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Shale oil micro-migration characterization: Key methods and outlook

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Abstract:
Research has identified and increasingly explored the micro-migration phenomenon in shaly strata, which is currently one of the key scientific issues affecting shale oil accumulation and efficient development. Recently, qualitative and quantitative methods for characterizing hydrocarbon fractionation related to shale oil micro-migration have been proposed, which brought promising prospects to oil micro-migration research. Three key techniques in this field are summarized in this minireview, and the outlook for shale oil micro-migration characterization is prospected. Fourier transform ion cyclotron resonance mass spectrometry can be employed to distinguish subtle composition differences related to short-distance migration; core-flooding extraction experiments can be utilized for the quantitative characterization of micro-migration in organic-rich shale; and semi-open thermal simulation experiments are useful to analyze the chemical composition and structural evolution of expelled and retained oil. These three methods have different focus and advantages, while they provide different viewpoints and means for the characterization of shale oil micro-migration and have all achieved good results in different regions. Studies regarding the latest technologies deepen our understanding of the short-distance migration of shale oil, as well as improve our knowledge of the mechanisms of shale oil micro-migration, which is of great practical significance to the evaluation of shale oil content and mobility and further optimizes the identification of sweet spots and the effects of fracturing development.

1. Introduction

Medium-high maturity lacustrine shale oil serves as an important substitute for petroleum reserves and production globally (Speight, 2020; Wang et al., 2022; Zhao et al., 2023). With the continuous deepening of shale oil exploration and development, studies have recently established that shale oil accumulation cannot be simply summarized as "source-reservoir integration". Shale oil micro-migration, which has been identified and described by many researchers (Goodarzi et al., 2015; Mathia et al., 2016; Tuero et al., 2017; Zheng et al., 2023), refers to the oil migration between laminae or thin interlayers in a shale system with varying hydrocarbon generation and storage capability (Jin et al., 2021; Hu et al., 2024a). Shale formation has a clear definition: the thickness of a single layer of siltstone, fine sandstone and carbonate rock in the shale formation is less than 5 m, and the cumulative thickness accounts for less than 30% of the total thickness (Zou et

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al., 2020). Shale oil micro-migration is a bridge connecting the accumulation elements of shaly strata, reflecting the whole process of shale oil generation, expulsion and accumulation (Hu et al., 2024b). Furthermore, it affects oil mobility and efficiency during fracturing development, which is one of the key scientific issues in the efficient exploration and development of shale oil (Jin et al., 2021). Therefore, research on the characterization of shale oil micro-migration has great guiding significance for sweet spot prediction and the efficient development of global shale oil, which is one of the key factors to ensure that shale oil becomes an effective substitute for petroleum reserves and production (Hu et al., 2024c).

Whilst the micro-migration of shale oil has been investigated by many researchers with certain success, the mechanisms of oil micro-migration in organic-rich shales have yet to be revealed (Padin et al., 2014; Teixeira et al., 2017; Hu et al., 2024b). It has been confirmed that compositional changes are highly likely to occur during petroleum migration (Seifert and Moldowan, 1978; Mackenzie et al., 1983; England, 2007). However, due to the short migration distance of shale oil, the variations in shale oil composition are relatively minor, which are unrecognizable by conventional hydrocarbon fractionation identification methods. By tracking the latest techniques in the qualitative and quantitative characterization of hydrocarbon fractionation variations during shale oil micro-migration, including Fourier transform ion cyclotron resonance mass spectrometry, core-flooding extraction, and the semi-open thermal simulation experiment system, this minireview summarizes the latest research methods and specific applications of practical significance for shale oil exploration and development and highlights their outlook in shale oil micro-migration characterization. More than 50 papers employing these three methods were mainly referenced, with research objects including shales in different regions such as the Ordos Basin, Jianghan Basin in China, the Pannonian Basin in Hungary, the Lower Saxony Basin in Germany, and different lithofacies such as marine and continental facies, along with different thermal maturity evolution stages from immature to low-maturity to over-mature.

