

Original Paper

Hydrocarbon generation thermal simulation experiment of massive organic-rich tuff and laminar tuffaceous shale: A case study of the Chang 7 member of the Ordos Basin

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ARTICLE INFO

Article history:

Received 20 May 2025

Received in revised form

25 September 2025

Accepted 23 December 2025

Available online 6 January 2026

Edited by Xi Zhang and Jie Hao

Keywords:

Ordos Basin

Chang 7 Member

Organic-rich tuff

Tuffaceous shale

Hydrocarbon generation intensity

Experimental study

ABSTRACT

The Chang 7 Member of the Yanchang Formation in the Ordos Basin consists predominantly of airfall and water-borne tuff along with tuffaceous shale, which exhibit considerable hydrocarbon potential, yet their thermal evolution and hydrocarbon generation behaviors remain inadequately studied. As an emerging unconventional exploration target, this interval was investigated through systematic thermal simulation experiments utilizing a novel programmable dual-pressure constant-flow system, which enables precise control of fluid pressure and automated monitoring of hydrocarbon generation and expulsion processes under semi-open/semi-closed conditions. The study focuses on low-maturity, organic-rich massive tuff and laminated tuffaceous shale from Well Z40, revealing that although all samples are sedimentary in origin, the airfall tuffs are rich in vitric pyroclasts—including accretionary pellets (AP1)—while water-borne tuffs are dominated by feldspar crystal pyroclasts. Key findings demonstrate that massive tuff displays markedly higher hydrocarbon expulsion efficiency compared to laminated tuffaceous shale, with an earlier onset of expulsion and contributions derived from both kerogen cracking and heavy oil components. Thermal evolution analysis further indicates that massive tuff attains higher organic maturity under identical conditions. In laminated shale, expulsion efficiency correlates increases with TOC content, reflecting the role of organic matter in pore development and fluid connectivity. The superior expulsion performance of massive tuff is attributed to its higher volcanic glass content, low clay abundance, and dispersed organic matter distribution, which create highly efficient migration pathways. In contrast, laminated tuffaceous shale exhibits stratified organic matter closely associated with clay minerals, requiring higher thermal maturity ($R_o > 0.6\%$) and elevated TOC to form an interconnected organic network that facilitates efficient expulsion. This study highlights that variations in pore structure, organic matter distribution, and thermal maturity represent the primary controls on differential expulsion efficiency between organic-rich tuff and tuffaceous shale in the Chang 7 Member, providing crucial insights for the exploration and development of shale oil in volcanic-lacustrine basins.

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Peer review under the responsibility of China University of Petroleum (Beijing).

1. Introduction

Volcanic activity is a relatively common geological phenomenon in nature, and volcanic eruptions are an important process and direct manifestation of volcanic activity. Volcanoes are divided into red volcanoes (plate rifting, without erupted materials) and gray volcanoes (plate compression collision, explosive volcanoes, containing a large amount of volcanic ash). Tuff is produced by explosive volcanic eruptions and is widely deposited in geological strata from the Precambrian to the Cenozoic (Zhang et al., 2017, 2022; Wu et al., 2024). The frequent ancient volcanic eruptions in the Chang 7 Member of the Ordos Basin were caused by the continuous compression and collision of the southern Qinling Orogenic Belt, forming a large amount of volcanic ash during the eruption process. The volcanic ash entered the Chang 7 lake basin in the form of airfall or water transport, forming a large amount of tuff or tuffaceous laminae. The Chang 7 Member belongs to the late Ladinian of the Middle Triassic, deposited approximately 10 million years after the Permian–Triassic mass extinction (PTME). The paleontological community (and/or sedimentary environment) of this member demonstrates a new stage reached by the Middle Triassic marine/terrestrial ecosystem after a prolonged recovery, reflecting the ultimate outcome of the synergistic evolution between organisms and the environment following the PTME (Zhao et al., 2020). Zheng et al. (2011), based on extensive testing of major, trace, and rare earth elements, concluded that the volcanic ash of the Yanchang Formation originates from a volcanic arc calc-alkaline magmatic rock area.

The Chang 7 Member of the Ordos Basin is rich in shale oil resources. As a key area, the Qingcheng Oilfield has established China's first billion-ton-scale shale oil field within the Member's black shale series, demonstrating its huge potential (Zou et al., 2023a, 2023b). The shale series of the Chang 7 Member is generally characterized by the development of exceptionally high organic matter shale and interbedded tuff, with an average total organic carbon (TOC) content of up to 11.02%, and the highest reaching 35.8% (Liu et al., 2021). Tuff is highly developed in the Chang 7 Member, mainly consisting of vitric pyroclast/crystal pyroclast altered tuff and sedimentary tuff (Zhang et al., 2009). Initial research on the tuff layers of the Chang 7 Member has examined their petrology, genesis, and sedimentation, indicating their potential as a target for unconventional oil and gas exploration. The R_o of the Chang 7 Member mainly ranges from 0.5% to 1.3%, with the developed tuff mostly having massive and laminated structures, and the organic matter content in the tuff generally ranges from 0.2% to 5%. However, the hydrocarbon generation characteristics of organic-rich tuff and laminated tuffaceous shale during thermal evolution are not yet clear.

Previous studies on the hydrocarbon generation thermal simulation of the Chang 7 Member have mainly focused on clay-rich shale, however, the hydrocarbon generation characteristics of organic-rich tuff and tuffaceous shale, especially the generation characteristics of retained oil, have not been reported. The continental oil shale of the Chang 7 Member has a huge potential for hydrocarbon generation, and the content of retained oil in the oil shale of the Chang 7 Member per ton of rock is nearly 7 times that of marine shale, and the content of retained oil per ton of organic carbon is nearly 60% higher than that of marine shale (Wang et al., 2021; Zou et al., 2023a, 2023c). A number of studies have focused on the factors controlling oil generation and oil retention during the thermal simulation of the Chang 7 Member. For example, Qi et al. (2019) compared the hydrocarbon generation characteristics of black shale and dark mudstone of the Chang 7 Member, and found that the activation energy of black shale is significantly higher than that of dark mudstone, showing a rapid hydrocarbon

generation characteristic; the main hydrocarbon generation period of black shale corresponds to the R_o range of 0.70%–0.87%, while the main hydrocarbon generation period of dark mudstone corresponds to the R_o range of 1.06%–1.72%. Wang et al. (2021) found that when the proportion of argillaceous siltstone in mud shale increases, both the oil yield and the content of retained oil increase significantly. Li et al. (2022) found that pyrite can promote the increase of the total oil yield of the Chang 7 Member shale, and at the peak of oil production, it can increase the total oil yield by up to 54.17 mg/g. Dang et al. (2022) believe that an R_o of 0.75% is the maturity boundary for the occurrence state and mobility transformation of shale oil in the Yan'an area of the Chang 7 Member, while an R_o of 0.85%–0.90% is the lower limit of the best maturity window for shale “with high oil content and good mobility”.

