max} distribution ranges from 413℃ to 453℃ with a mean of 440℃ (Table 1), indicating that the shales of the Qingshankou Formation are in medium to high stages of thermal maturity, which is conducive to shale oil enrichment and with broad exploration and development prospects.

4.1.3. Lithology

The lithology of the Qingshankou Formation in the Gulong Sag is identified by polarizing microscope and core observation. It is dominated by shale, which mainly contains felsic shale and clayed shale, shelly limestone and dolomite, and a little siltstone as well (Fig 7). Among them, the structure of shelly limestone and dolomite are mostly massive, and usually interlayered with other rocks. Clayed shale and felsic shale account for more than 80%, which are favorable lithologies for shale oil formation and enrichment (Liu et al., 2019; Gao et al., 2022; Pang et al., 2023b). Thin section observation displays that four types of minerals can be identified including felsic, clay, carbonate, mixed, and organic matter (Fig 7A-D). Among them, the content of clay minerals is the highest, followed by felsic minerals. The content of mixed and organic matter minerals is the lowest.

The felsic shale is distributed in the whole Qingshankou Formation, accounting for 20%. Several lake level decreases-increase circles occurred during the deposition of K_2qn_1 resulting in deposition of shale interbedded with fine-grained siltstones (Liu et al., 2019). Conversely, the supply of terrigenous clastic rocks increased. Therefore, the content of felsic minerals increased, and felsic shale increased as well. The core observation shows that this type of rock is grayish black with obvious laminae. Affected by liquefaction, some of the laminae and lamellation are disturbed (Fig 7F, Fig 7G). Under the Leica polarizing microscope, most of the detrital grains are grayish brown, quartz and anorthose, which accounts for 70%, and subangular and of high sphericity (Fig 7A). Calcite and ankerite minerals are rarely observed, and are mostly the products of cementation and metasomatism in the late evolution stage. It can be seen that pyrite in the form of particles or aggregates is distributed among the particles and are mostly associated with clay minerals (Fig 7A).

Clay-prone shale is widely distributed in the study area, accounting for ~65% of the studied succession. The core is grayish black to dark, with laminae and lamellation developed. The boundary between some beddings or laminae is not clear, and it can be seen that a large amount of pyrite is distributed in star-point shape, with the diameter of particles up to 3 mm. Some of them are connected to the laminae laterally, and there are many small calcite veins distributed along the laminae (Fig 7E). Under the parallel polarized light, the clay-prone shale is characterized by brown, brown-yellow, and black-brown color. The clastic particles are mainly quartz, with a subangular to circular shape, with a particle size less than 0.05 mm, and are mostly distributed among clay minerals in bands (Fig 7E). The content of carbonate minerals is small, mainly calcite and ankerite. The calcite is filled among the particles as cement, and a small amount of them is distributed along microfractures and lamellation fractures. Ankerite is of good crystal form and scattered among clay minerals in a star-point pattern. The pyrite is mainly granular and aggregates, and some pyrite appeared along the bedding, showing a long black strip under parallel polarized light (Fig 7B, Fig 7C).

The shelly limestone facies is less common, accounting for 7% of the studied succession. From the core observation, it is mostly grayish white, with obvious layers of shell debris (Fig

7H, Fig 7I). The shell debris mostly comprises fragments of lamellobranchiate organisms, as evident under the optical microscope; biologenic cavities (moulds) are mostly filled with calcite, organic matter or bitumen during diagenesis. Quartz particles are angular to subcircular, distributed unevenly in the matrix, with varying particle sizes up to 0.2 mm (Fig 7D).

The distribution of dolomite in the study area is similar to that of shelly limestone, and it is apparently less abundant in the Qingshankou Formation, accounting for 8%. The core is gray and characterized by massive structure, with no visible layers (Fig 7J).