2. Characterization using fourier transform ion cyclotron resonance mass spectrometry

The advent of fourier transform ion cyclotron resonance mass spectrometry (FT-ICR MS) technology has offered a powerful option for examining the content and composition of non-hydrocarbons in crude oil (Poetz et al., 2014; Marshall and Chen, 2015). Non-hydrocarbons have a more intricate composition compared to hydrocarbons, serving as effective tracers for the generation, migration and accumulation processes of shale oil (Yue et al., 2021; Han et al., 2022).

Focusing on a shale section of Zhang 22 of the Ordos Basin, which is known for short-distance petroleum migration within shales (Zou et al., 2019), the compositional fractionation of non-hydrocarbons was investigated and further utilized to identify “sweet spots”. To unravel the fractionation characteristics and mechanism of non-hydrocarbons during migration, three core types were chosen:

1) Typical petroleum-generation shales with positive expulsion efficiency, which maintain fewer oils than they generate, representing typical source rocks that expel oils outward.
2) “Sweet spot” shales that exhibit negative expulsion efficiency, accumulating extra liquids beyond their generative capabilities.
3) Siltstones, which also exhibit negative expulsion efficiency, representing typical reservoirs without substantial petroleum-generation capacity (Zou et al., 2019).

Compared to typical petroleum-generation shales, “sweet spot” shales, that behave like siltstones, exhibit enriched non-hydrocarbons characterized by long alkyl side chains and a low degree of condensation. This may be attributed to the alkyl side chains around the polar functional group, which shields the polarity of functional groups, reduces rock adsorption and enhances molecular mobility. Additionally, low aromatization molecules weaken the π-π bond in aromatic rings, inhibiting crude oil aggregation and precipitation, thus facilitating petroleum flow. Furthermore, non-hydrocarbons with long alkyl side chains and a low condensation degree are the early-generation products from kerogen (Yuan et al., 2023). During the early stage, the formation is water-saturated with favorable porosity and permeability (Begum et al., 2019; Gholinezhadateni and Rostami, 2021), also facilitating the migration and accumulation of early-generated molecules with long side chains and low aromaticity.

After noise elimination and intensity normalization of the non-hydrocarbon data, compositional similarities among samples can be quantified using cosine distance (Yuan et al., 2024). Taking interlayer siltstones as benchmarks, the shales with high compositional similarities indicate “sweet spots”, since they might have undergone similar compositional fractionation. Taking Z22 well as an example, comparing B1/B2 siltstone (columns 2/3), “sweet spot” shales (C1/C2) show high similarity, while typical petroleum-generation shales (A1/A2/A3, line 1/4/7) display weaker similarity (Fig. 1).

Within individual units of typical petroleum-generating shales and “sweet spot” shales, variations in shale oil composition are relatively minor. However, as the composition gradually changes during petroleum migration, substantial compositional differences appear between these two units, and this gradual shift in compositional similarity can be used to infer the potential migration direction of shale oil. Taking the Z22 well as an example, when a typical petroleum-generating shale is used as a reference, it shows a high compositional resemblance with neighboring petroleum-generating shales. Yet, transitioning toward “sweet spots”, a noticeable decline in compositional similarity occurs (Fig. 1). Revealing these changes is anticipated to deepen our understanding of the short-distance migration of shale oil and refine the existing exploitation strategy in unconventional petroleum systems.

3. Characterization using core-flooding extraction

Core-flooding extraction can be used to both qualitatively and quantitatively characterize the micro-migration of shale