However, despite these valuable insights, a critical gap remains. Previous thermal simulation studies have predominantly focused on clay-rich shale or mudstone, largely neglecting the role of ubiquitous tuffaceous materials (Wang et al., 2021; Dang et al., 2022; Zhai et al., 2024). The hydrocarbon generation characteristics, retention mechanisms, and expulsion efficiency of organic-rich tuff and laminated tuffaceous shale—key components of the Chang 7 Member—are still poorly constrained. This lack of knowledge significantly hinders our ability to accurately evaluate the shale oil enrichment potential and exploration prospects in these complex hybrid formations (Hu et al., 2023). Therefore, this study aims to fill this research gap by explicitly investigating the hydrocarbon generation and retention behaviors of tuffaceous rocks. The specific objectives of this study are: (1) to characterize the geological and petrological features of organic-rich massive tuff and laminated tuffaceous shale from the Chang 7 Member; (2) to conduct systematic hydrocarbon generation thermal simulation experiments on these tuffaceous samples; (3) to quantify and compare the yields, composition, and generation intensity of expelled and retained hydrocarbons from these rocks; (4) to evaluate the hydrocarbon expulsion efficiency and discuss its implications for shale oil accumulation. The novelty of this work lies in its dedicated focus on tuffaceous rocks as a primary subject for hydrocarbon generation simulation, rather than as a secondary component. This approach provides unique insights into how volcanoclastic materials, when enriched with organic matter, contribute to and influence the hydrocarbon generation process. Our findings are expected to establish a foundational model for evaluating the hydrocarbon potential of tuffaceous shale sequences, offering a new perspective for unconventional oil and gas exploration in volcanic-rich lacustrine basins.

This study employs geological analysis, thin-section petrography, and thermal simulation experiments of hydrocarbon generation on massive organic-rich tuff and laminated tuffaceous shale samples from the Z40 well in the Chang 7 Member of the Ordos Basin. A systematic analysis of oil and gas generation components was conducted, and a comparison was made on the changes in hydrocarbon production, differences in hydrocarbon generation intensity, and expulsion efficiency. This study helps researchers better understand the hydrocarbon generation characteristics and exploration potential of such rocks.

2. Geological background

The study area is located in the southern region of the Ordos Basin, where the massive organic-rich tuff and laminated tuffaceous shale of the Chang 7 Member are taken from the key exploration well Z40 (Fig. 1(a)). The thickness of the tuffaceous shale in the Chang 7 Member of the well Z40 is 25 m. There have been numerous reports on the geochemistry and exceptionally high organic matter enrichment pattern of the clay-rich shale

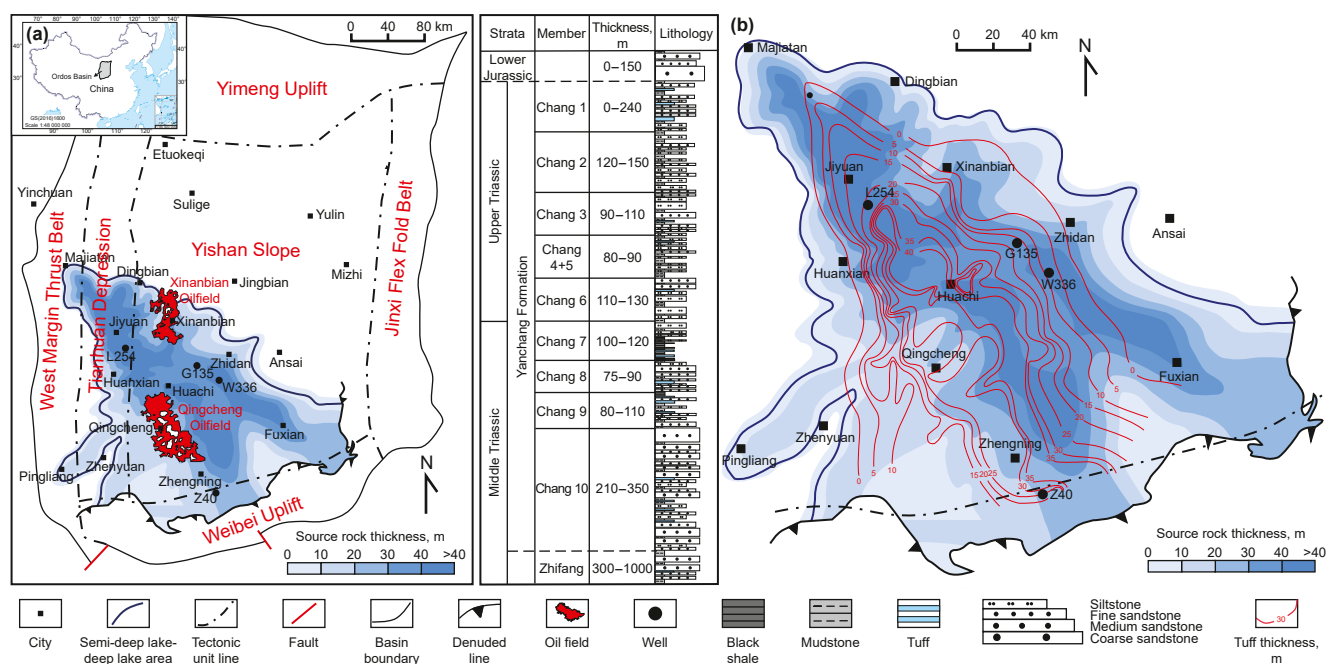


Fig. 1. The study area is located in the southern part of the Ordos Basin, and the samples are taken from the Chang 7 Member in the Yanchang Formation of Well Z40. Notes: Delineation of stratigraphic age according to the references Cohen et al. (2013); Li et al. (2016); Deng et al. (2018). (a) The location of the study area and sampling wells, (b) planar distribution map of the thickness of tuff in the Chang 7 Member of the study area (modified after Zhang et al., 2009).