In addition, previous studies have pointed out that in the lacustrine depositional environment of the Qingshankou Formation, various types of gravity flow deposits, including clastic flow and turbidity flow deposits, can be observed (Fu et al., 2014; Du et al., 2015; Liu et al., 2019). However, they are not widespread and mostly occur in the western slope area of the basin (Du et al., 2015). Gravity flow deposits can also be seen in the Qingshankou Formation in the Gulong Sag (Fu et al., 2014; Du et al., 2015) (Fig 7K). The lithology of these deposits is mainly siltstone, silty mudstone, and mudstone. Controlling factors for gravity flow deposition are complex. Previous studies showed that there was intense volcanic activity during the early stage (Huoshiling and Yingcheng formations) (Cheng 2019; Wang et al., 2021), and the volcanic eruption processes likely induced earthquakes (Cheng et al., 2019). These earthquakes may have acted as a triggering mechanism for gravity flows in this region (Liu et al., 2005). These gravity flows carried a large amount of terrigenous debris into the depositional center of the lake. The sedimentary sand bodies of gravity flow origin are thick (~1 to 2 m) with high reservoir quality (Du et al., 2015; Tang, 2015; Zou et al., 2023). Therefore, these deposits types form high-quality reservoir rocks through which oil and gas preferentially migrated, charged, and were preserved after hydrocarbon generation and expulsion from neighboring source rocks. Reservoirs formed of gravity flow deposits are commonly act as sweet spots in unconventional oil and gas exploration (Zou et al., 2023). The turbidity is commonly characterized by low-density and mainly fine silt and clay sediments. The rocks are mainly black shale, dark gray siltstone, and gray fine sandstone, and parallel bedding, horizontal bedding, and cross-bedding are easily observed. The speed of this type of flow is relatively slow and the duration is widely attributed to the weak hydrodynamic energy, and it mostly appears at the tail of a gravity flow activity, forming when rivers flood into lakes (Zhang et al., 2005; Li et al., 2020). Low-density turbidity has typical sedimentary structures and sequences, viz the Bouma sequence (Li et al., 2020; Zou et al., 2023). In the Qingshankou Formation of the Gulong Sag, a prominent bed preserved with a Bouma-type stratal expression with a deposition thickness of about one meter was found in Well 17 (Fig 7K). The core photo shows that Section A is mainly composed of sandstone, but no gravel. An obvious erosion interface can be observed at the bottom of sandstone. In Section B, the dominant lithology is siltstone with parallel bedding. Section C is characterized by wavy bedding. Horizontal bedding occurs in sandstone in Section D. On the top of this sequence named Section E, the main lithology is a massive (i.e. internally structureless) mudstone.

4.2. Fluid types

4.2.1. Classification of fluids types via 2D NMR experiment

The 2D NMR experimental results showed that the six fluid types were identified corresponding to six segments (A, B, C, D, E, and F) divided in the T_1-T_2 map. Region A represented bitumen characterized by T_2 value being less than 1 ms and T_1/T_2 value greater than 10. Region B displayed bound oil in micropores, mainly in organic pores, with T_2 ranging from 1ms to 10 ms and T_1/T_2 greater than 3. The movable oil in region C was mainly concentrated in macropores with T_2 greater than 3 and T_1/T_2 greater than 3. Region D was clay-bound water with T_2 value less than 1ms and T_1/T_2 less than 10. Region E represented immovable water, which was mainly concentrated in small pores, with T_2 ranging from 1 ms and 10 ms and T_1/T_2 being less than 3. Region F corresponded with movable water, mainly enriched in large inorganic pores, with T_2 values ranging from 10 ms ~ 100 ms and T_1/T_2 less than 3 (Fig 8, Table 2). Kerogen as an organic matter is characterized by being insoluble in organic solvents. As a mixture of various large molecular organic compounds, the composition of kerogen is complex and not fixed. Bitumen is another type of organic matter that is insoluble in water but soluble in organic solvents. Kerogen cannot be recognized at the low magnetic field (Fleury et al., 2016; Silletta et al., 2022). Therefore, region A is mainly bitumen or solid-like organic matter. Under experimental conditions, it is difficult to completely separate clay structural water, bound water and bound oil in micropores, and oil and water in larger pores (Li et al., 2018; Khatibi et al., 2019; Zhang et al., 2020). Therefore, regions B, C, E, and F exhibited overlap. The results of 2D NMR test under the condition of after being dried at 105°C showed that the signal intensity in the regions B, C, E, and F was weakened (Fig 8). The signal in the regions C and F representing movable fluid almost completely disappeared. Although the signal in regions B and E was weakened, the presence of fluid can still be detected (Fig 8). The reduced signal may be the loss of water in the small pores in the inorganic pores. Some reduced signals are attributed to oil loss because high temperatures would reduce the viscosity and increase the fluidity of oil, which resulted in the discharge of the oil along the connected pores and throats. On the condition of kerosene imbibed, the signal in regions B, C, E, and F of the T_1-T_2 map was significantly enhanced (Fig 8). The kerosene firstly injected into the large pores and throats and then entered the micropores and small pores. Generally, kerosene cannot flow into intercrystalline pores. However, due to the adsorption of clay minerals, part of the kerosene was attached to the surface of mineral particles or between layers (Zhang et al., 2020; Xi et al., 2023). Therefore, the signal in the region B was enhanced. Obvious fractures (high-angle fractures and lamellation fractures) can be observed in sample C3 (Fig 8). The T_1-T_2 map of C3 on the condition of after being dried at 105°C exhibited that the signal intensity in regions B, C, and E was significantly weakened, indicating that the development of fractures greatly improves the petrophysical properties of reservoirs and enhances the connectivity between pores and throats, especially for reservoirs without large pores (Fig 8). The occurrence of fractures has a significant positive effect on fluid migration and discharge in small pores.