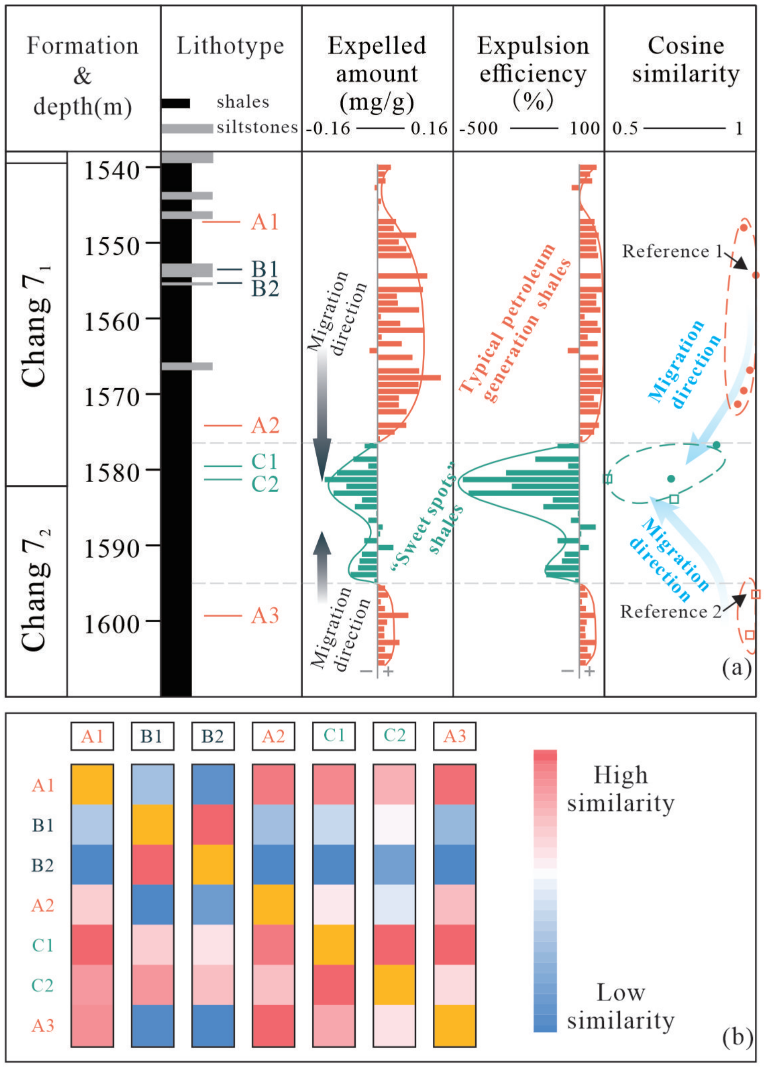

Fig. 1. (a) Compound-specific mass balance calculation of the Zhang 22 well (modified from Zou et al. (2019)), and the non-hydrocarbons similarities of rock extracts to two specific typical petroleum generation shales. Note: Series A (orange) represents typical petroleum generation shales; Series B (dark blue) indicates siltstone interlayers; Series C (green) corresponds to "sweet spot" shales. The circular dots represent the compositional similarity with a shale (Reference 1) from the upper typical petroleum generation unit, whereas the square dots indicate similarity with a shale (Reference 2) from the lower typical petroleum generation unit. (b) Compositional similarity analysis among extracts calculated by cosine similarity. The sampling locations are shown in (a). Note: For each column, the redder the color, the higher the similarity between the rock extract and the top first sample (boxed codes), while the bluer the color, the lower this similarity.

oil in organic-rich shales. This method requires:

1) A confining stress value at or close to the lithostatic pressure that enables the intact cylindrical rock plugs to maintain their pore structure under reservoir conditions.
2) A pressure difference of 10 MPa on two sides of the plugs, acting as the driving force for hydrocarbon migration.
3) A steel cylinder containing compressed helium for gas permeability measurements prior to and post core-flooding extraction.
4) An isolated helium pycnometer for the determination of porosities before and after extraction.

Based on the specific aim of a study, other approaches can be adopted, such as total organic matter content, Rock-Eval pyrolysis, optical microscope, scanning electron microscopy, Micro-CT, Thin-Layer Chromatography-Flame Ionization Detection, gas chromatography coupled with a flame-ionization detector, and gas chromatography coupled with mass spectrometry. At present, there are three key considerations derived from core-flooding experiments:

1) The quantity of movable shale oil.
2) Compositional fractionation.
and
3) Porosity and permeability changes with the removal of

soluble organic matter in the pore system of organic rich shales.