section of the Chang 7 Member in well Z40, while there are few reports on the tuffaceous components (Liu et al., 2021, 2022, 2023). Additionally, previous research has accurately reconstructed and classified the paleogeomorphic units of the Chang 7 Member by combining the impression method with sedimentological approaches (Lv et al., 2022). This study fully considers and draws on the paleogeomorphic features and sedimentary environment of the lake basin. The Chang 7 Member of Well Z40 belongs to a deep lacustrine facies deposition and is located near the southern part of the lake basin, having experienced intense volcanic activity. Therefore, selecting this well for diagenetic thermal simulation research of tuffaceous components is both representative and offers unique advantages.

The depositional period of the Chang 7 Member is the Late Ladinian of the Middle Triassic, during which volcanic eruptions were frequent, forming tuff layers with a cumulative thickness of 0–35 m (Fig. 1(b)). The planar distribution of the tuff layers is basically consistent with the ancient lake basin of the Chang 7 Member (Fig. 1(b)), indicating that the tuff of the Chang 7 Member is mainly of sedimentary origin. The structure of the Chang 7 Member in the Ordos Basin is relatively gentle, with the main development of small amplitude nose-like uplifts with a rise of less than 10 m within a distance of a kilometer.

Earth's orbital cycles generally refer to the three periods controlled by Earth's orbital parameters: eccentricity, obliquity, and precession. Eccentricity describes the degree of deviation of Earth's orbit from a perfect circle, ranging from 0.05% to 6.07%, with an average value of 3%. Eccentricity cycles can be divided into long and short eccentricity cycles (Shi et al., 2023). Fig. 2 illustrates the sedimentary cycle characteristics of Well Z40, where 5 m represents the short eccentricity cycle. The sub-members Chang 7₁ to Chang 7₃ each contain six short eccentricity cycles. The R_0 value of the shale in the Chang 7 Member of well Z40 is low (0.5%), making it suitable for thermal simulation studies. Previous studies have combined imprint methods with sedimentological methods to more accurately restore and divide the paleogeographic units of

the Chang 7 Member; the sediments in well Z40 are all deep lake facies, mainly distributed in the central part of the ancient lake basin's depression (Lv et al., 2022). The Chang 7₃ sub-member at the bottom of well Z40 is developed with tuff, and the samples of this study are all taken from the bottom section of the well (Fig. 2). The sample numbers are HLP-726, HLP-707, HLP-708, and HLP-710, respectively. Among them, sample HLP-726 is a massive altered organic-rich tuff, and samples HLP-707, HLP-708, and HLP-710 are laminated tuffaceous shales (Fig. 2).

3. Methods

3.1. Total organic carbon (TOC) and vitrinite reflectance (R_0)

The determination of TOC is performed using a CS744 carbon and sulfur analyzer, in accordance with the standard GB/T 19145-2022 "Determination of Total Organic Carbon in Sedimentary Rocks". The measurement of R_0 is conducted using a CRAIC508PV microspectrophotometer and a DM4P microscope, following the standard SY/T 5124-2012 "Method for Determining the Reflectance of Vitrinite in Sedimentary Rocks".

3.2. Thin-section identification

Thin section observation is used to identify the microstructures of tuff and shale. The thin section identification is carried out using a Nikon E600 research-grade polarizing microscope, in accordance with the method of scanning electron microscope analysis of rock samples, SYT 5162-2014. The sample preparation method is as follows: first, select a location and cut it into a square block; then, grind the bottom surface of the cured rock sample through coarse grinding → medium grinding → fine grinding → precision grinding until the bottom surface becomes very flat; finally, attach a glass slide to the rock sample, cut the rock to reserve a mineral section of less than 1 mm, and polish the surface of the mineral section. A cover slip is then placed over the surface of the mineral section.