4.2.2. Different fluid proportion calculation

According to the classification criteria of six fluid types, the incremental porosity data under T_1 and T_2 obtained by two-dimensional nuclear magnetic resonance experiments were used to determine the proportion of different fluid types. The results showed that on the condition of AS, the contents of A (bitumen and solid-like organic matter), B (bound oil), C (movable oil), D (clay-bound water), E (irreducible water), and F (movable water) ranged from 0.09 ~ 0.48, 0.02 ~ 0.26, 0.01 ~ 0.08, 0.23 ~ 0.85, 0.04 ~ 0.11, and 0.00 ~ 0.01, with averages of 0.21, 0.12, 0.03, 0.59, 0.06, and 0.001, respectively (Table 3). On the state of being dried at

105°C, the signal percentages A, B, C, D, E, and F variated from that of on the condition of AS, with contents ranging from 0.19 ~ 0.54, 0.04 ~ 0.24, 0.01 ~ 0.08, 0.19 ~ 0.70, 0.01 ~ 0.10, and 0.00 ~ 0.01, with averages of 0.35, 0.11, 0.04, 0.46, 0.05, 0.001, respectively (Table 3). On the condition of kerosene imbibed, the contents of A, B, C, D, E, and F ranged from 0.06 ~ 0.50, 0.05 ~ 0.41, 0.02 ~ 0.09, 0.26 ~ 0.70, 0.01 ~ 0.30, and 0.00 ~ 0.09, with averages of 0.23, 0.15, 0.05, 0.40, 0.12, 0.05, respectively (Table 3). The little change of content of A under different conditions indicated that the properties of bitumen and solid-like organic matter were natural and these environments had no effect on them. This fluid type (bitumen and solid-like organic matter) was mainly enriched in micro and nano pores such as organic matter pores. Notably, higher temperature (more than 110°C) could change the structure and molecular composition of clay minerals (Ma et al., 2020). The content of bound water (both clay-bound water and irreducible water) ranged from 0.25 ~ 0.88 (with an average of 0.65), from 0.21 ~ 0.72 (with an average of 0.50), and from 0.32 ~ 0.72 (with an average of 0.52), under the conditions of AS, D, and KI, respectively (Table 3). The content variation indicated that only a little irreducible water was volatile during heating. The content of movable water and movable oil was almost unchanged from conditions AS to D. On the condition of KI, the content of these fluid types variated a lot implying there were large pores and connectivity pores and throats in these rocks. Movable oil filled these reservoir spaces underground. After coring, movable oil was lost along the connectivity pore structure. With the injection of kerosene, fluid was reaccumulated in these spaces. Then, signals could be detected in these regions (Fig 8, Table 3).