Three studies analyzed the quantity of movable shale oil, and the results show that the extraction is never exhaustive irrespective of shale type. For the marine Posidonia Shale, it was concluded that 40%∼61% of the total extractable organic matter can be extracted by a core-flooding experiment depending on the thermal maturity stage (0.53%, 0.88% and 1.45% VRo) and the amount of petroleum remaining in the pore system (Mohnhoff et al., 2016). For the lacustrine Shahejie Shale, it was proved that 56%∼84% of free hydrocarbons and 0.4%∼29.2% of heavy hydrocarbons can be extracted by a core-flooding experiment based on Rock-Eval pyrolytic analysis (Xie et al., 2019). It is noteworthy that the duration of experiment was different between Mohnhoff et al. (2016) and Xie et al. (2019); Mohnhoff et al. took 10 days for all their studied samples, while 3 to 27 days were used for different samples in Xie et al. (2019). Data collected by Mohnhoff et al. (2016) and Xie et al. (2019) was integrated with other results obtained from early mature clay-rich and carbonate-rich Posidonia Shale to calculate the extraction efficiency, which is defined as the ability of the rock to release hydrocarbons during core flooding extraction in a given time, pressure difference, cross sectional area, sample length and volume (Zhang et al., 2021). The resulting extraction efficiency in Zhang et al. (2021) is much lower than that in Mohnhoff et al. (2016) and Xie et al. (2019), which is closely related to the permeability of studied samples rather than differences in thermal maturity and type of shale. Based on the results of the above three studies, the micro-migration of shale oil is likely controlled by the permeability of organic-rich shales.

Fractionation effects during core-flooding test were observed in all three studies but from different aspects. Significant variations in compound groups were discovered, including saturates, mono/diaromatic, polyaromatic, polars, and asphaltenes (Mohnhoff et al., 2016). The compound group variation patterns were different among different specimens. Polyaromatic compounds were the predominant group in the sequential extracts of the oil immature sample, followed by saturates and mono-/diaromatic compounds with equal abundance. In contrast, mono-/diaromatic compounds took the largest proportion in the oil mature sample, followed by polyaromatic and saturate fractions. In the oil over-mature sample, saturates comprised the most abundant fraction at the early stage of extraction, which were later substituted by polyaromatic compounds. Different from Mohnhoff et al. (2016), Xie et al. (2019) claimed that lighter hydrocarbons (<C19) were preferentially extracted during a core-flooding test, with slightly enriched low molecular weight hydrocarbons at the early stage of extraction. In stark contrast, acidic compounds were preferentially extracted at the early stage, and pristane and phytane in the whole process compared to n-alkanes of similar chain length (Zhang et al., 2021) (Fig. 2). Besides, sequential extracts show a higher maturity compared with those retained in the rock after core-flooding extraction (Zhang et al., 2021); however, in this study, preferential extraction of low-molecular-weight hydrocarbons did not occur. Obviously,

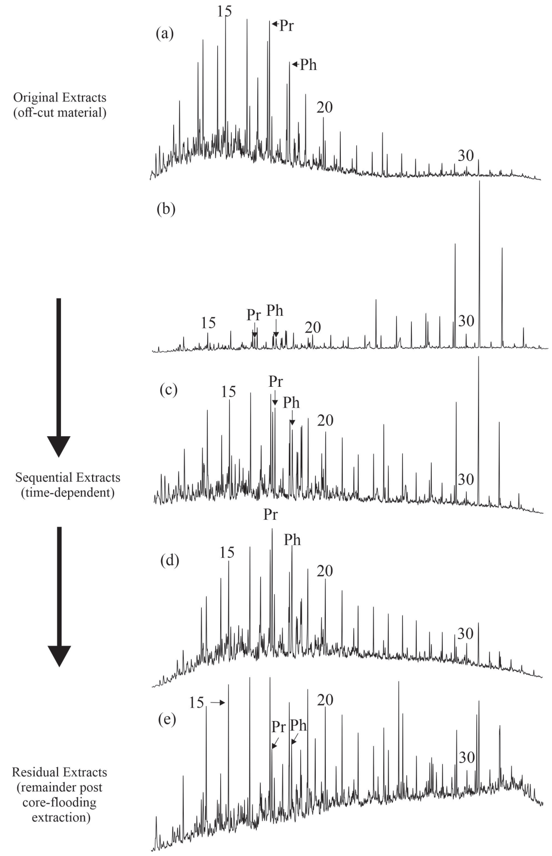

Fig. 2. (a) Chromatograms of original, (b)-(d) sequential and (e) residual extracts (modified from Zhang et al. (2021)).