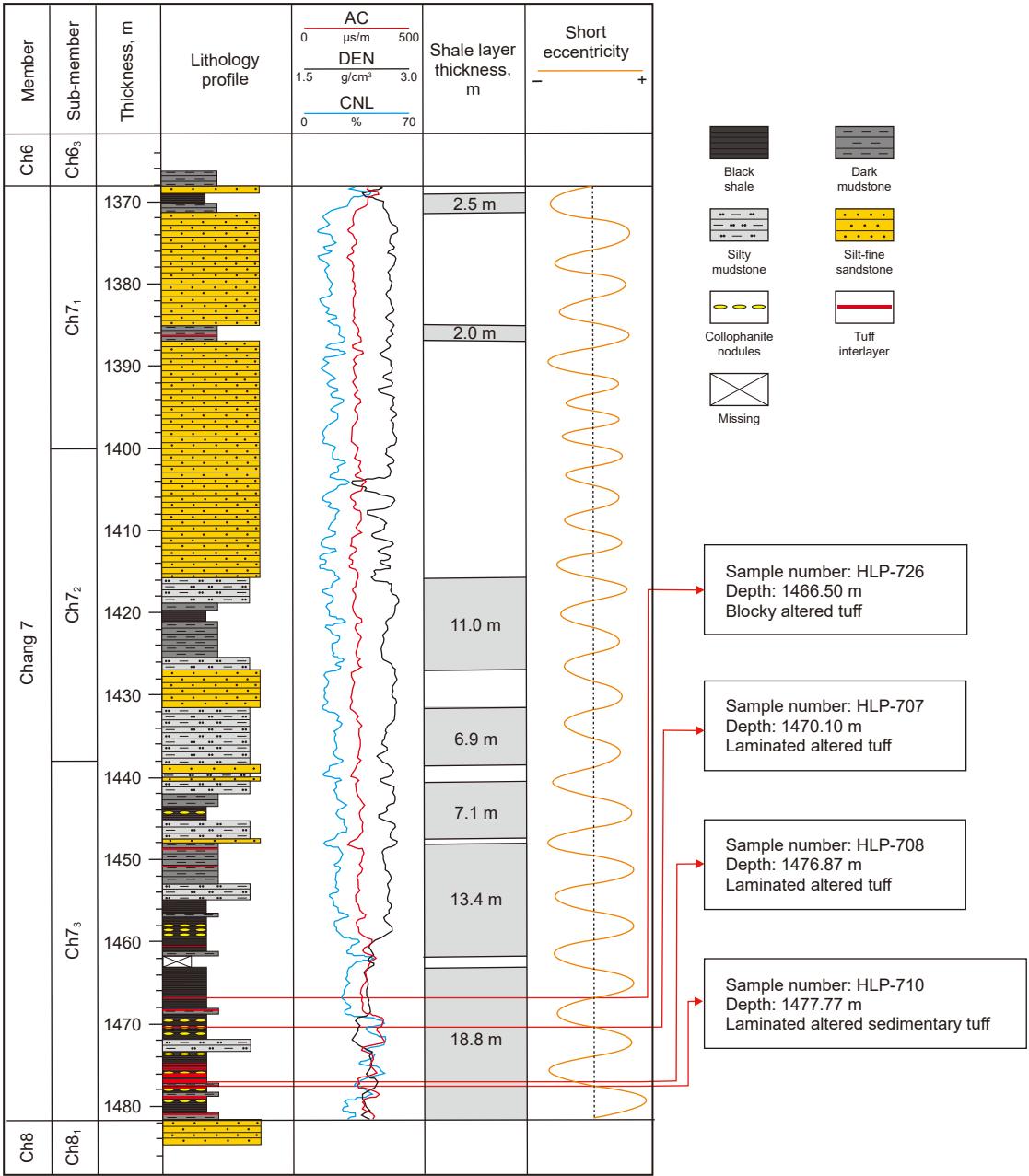


Fig. 2. Rock lithology columnar diagram of the Chang 7 Member in the Yanchang Formation of Well Z40.

The basic principle of thin-section observation is as follows: polarized light generated by a polarizer is directed at an anisotropic sample. The polarized light is decomposed and modified, producing a phase difference. It then interferes through an analyzer orthogonal to the polarizer, ultimately forming an interference image with brightness, darkness, and color contrasts. This reveals the microstructure and optical characteristics of the sample.

3.3. Scanning electron microscope (SEM) observation

Scanning electron microscopy (SEM) observations were conducted using an FEI QUANTA FEG 650 instrument. Equipped with a thermal field emission gun (FEG), the system generates a high-brightness, fine-focused electron probe. Under the control of

scanning coils, the probe scans the sample surface. By detecting various signals emitted from the sample—such as secondary electrons and backscattered electrons—the instrument provides ultra-high-resolution morphological and compositional information of the sample surface (Marzieh and Mohammad, 2023).

3.4. Thermal simulation apparatus and experimental procedure

This study uses a semi-open and semi-closed system, which can simulate the process of generating and expelling hydrocarbons simultaneously (Berner and Raiswell, 1983; Guo et al., 2017). The expulsion process is controlled by pressure or temperature, and the products also undergo secondary cracking, but it is not intense, making the simulation results more similar to actual geological conditions (Dai et al., 2020; Wu et al., 2022). This study employs

the newly developed constant pressure and steady flow device from the Guangzhou Institute of Geochemistry, Chinese Academy of Sciences (Fig. 3), which can achieve accurate control of pressure. Previous experiments maintained stable fluid pressure by manually intermittently opening the hydrocarbon expulsion valve to release flow, but this manual valve opening method could not accurately control the fluid pressure, leading to too fast hydrocarbon emission speed, thereby affecting the subsequent reactions of retained gas. This device, through the cooperation of dual valves alternating on and off and the buffer tube, can precisely control the fluid pressure and achieve automated control of hydrocarbon generation and expulsion.

Cylindrical samples (25 mm diameter, ~50 g weight) were prepared, with Rock-Eval pyrolysis performed on residual fragments to represent their pyrolysis parameters. Hydrocarbon generation and expulsion experiments were conducted under 50 MPa static rock pressure and stable fluid pressure using a programmed heating regime. For the sample HLP-707, the experimental parameters are set as shown in Table 1. Expelled oil was collected by rinsing the system with dichloromethane, while retained oil was extracted from ~1000 mg solid samples via 48-h dichloromethane soaking. Oil yields were analyzed by gas chromatography (GC model 7890B, deuterated *n*-C24 internal standard, column: 50 m × 0.32 mm × 0.45 μm; oven program: 40 °C (5 min), 4 °C/min to 290 °C (25 min)) for C5–C14 hydrocarbons, and by volatilization gravimetry for C14+ fractions.

The experiment consists of four sets of samples, with sample numbers HL-726, HLP-707, HLP-708, and HLP-710. Each set of samples includes five small cylindrical samples (5 cm in length, 2.5 cm in diameter), which are used to conduct diagenesis thermal simulation under different R_o conditions through constant temperature, constant pressure, and adjusted duration methods. Taking sample HLP-707 as an example, the parameters and step design of the diagenesis thermal simulation experiment are shown in Table 1. The highest heating temperature in the experiment design is 370 °C. This is mainly because the critical temperature of water is 374.3 °C, and the critical pressure is 22.12 MPa (Xu et al., 1995). When the temperature and pressure of water exceed these critical values, it is in a supercritical state. At this time,

supercritical water has a very strong oxidizing ability, can dissolve many substances, especially with a very high solubility for organic matter. In addition, the density and viscosity of water will drop sharply, and the chemical activity is enhanced. However, supercritical water does not exist in the process of hydrocarbon generation in natural source rocks, so the constant temperature in the experiment design is set at 370 °C. Furthermore, the designed R_o range of the experiment is between 0.7% and 1.3%, which is the main distribution interval of the Chang 7 Member in the Ordos Basin.

3.5. Chromatographic analysis

The full oil analysis of liquid hydrocarbons is completed on an Agilent-7890 chromatograph. The column model is HP-PONA (with a length, inner diameter, and film thickness of 50 m, 0.2 mm, and 0.5 μm, respectively). The injection port temperature is 300 °C, with no split injection. The temperature program is as follows: hold at 35 °C for 5 min; then ramp up to 70 °C at a rate of 3 °C/min; and then increase the temperature to 300 °C at a rate of 4.5 °C/min, holding for 35 min. The detector temperature is 300 °C, with a hydrogen flow rate of 40 mL/min and an air flow rate of 400 mL/min.