4.2.3. Fluid identification via 2D NMR logs

There are five methods to divide energy clusters, including Expert Mode, Recommended, More Cluster, Manual Parameter, and Difficult Cases. Each method provides several interpretation results. Taking Expert Mode for example, five types of different energy group numbers were divided, including Clustering No.6 which divides six regions, Clustering No.2 in which two regions are classified, Clustering No.3 which divides the signal into three regions, Clustering No.4 that divides four regions, and Clustering No.5 that divides five regions (Fig 9). Of all of these, Clustering No.5, in which the method of dividing six energy clusters is the best (Fig 9). Similarly, the Recommended model has 9 interpretation methods. The More Cluster model provides 13 interpretation methods (Table 4). The Manual Parameter explains 5 classification schemes. The Difficult Cases method has 7 interpretation methods (Table 4).

According to the classification criteria of six fluid types of experimental conditions, the fluid type of each energy cluster in T_1-T_2 maps derived from CMR-NG logging was defined (Fig 10). Compared with the experimental data, the value ranges of T_1 and T_2 recorded in the CMR-NG logging were smaller (Fig. 10). The component OH Groups with short relaxation time cannot be detected, nor can solid-like organic matter and kerogen be obtained. The number of energy clusters divided by the optimal classification of each model is not the same, and the signal peak position of each energy cluster is also different. Since the boundary of energy clusters is not exactly consistent with the boundary of fluid types division, it might bias different components in CMR-NG logging. For example, region 5 of the T_1-T_2 map in Fig 10A is defined as bound oil, but in fact, it also contains bound water. Therefore, there is a certain error in the interpretation of the results of the well log data.

5. Discussion

5.1. T_1-T_2 maps of various lithologies

The two-dimensional NMR spectra of different lithology samples on three conditions (AS, D, and KI) were measured. The results show that the T_1-T_2 maps of different lithologies characterized by different mineral compositions are different.

The C7 sample is a shelly limestone that contains calcite, felsic, and clay minerals in the value of 54%, 34%, and 4% respectively with low total organic carbon and sulfur (Fig 11A_1, A_2, Table 5). On the condition of the original state (as received), the T_1-T_2 map showed that the main fluid types were D (clay structural water) and E (bound water and immovable water) in small pores (Fig 11A_3) (Li et al., 2018; Khatibi et al., 2019). The signal intensity of these two regions was not high. The main peak signal in region D was with the T_2 value of 0.4 ms and T_1/T_2 value of 2 (Fig 11A_3). The main peak signal in region E was located at T_2 value of 2 ms and T_1/T_2 of 3 (Fig 11A_3). After being dried, the signals in both regions were weakened (Fig 11A_4). After the samples were saturated with kerosene at normal temperature, the signals in regions B and C were significantly enhanced, which may be attributed to the development of lamellaion fractures resulting in the injection of kerosene into the rock along the fractures (Fig 11A_1, A_5).

Sample D2 was defined as dolomite consisting of ankerite, clay minerals, and felsic with the values of 24%, 25%, and 40% respectively (Fig 11B_1, B_2, Table 5). As received state, the T_1-T_2 displayed that the fluid types of multi-porous media were mainly A, B, D, and E, that were, bitumen, clay bound water, structural water, and immovable oil (Fig 11B_3). The values of T_2 and T_1/T_2 of the main peak signal were 0.2 ms and 40 in region A (Fig 11B$_3$). The high ratio of longitudinal to transverse relaxation time indicated that the content of macromolecular compounds in solid organic matter increased. The main peak signal of region D is located at T_2 of 0.2ms and T_1/T_2 of 3 (Fig 11B$_3$). The signal in this region is significantly weaker than that in other regions. The signal in region B was concentrated at T_2 value of 2 ms with the value T_1/T_2 of 7, indicating mainly bound oil in micropores and small pores. On the condition after being dried at 105°C, the signal in D region was weakened because of some water evaporating, whereas the signal in regions B and E had no obvious change (Fig 11B$_4$). On the condition of kerosene imbibed, the signal in region C was enhanced after the kerosene first entering the larger connecting pores (Fig 11B$_5$).