the composition of extracts collected at different stages of core-flooding extraction were different. Nevertheless, whether better quality hydrocarbons (with relatively low molecular weight) could be preferentially extracted needs further investigation. These studies confirmed that compositional changes are likely to occur during the micro-migration of shale oil, which process can contribute to the accumulation of a certain type of petroleum compounds in more opened pore spaces.

Absolute porosity increments of 0.6% 1.7% were documented by Mohnhoff et al. (2016) and those of 1.14% 1.4% by Xie et al. (2019). Similarly, permeability was reported to increase from tens to about one thousand nD in Mohnhoff et al. (2016). In contrast, both porosity and permeability were decreased in Zhang et al. (2021)'s work. Meanwhile, the ambiguity of these three studies is that samples were dried under stressed condition by Zhang et al. (2021) but at an unconfined condition and a temperature of 105 °C by Mohnhoff et al. (2016) and Xie et al. (2019). Drying the samples containing organic solvent at unconfined stress condition and high temperature (105 °C) is thought to change the frame of the shale. Notably, the micro-migration of shale oil always occurs under stressed condition. Thus, the boundary conditions in Zhang et al. (2021) are closer to reservoirs and experimental procedures should be optimized based on the realistic geological conditions, although laboratory circumstances are never the same as those in the subsurface.

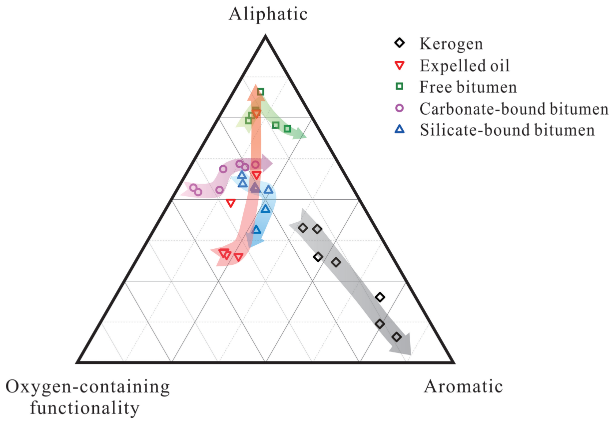

Fig. 3. Ternary diagram showing the relative distribution of three major functional groups (i.e., aliphatic, aromatic and oxygen-containing functionalities). The arrowed curves indicate the thermal evolutions of kerogen, expelled oil and bitumen fractions during maturation within the oil window. The comparative stability of compositional structures of bitumen throughout the oil window stage suggests that the retention of generated hydrocarbons is largely related to the interaction with mineral matrix and kerogen (modified from Pan et al. (2023)).

4. Characterization using thermal simulation

Exploring the chemical composition and structural evolution of the expelled and retained oil in different occurrence states during the dynamic evolution of hydrocarbon generation in organic-rich shale is of great significance for studying the hydrocarbon expulsion process of shale and the retention behavior of liquid hydrocarbons. Hydrous thermal simulation experiments in semi-open systems are often used to describe the geological processes of hydrocarbon generation, expulsion and retention in shales (Lewan, 1985; Le Doan et al., 2013). Pan et al. (2018a) quantitatively analyzed the hydrocarbon generation rate and oil group composition of thermal simulation sequence in organic-rich shale, and the results indicated that the oil generation process is a two-step transformation of "kerogen→bitumen→oil". In addition, with increasing thermal maturity, the expelled oil gradually becomes rich in hydrocarbons. These small molecules formed by the thermal cracking of bitumen improve the oil fluidity, thereby enhancing the hydrocarbon expulsion effect. Studying the composition of hydrocarbon generation products via thermal simulation experiments has revealed the shale oil generation, storage and preservation process at the component or molecular level to a certain extent.