To prevent the dissolution of gases such as H₂S, the gases generated during experiments are collected using vacuum air bags. This method avoids the use of conventional water displacement, thus preserving the original gas composition. The quantification of the gas volume in the bag is done using the displacement method, which involves subtracting the volume of the bag itself from the total volume of the bag and the gas. The analysis of gas composition is performed on a Was-son-Agilent 7890 type gas chromatograph. This instrument is equipped with one flame ionization detector and two thermal conductivity detectors, allowing for the detection of hydrocarbon gases and non-hydrocarbon gases in a single analysis. The temperature program for the chromatography is as follows: heat from room temperature to 68 °C, hold for 7 min; then increase the temperature to 90 °C at a rate of 10 °C/min, hold for 1.5 min; and finally increase the temperature to 175 °C at a rate of 15 °C/min, hold for 1.5 min. Before sample analysis, the

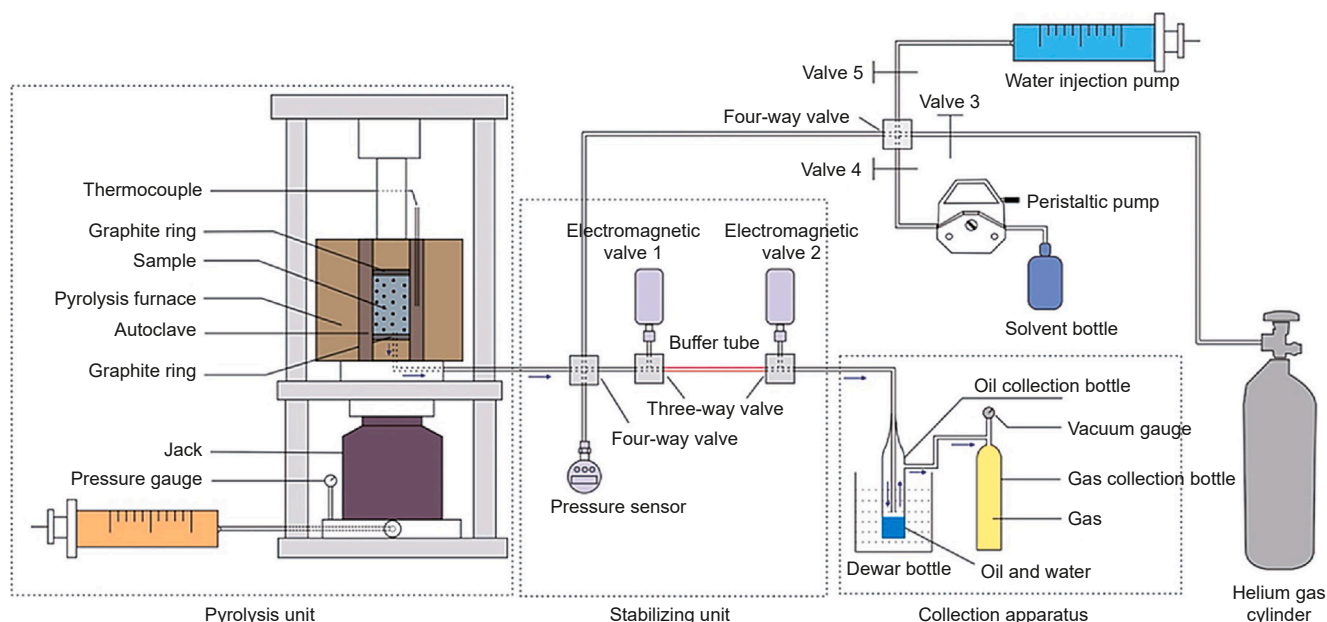


Fig. 3. Schematic diagram of the hydrocarbon generation thermal simulation experimental setup.

Table 1

Table of basic parameters and step design for thermal simulation experiment (taking HLP-707 as an example).

Sample	Sample weight, mg	Water addition volume, mL	Initial temperature, °C	Heating rate, °C/h	Isothermal temperature, °C	Isothermal time, h	R_o , %	Total time, h	Static rock pressure, MPa	Fluid pressure, MPa	Oil collection cold trap temperature, °C
HLP-707-1	51140	10	Room temperature	30	370	0.20	0.7	10.2	50	20	<−5
HLP-707-2	50530	10	Room temperature	30	370	4.92	0.9	14.92	50	20	<−5
HLP-707-3	49970	10	Room temperature	30	370	10.66	1.0	20.66	50	20	<−5
HLP-707-4	52860	10	Room temperature	30	370	20.50	1.1	30.5	50	20	<−5
HLP-707-5	49520	10	Room temperature	30	370	69.50	1.3	79.5	50	20	<−5

chromatograph is calibrated using a mixed standard gas containing C1–C5 normal alkanes and non-hydrocarbon gases. The error in each analysis does not exceed $\pm 0.1\%$.

4. Results

4.1. Petrological characteristics

All four groups of samples are taken from Well Z40, and the characteristics of their cores and thin sections are shown in Fig. 4. Sample HLP-726 is a massive altered sedimentary tuff, containing oriented discontinuous oil shale tear fragments (Fig. 4(a)). The HLP-726 sample interior contains abundant crystal pyroclasts and minor vitric pyroclasts, most of which show signs of devitrification. These crystal pyroclasts are primarily composed of quartz, with feldspar being less common (Fig. 4(a)). In addition, volcanic dust is mixed with normal sedimentary mud, and the tuffaceous components are generally impregnated with organic matter.