Sample D3 mainly contained clay minerals, felsic, and calcite felsic with the value of 45%, 47%, and 4%, respectively, which was a clayed shale (Fig 11C$_1$, C$_2$, Table 5). As received state, T_1-T_2 map, the main peak signal in region A was located at T_2 value of 0.2 ms and the ratio of T_1 to T_2 being 30 (Fig 11C$_3$). The main peak signal in region D was at T_2 value of 0.2 ms and T_1/T2 of 3. The signals in regions B and E cannot be separated accurately, which was concentrated in the T_2 ranging from 1 ms ~ 10 ms and T_1/T_2 ranging from 2 ~ 10 (Fig 11C$_3$). The dominant fluid type of regions B and E was bound oil (irreducible oil). After being dried, the signal in regions A, B and E of the T_1-T_2 map was weakened (Fig 11C$_4$). The decreased signal intensity in region A may be attributed to the rising temperature. Higher temperature contributes to the composition changes of organic matter, the decreasing viscosity of the polymer and the macromolecular compound. All of these reactions can result in a decrease in T_1. After the kerosene was imbibed, a new signal in region C appeared, indicating that there were a small number of large inorganic pores in the rock (Fig 11C_5). Movable oil stored in these pores before was lost during the process of drilling coring. Therefore, this component cannot be detected on the condition of AS.

The main mineral composition in Sample C1 was clay, felsic, and shale with the value of 40%, 54%, and 3%, respectively (Fig 11D_1, D_2, Table 5). This sample was defined as felsic shale. The signal intensity in region A was low, whereas the signal in region D was strong with the main peak located at the T_2 value of 0.2 ms and T_1/T_2 value of 3 (Fig 11D_3). The main fluid type of this sample was clay-bound water. After being dried at 105℃, the signal intensity in region D decreased significantly, and the weak signal in region E completely disappeared (Fig 11D_4). On the condition of kerosene imbibed, the signals in all six regions are significantly enhanced, which is attributed to the adsorption of clay minerals (Fig 11D_5). In the process of kerosene injection, kerosene is absorbed by clay minerals and then attached to the surface of particles and interlayers (Zhang et al., 2020; Xi et al., 2023). Therefore, signals can be detected in different pore sizes.

The response characteristics of the above four samples representing four types of lithologies are different on T_1-T_2 maps. On the condition of AS, compared with the other three types, the shelly limestone (sample C7) displayed almost no signal in regions A and B with T_2 less than 1 ms (Fig 11A_3). However, the clay-prone shale (D2), dolomite (D3) and felsic shale (C1) showed stronger signals in these regions, indicating more organic carbon hosted in shales (Fig 11B_3, C_3, D_3). Compared with clay-prone shale, the T_1-T_2 map of felsic shale sample showed the signal intensity of region A (solid-like organic matter) was significantly weakened, indicating that organic matter in felsic shale has a high degree of transformation or the oil mainly came from near-source oil rather than in-situ (Fig 11A, D) (Liu et al., 2018; Gao et al., 2022; He et al., 2022). However, the hydrocarbons in clay-prone shale were mainly in-situ oil and gas (oil generation and storage in the same area). After the saturation of kerosene, the signals in regions B, C, E, and F in felsic shale and shelly limestone were significantly strengthened (Fig 11A$_5$, D$_5$). This was mainly because the development of lamellation fractures greatly improved the petrophysical properties and enhanced the connectivity of pores and throats of the reservoir (Fig 11A, D).