Shale oil occurs mainly in free, adsorbed and dissolved states (Jarvie, 2012; Li et al., 2018). In the early oil generation stage, because the physical/chemical properties of early hydrocarbon generation products are similar to kerogen, shale generally shows strong adsorption capacity. The existing geochemical characterization methods of shale oil occurrence mainly include pyrolysis and solvent extraction. The former is favored owing to the fact that shale oils in different occurrence states have different molecular thermal volatilization capacities. For example, shale oil in fractures and macro-pores is more likely to be released than oil in micropores; small-molecule compounds are more likely to be released than large-molecule compounds; and free compounds are more likely to be released than adsorbed compounds (Jiang et al., 2016; Li et al., 2024). The free and bound retained oil in shales can be separated by the experimental method of organic solvent extraction in conjunction with the dissolution of carbonate and silicate minerals. On the other hand, the retained oil in organic-rich shales can be divided into free phase and bound phase in association with carbonate minerals and silicate minerals/kerogen (Pan et al., 2018b). Free oil is mainly composed of light hydrocarbon components, which usually occurs in micro-fractures and large pores of shale and has good fluidity. Bound oil is mainly composed of medium-macromolecules and polar components with poor fluidity, which are closely bound to the mineral surface and kerogen mainly by physical adsorption and non-covalent chemisorption. Apparently, differences exist in the chemical composition of shale oil (bitumen) in different occurrence states obtained by stepwise solvent extraction, which may be related to the adsorption of inorganic minerals and kerogen as well as the primary petroleum migration (Price and Clayton, 1992; Li et al., 2023). It has also been suggested that oil mainly exists in reservoir pores but is mainly adsorbed by kerogen in shales (Pepper, 1991; Sandvik et al., 1992; Dang et al., 2022; Cai et al., 2024).

Electrospray ionization coupled with FT-ICR MS was utilized to investigate the chemical composition and distribution of acidic and neutral nitrogen-containing compounds in different shale oil occurrence states, and it was found that neutral nitrogen-containing compounds mainly exist in free oil, while carboxylic acid compounds occur predominantly in bound oil (Pan et al., 2019). Oxygen-containing polar functional groups in these acidic compounds are considered to be responsible for the main bonding force that promotes the interaction between organic matter and inorganic minerals/kerogen. Similarly, the structural evolution characteristics of kerogen and shale oil in different occurrence states during the process of hydrocarbon formation and evolution of organic-rich shales were revealed (Pan et al., 2023). These results showed that with increasing maturity, the chemical structure evolution of kerogen and expelled oil display opposite trends: the aromaticity of kerogen molecules continues to increase, while the expelled oil gradually enriches aliphatic hydrocarbons with growing maturity, especially after a significant hydrocarbon expulsion from the shales. However, within the entire oil generation window, the composition and structure of free and bound retained oil exhibit minor changes (Fig. 3). The above results indicate that the thermal evolution of organic matter and the compositional fractionation of chemical components related to primary oil migration are both responsible for the variations in the chemical composition and structure of retained oil. Affected by the adsorption of inorganic minerals and kerogen as well as the porous space of shale, high molecular weight polar components (e.g., asphaltene and resins) are preferentially adsorbed onto the mineral surface and kerogen, whereas hydrocarbon-rich components are mainly enriched in the pores/fractures of shales due to their relatively weak polarity. To some extent, this clarifies the oil micro-migration and enrichment mechanism in shales during hydrocarbon generation and evolution from the perspective of molecular organic geochemistry.