Samples HLP-707, HLP-708, and HLP-710 are laminated altered tuffaceous shales, with the tuffaceous components existing in a laminated form (Fig. 4(b)–(d)). In sample HLP-707, spherical chitinozoan spore fossils can be seen in the shale layers, and the sedimentary tuff layers are composed of feldspar, quartz crystal pyroclasts, volcanic dust, and terrestrial fine silt and mud (Fig. 4(b)). The bottom layer ① of sample HLP-708 is mainly shale, and the top layer ② is a typical altered tuff layer about 0.2–0.6 mm thick (Fig. 4(c)). Sample HLP-708 contains a large number of vitric and crystal pyroclasts, and accretionary pellets (AP1 aggregates, Fig. 5) have been found (Yang et al., 2023; Xu et al., 2024). AP1 aggregates are formed by the rapid aggregation of fine volcanic ash driven by hydrogen bonds and electrostatic forces under relatively moist conditions, and they are non-luminescent under cross-polarized light. The diameter of the AP1 aggregates found is usually around 160 μm , indicating a distal source, typically more than 100 km away from the volcanic vent (Yang et al., 2023; Lei et al., 2024), which also suggests that the tuff layers are of airfall origin. Sample HLP-710 mainly develops a large number of feldspar crystal pyroclasts, with frequent interbedding of tuffaceous laminae and shale layers (Fig. 4(d)).

Although the tuff layers in all four sample groups are clearly of sedimentary origin, distinct differences are observed in their structure and material composition. In terms of structure, the tuff in sample HLP-726 has a massive structure, while the tuffaceous components of the other three groups of samples have a laminated structure. In terms of genesis, samples HLP-726, HLP-707, and HLP-708 all contain a large number of vitric pyroclasts and AP1, indicating an airfall origin; whereas the tuff layers in sample HLP-710 mainly develop feldspar crystal pyroclasts, indicating a surface material deposition and transportation origin. The AP2 aggregates

formed within ground-hugging density currents are typically characterized by concentric laminations, which clearly distinguish them from AP1 aggregates (Yang et al., 2023). However, the difficulty in identifying AP2 aggregates in the Chang 7 Member shale is primarily due to a combination of their inherent fragility and intense subsequent diagenetic alteration (Cullers and Podkovyrov, 2000; Liu et al., 2022).

4.2. Comparison of initial diagenetic characteristics

The initial diagenetic parameters for the four samples tested were determined and are presented in Table 2 and Fig. 6. There is a significant difference in the TOC content among the four groups of samples. HLP-726, which is a massive tuff, has the lowest TOC content of 2.83%, while the TOC content of the other three groups of samples ranges from 16.35% to 25.75%. The layers with well-developed tuffaceous laminae generally have higher TOC content. This is due to the fact that the beneficial effect of volcanic activity on the enrichment of organic matter deposition slightly “lags” behind the “explosive” growth of primary productivity caused by volcanic activity on a short time scale (Liu et al., 2023; Zhang et al., 2024). The T_{max} and HI values of each group of samples do not differ much; the S1 and S2 values of sample HLP-726 are lower than those of the other samples. In addition, the type of organic matter in the four groups of samples is Type II (Fig. 6), with a sample R_o of 0.5%, indicating that the samples are in the low maturity stage.

4.3. Characteristics of expelled oil, retained oil, and total generated oil

Shale is highly heterogeneous. In thermal simulation experiments, we control the pyrolysis process by employing a low heating rate to ensure uniform heating. The independent variable R_o is the mean random vitrinite reflectance ($\%R_o$) from the organic matter within the same heated cylinder. Therefore, the independent variable R_o is representative for the tested sample. The total generated oil in the thermal simulation experiment is the sum of expelled oil and retained oil (Cullers and Podkovyrov, 2000; Liu et al., 2022). Both the total generated oil, expelled oil, and retained oil include light hydrocarbon components and heavy hydrocarbon components. The light hydrocarbon components are the C5–C14 fraction, while the heavy hydrocarbon components are the C14+ fraction.

(1) Massive organic-rich tuff

The expelled oil, retained oil, and total generated oil characteristics of the massive organic-rich tuff sample HLP-726 are