5.2. Contribution of organic matter and mineral composition

To investigate the factors controlling and accumulation status of multi-porous media fluid types, relationships between the content of each fluid type and pyrolysis parameters and mineral components content were established. Correlation coefficients square (R^2), defined for NMR signal of native-state samples (as received) from regions A ~ F and TOC, S_1, S_2, Pg, CO_2, S_3, and S_4, were summarized. For further analysis, the threshold of R was defined as 0.7 (Mukhametdinova et al., 2021). The correlation between TOC content and hydrocarbon generation potential is not always positive. Some source rocks contain high TOC content but show poor hydrocarbon generation potential. Because TOC is mostly provided by ineffective carbon and these carbon compounds hardly contribute to hydrocarbon generation in these rocks (Hu et al., 2021). The cross plot of TOC and rock-eval pyrolysis parameters showed that the TOC content of Qingshankou Formation in the Gulong Sag was positively correlated with the potential of hydrocarbon generation, but was relatively weakly correlated with the content of soluble hydrocarbons (free hydrocarbons), and had nothing to do with residual hydrocarbons

(Fig 12).

As received (AR): the most significant correlations are for region A (bitumen, kerogen, and group OH) versus TOC, S_2, and Pg (Hydrocarbon generation potential) regions; region B (bound oil) versus TOC, S_1, S_2, and Pg (Table 6, Fig 13A, 13B). According to the value of R^2, component A is controlled by S_2 and B is mostly affected by S_1. A strong positive correlation between region D (irreducible water) and TOC, S_1, S_2, and Pg is displayed, indicating that when organic matter or oil and gas are enriched in the rocks, the pores are mostly enriched with hydrocarbon components.

After being dried at 105 °C (D): the value of R^2 is lower than that of the original state, implying a weaker correlation between pyrolysis parameters and regions A, B, and D (Table 7, Fig 13C, 13D). Region A (bitumen, kerogen, and group OH) shows a strong positive correlation with S_2, followed by Pg, and then TOC content. There is a strong relationship between Region B (bound oil) and S_1. Similar to the original state, there is a good negative correlation between region D and the pyrolysis parameters (Table 7).

Kerosene imbibed (KI): the correlation between different fluid types and geochemical parameters is very weak. Only region A (bitumen, kerogen, and group OH) has a good positive correlation with S_2, Pg, and TOC (Table 8). No matter what condition it is, region A shows a good relationship with S_2 (Tables 6-8). This may be explained by the stable properties of component A. At the dried state, some water in components B (bound oil) and E (irreducible water) is lost due to high temperature. Notably, water signals can be captured at the border of regions B and E. At the saturated kerosene state, kerosene and hydrocarbon enriched in rocks will dissolve together due to their similar chemical properties (the same polarization), as well as the absorption of clay minerals and organic on kerosene (Zhang et al., 2020; Xi et al., 2023).

Although the principle of NMR experiment is the interaction between the spin nuclei and the external magnetic field and the rock matrix has little influence (Lai et al., 2018; Khatibi et al., 2019). Previous studies have found that some mineral components such as clay minerals and quartz may affect the results of nuclear magnetic measurement (Hu et al., 2021; Mukhametdinova et al., 2021; Wu et al., 2021). In addition, Mukhametdinova's study shows that clay mineral content has a strong correlation with structural water at dried state. At water-saturated state, quartz content has a good correlation with bitumen, bound oil and mobile oil and quartz content has a good correlation with macropores and fluid enriched in macropores (Mukhametdinova et al., 2021). The main mineral components in this study are quartz, feldspar, and clay minerals. For better analysis of the factors controlling of different fluid types, relationships between various mineral contents and different fluid contents are established. Table 9 shows the value of R^2 between different fluid types and rock mineral compositions under three states. The results show that there is no obvious relationship between fluid types and the content of various minerals.