5. Outlook

Oil content and mobility are important parameters for shale oil exploration and efficient development, directly controlling the distribution of “sweet spots” and the effect of fracturing development (Wang et al., 2025). As a key factor controlling oil content and mobility, shale oil micro-migration is a bridge connecting the accumulation elements of shale strata. Therefore, the three discussed methods for characterizing shale oil micro-migration not only improve our understanding of the mechanism of shale oil micro-migration but also have important practical significance for shale oil sweet spot exploration and efficient fracturing development, providing great application prospects. Nevertheless, they also have their limitations and shortcomings that need to be addressed.

Specifically, FT-ICR MS can provide a detailed depiction of the molecular composition of complex organic compounds, rendering it a powerful tool in petroleum geology studies: it has been tentatively deployed to investigate crude oil properties, oil-rock interactions, oil-source correlations, sedimentary environments, microbial interactions, maturities, sulfate thermochemical reduction reactions, migrations, etc. Despite its widespread applications, however, the composition and governing factors of non-hydrocarbons exhibit a significantly more intricate nature compared to their hydrocarbon counterparts. Currently, our understanding of the non-hydrocarbon compounds in crude oil remains relatively limited, demanding intensified investigative efforts to delve deeper into this challenging subject matter. Moreover, considering FT-ICR MS as a data-intensive analysis technology, integration with big data technologies is pivotal to efficiently manage vast datasets and extract the key information, which can help to better unravel the formation and alteration processes of shale oil.

The most remarkable shortcoming of core-flooding extraction is the artificial pore pressure exerted by using pumps. However, in the shale oil system, constant pore pressures are unlikely exist at the geological timescale. This experimental drawback prompts the important question of what the driving force for shale oil micro-migration is and how it is formed. Therefore, future works will be of merit in deciphering the reasons attributed to the micro-migration of shale oil. If new insights were gained on this matter, the behavior of petroleum in organic-rich shales could be grasped and the pathway of shale oil micro-migration could be figured out, and the spatiotemporal distribution of high-quality shale oil resources could be determined.

Unlike conventional oil and gas reservoirs, the micro-migration of hydrocarbons is closely related to their own physical/chemical properties, the adsorption of inorganic minerals and kerogen, the pore structure, connectivity, and the degree of microcrack development of the source rocks. Quantitative characterization of the occurrence state of retained hydrocarbons and a detailed description of the chemical composition and structure of hydrocarbons will help to deepen research on the micro-migration and enrichment mechanism of shale oil. At present, the dynamics of oil generation is gradually advancing towards the component level. To this end, the phase evolution of hydrocarbon components and the generation-expulsion-retention dynamics of crude oil group components are important directions for studying the occurrence mechanism of shale oil, and exploring relevant geochemical parameters at the molecular level is expected to be an important topic in the study of shale oil micro-migration.

6. Conclusions

Employing FT-ICR MS in the analysis of petroleum reveals its exceptional ability to detect thousands of individual molecules. The rich dataset generated by this technique is highly accurate and sensitive to the input, allowing it to distinguish subtle variations among different extracts. This fine resolution of compositional variations can decode petroleum fractionation within shale formations, concurrently presenting a novel approach for identifying “sweet spots” within shales and offering fresh opportunities for enhancing resource targeting in shale oil exploration.

Based on core-flooding experiments, the shale oil micro-migration process is better understood. The micro-migration speed can be expected to decrease from the start of petroleum expulsion, and this process is likely associated with compositional fractionation as well as changes in the transport and storage capacity of shales. Certain types of hydrocarbons compounds may accumulate in more opened pore spaces, which can be regarded as potential “sweet spots” for shale oil exploration.

During the thermal evolution of shales, weakly polar or non-polar saturated hydrocarbons, aromatic hydrocarbons, and neutral nitrogen-containing compounds mainly exist in the pores/ fractures of source rocks in a free state or in a weakly physically adsorbed form, and are preferentially expelled when the hydrocarbon expulsion proceeds. However, oxygen-containing functional groups are relatively abundant in macromolecular-polar compounds, which are easily combined with inorganic minerals or kerogen via ionic bonds and/or hydrogen bonds to produce adsorption and have relatively poor fluidity.

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Conflict of interest

The authors declare no competing interest.

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