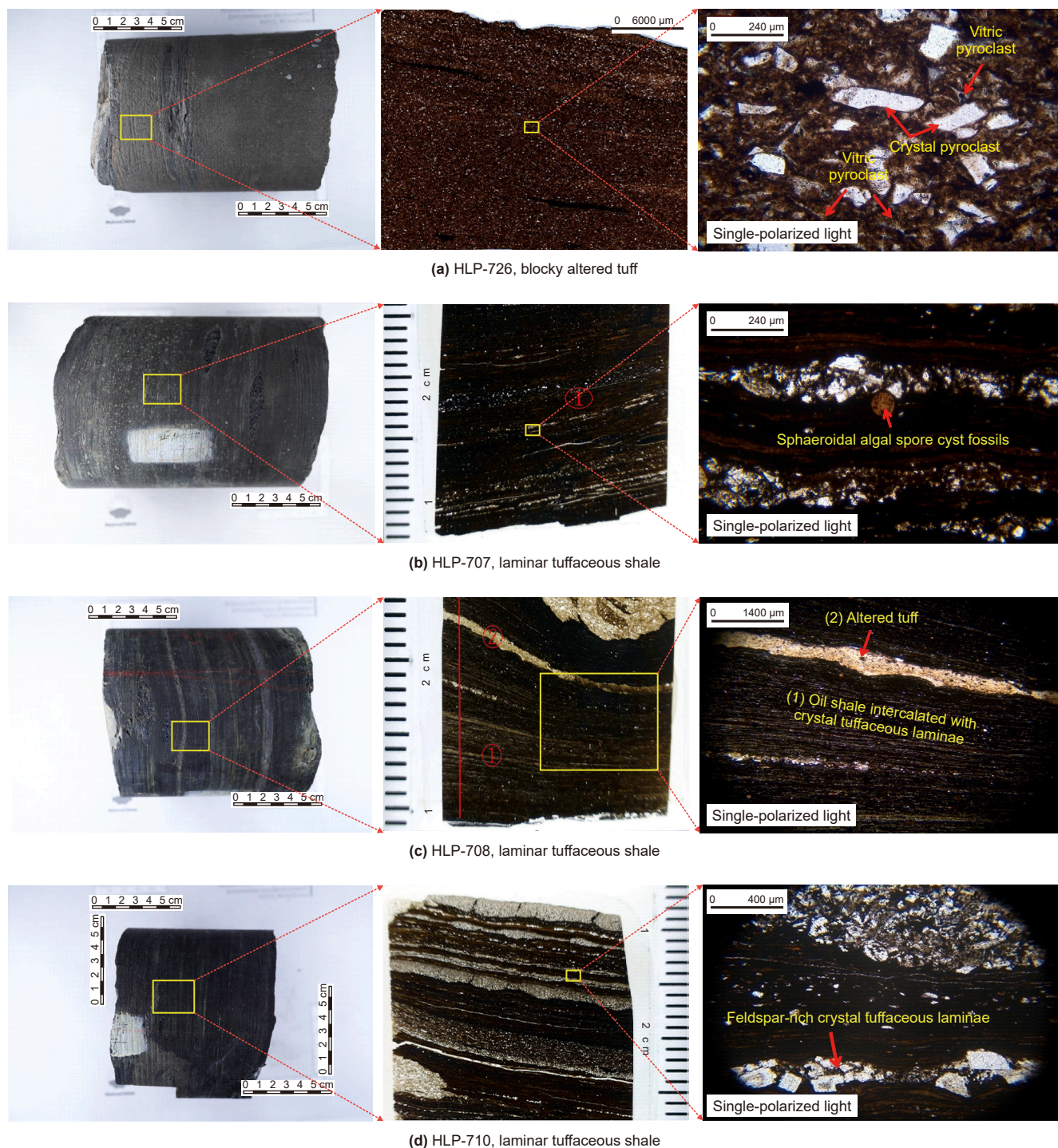


Fig. 4. Core and thin section images from four sets of samples taken from the bottom of the Chang 7 Member in the study area, Well Z40. (a) HLP-726, blocky altered tuff. (b) HLP-707, laminar tuffaceous shale. (c) HLP-708, laminar tuffaceous shale. (d) HLP-710, laminar tuffaceous shale.

shown in Fig. 7. It can be observed that as the R_0 increases, the amount of expelled oil continuously increases (Fig. 7(a)), while the amount of retained oil continuously decreases (Fig. 7(b)). For the expelled oil, it begins to steadily increase from the initial point of R_0 0.7% and reaches a stable state or peak value at R_0 1.1%. For the retained oil, the most significant reduction is observed in the C14+ fraction as R_0 increases from 0.7% to 1.1%, while the reduction in the C5–C14 fraction is not pronounced (Fig. 7(b)). The total generated oil increases with increasing R_0 from 0.7% to 1.1%,

peaking near $R_0 = 1.1\%$, before decreasing as R_0 further increases to 1.3% (Fig. 7(c)).

(2) Laminar tuffaceous shale

The expelled oil, retained oil, and total generated oil characteristics of the laminated tuffaceous shale samples (HLP-707, HLP-708, HLP-710) are depicted in Fig. 8. It is evident that the oil generation features of the three sets of samples are similar. As the

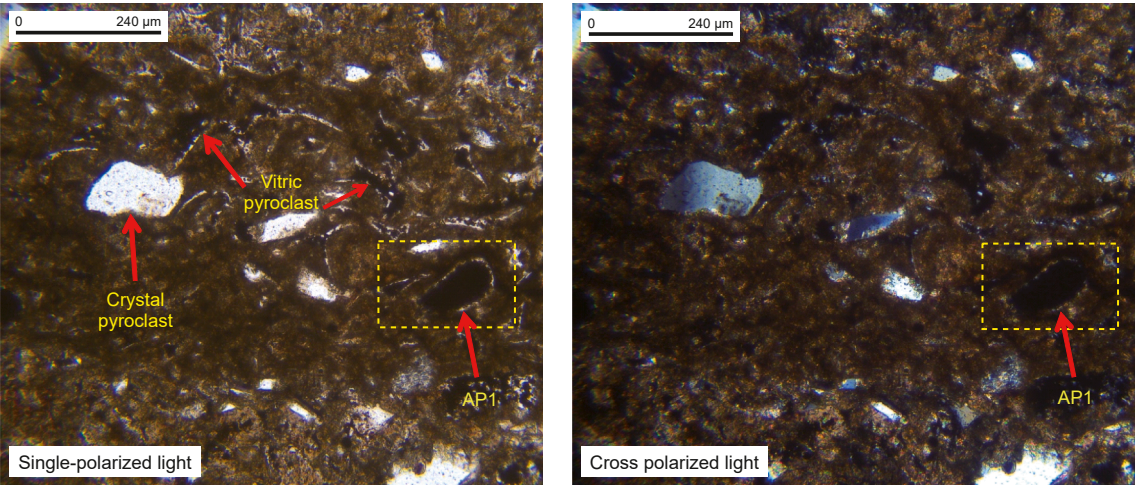


Fig. 5. Microscopic photograph of HLP-710 sample, Chang 7 Member, altered tuff layer, poor sorting, matrix-supported.

Table 2
Original sample Rock-Eval/TOC data table.

Sample	Lithology	S1, mg HC/g rock	S2, mg HC/g rock	S3, mg CO ₂ /g rock	T _{max} , °C	TOC, %	HI, mg/g TOC
HLP-726	altered tuff (massive)	3.29	10.02	0.46	439	2.83	354.06
HLP-707	altered tuffaceous shale (lamellar)	6.01	103.02	0.45	438	25.75	400.08
HLP-708	altered tuffaceous shale (lamellar)	5.60	80.08	0.53	436	22.30	359.10
HLP-710	altered sedimentary tuffaceous tuff (lamellar)	3.94	63.39	0.33	439	16.35	387.71

Notes: S1 represents free hydrocarbons, S2 represents pyrolytic hydrocarbons, S3 represents CO₂ produced during pyrolysis, T_{max} represents the temperature at which the maximum pyrolysis rate is reached during the pyrolysis process, HI is the hydrogen index of the organic matter.

R₀ increases, the expelled oil amount initially shows a lower degree of increase, and then rapidly rises after R₀ exceeds 0.9% (Fig. 8(a1), (b1) and (c1)); the C5–C14 fraction sharply increases after 1.1% R₀. The retained oil all exhibit a trend of increasing first

and then decreasing, with the peak value occurring between 1.0 and 1.1% R₀ (Fig. 8(a2), (b2) and (c2)). Regarding the total generated oil, it gradually increases from R₀ 0.7%–1.1%, reaching the maximum value at R₀ 1.1%, and then experiences a certain degree of reduction (Fig. 8(a3), (b3) and (c3)). The total generated oil reaches its maximum value near 1.1% R₀.