5.3. Distribution of fluid saturation in a single well

Among these five methods, Expert Mode and Manual Parameter models had completely consistent interpretation results (Fig 10). Five methods were provided and the optimal solutions were the one that divided the signal into six regions. The peak signals in each region were the same as well. The Recommended model and the Difficult case model have the same explanatory conclusions, but their explanatory processes are slightly different. The Recommended model provides 9 methods, among which the scheme divided the signal into 8 regions is the best. The Difficult case model provides 7 methods and the method divided into 8 regions is the most reasonable. Although the favorable method is to classify the signal into 8 regions in both models, the parameter values of each region are slightly different. The interpretation of the results of the More Cluster model is quite different from that of the other four models. As more regions are divided, more information can be extracted. Fig 9H showed that signal appeared in a long relaxation time (larger than 10 ms) indicating movable fluid components (movable oil and water). Due to the lack of oil saturation data measured by experiment, oil tests and production data were used to determine and validate the optimal model. The oil test results showed that the output of well GY7 was low (Fig 14). The calculated result of the Expert Mode model showed the closest interpretation to the oil test. Therefore, the Expert Mode model was selected to calculate the fluid saturation in the subsequent evaluation process.

Logging evaluation of fluid saturation can be identified by Expert Mode model. It should be noted that bound oil, bound water, mobile oil, and mobile water cannot be completely separated due to the limitations of experimental conditions and accuracy and the complexity of fluids composition. The T_1 and T_2 responses of different fluids have a certain overlap. Therefore, there are some deviations in the calculation results of fluid saturation using CMR-NG logging calibrated by experiment. Fig 15 shows the calculation results that bound oil and bound water are the dominant fluids of the Qingshankou Formation in GY 7. The frequency distribution histograms of bound oil and bound water saturation in each stratum from Q1 to Q9 are shown in Fig 15. The variation of bound oil saturation and bound water saturation in six strata (Q1 ~ Q5 and Q7) is slight. The bound oil saturation ranges from 30% to 60%, and the bound water saturation ranges from 40% to 70%. On the contrary, in strata Q6, Q8, and Q9, water and oil saturation change quickly. The average bound water saturation is about 46%, which is slightly higher than the bound oil saturation of about 54%.

6. Summary and conclusions

The first member and lowermost part of the second member of the Cretaceous Qingshankou Formation, which is characterized by high TOC content with an average value of 3.46%, is present within the Gulong Sag, Songliao Basin located in northeastern China. Parameters (I_H, I_O, T_{max}, and R_o) obtained from Rock-Eval pyrolysis analyses illustrate the presence of kerogen of types I and II, implying that the source rocks are in the main stage of oil generation.

Five types of lithologies were identified. The dominant lithologies are clay-prone shales and felsic shales, which together account for 80%. With the development of algae and burial history evolution (compaction and cementation), the shale in the Qingshankou Formation of the Gulong Sag was featured with abundant laminae (beddings) and lamellation fractures. The main reservoir spaces were organic matter pores, intercrystalline pores, intraparticle dissolution pores, and lamellation fractures.

According to the value of T_2 and the ratio of T_1 to T_2 obtained from 2D NMR experiment, six fluid types were classified, including kerogen-bitumen-Group OH, irreducible oil, movable oil, clay-bound water, irreducible water, and movable water. Due to the resolution limitation of CMR-NG, the signal of kerogen-bitumen-Group OH and clay-bound water cannot be detected, therefore, only four fluid types can be identified. Among the five models of fluid interpretation conducted by *Techlog 2019*, Clustering No. 6 in Expert Mode was proposed as being the most favorable method for logging evaluation of fluid types. The contents of bitumen and irreducible oil are controlled by S_2 and S_1 respectively, with no response to the mineral composition. The main fluid was irreducible oil and water in the Qingshankou Formation in GY 7. Strata Q8 and Q9 were selected as the highly prospective shale oil reservoirs.

Acknowledgments

This work is financially supported by the National Natural Science Foundation of China (Grant No. 42002133), 2022 AAPG Foundation Grants-in-Aid Program, and Science Foundation of China University of Petroleum, Beijing (No.2462024XKBH009). The authors would like to express their sincere thanks to the PetroChina Daqing Company and PetroChina Research Institute of Petroleum Exploration and Development for their assistance in providing the data, and for their technical input to this work. My joint doctoral studies were funded by China Scholarship Council for one year. I am profoundly indebted to this. We are also grateful to the editor and reviewers for providing excellent advice on the manuscript.

Declaration of interests

No conflict of interest exists in the submission of this manuscript, and the manuscript is approved by all authors for publication. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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