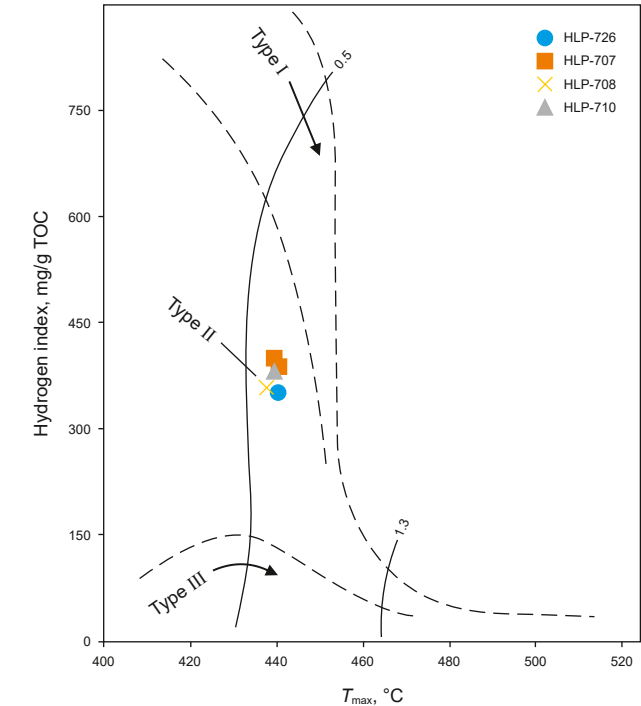


Fig. 6. Hydrogen index–maximum temperature (HI–T_{max}) plot of the tested samples.

4.4. Hydrocarbon gases, non-hydrocarbon gases

The hydrocarbon gases in thermal simulation originate from the pyrolysis of kerogen, oil cracking, kerogen condensation, and aromatization reactions (Qi et al., 2019; Loucks et al., 2012). Pyrolysis of kerogen forms various hydrocarbons and non-hydrocarbon substances. Oil cracking forms heavy hydrocarbons (C6–C14), light hydrocarbons (C2–C5, C1), and asphaltene materials. The cracking of heavy hydrocarbon gases primarily yields light gaseous alkanes (C2–C5, C1). Fig. 9 shows the characteristics of hydrocarbon gas and non-hydrocarbon gas generation in thermal simulation samples. It can be seen that as R₀ increases, the gas yield of the samples continues to increase. Generally, heavy hydrocarbon gas C5 cracking is intense after 1.3% R₀. Overall, the H₂S yield in laminated tuffaceous shale is greater than that in massive organic-rich tuff, which may be related to the presence of pyrite in the samples. Pyrite is a common sulfide mineral, widely present in various rocks. Laminated tuffaceous shale may contain more pyrite, which decomposes to generate hydrogen sulfide during the thermal simulation process (Cullers and Podkovyrov, 2000). Laminated tuffaceous shale typically contains more clay minerals and fine-grained detritus, which may be more likely to release sulfides during the thermal simulation process. In contrast, massive organic-rich tuff may contain more vitric material and volcanic detritus, which are less capable of releasing sulfides during the thermal simulation process.

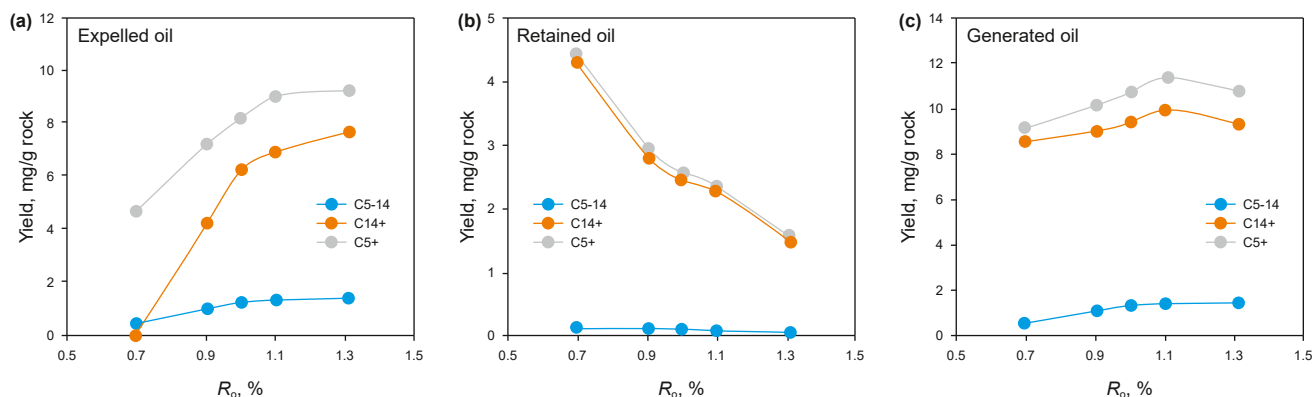


Fig. 7. Characteristics of expelled oil, retained oil, and total generated oil for the HLP-726 sample. The working conditions of the samples are shown in Table 1. (a) Expelled oil, (b) retained oil and (c) generated oil.

Although non-hydrocarbon gases were detected and recorded during the experiments, their specific influence on hydrocarbon generation pathways was not the core focus of this study. We fully recognize that these gases may affect hydrocarbon yield and composition by modifying redox conditions, catalyzing certain cracking reactions, or participating in water–rock interactions. However, addressing these mechanisms requires dedicated experimental designs beyond the scope of this work. In subsequent studies, we intend to apply more precise control-variable experimental protocols—for example, by introducing specific gas components into closed-system pyrolysis setups—to systematically assess the impact of non-hydrocarbon gases on kerogen degradation and hydrocarbon generation kinetics. The specific effects of non-hydrocarbon gases on thermal simulation reaction pathways will be a key emphasis of future in-depth research.

5. Discussion

5.1. Changes in hydrocarbon production and differences in hydrocarbon generation intensity

This thermal simulation experiment enables the collection and quantification of light hydrocarbon components. Fig. 10 presents the chromatogram of expelled oil from the massive tuff sample HLP-726, dominated by *n*-alkanes, with pristane and phytane eluting immediately after C17 and C18, respectively. Similarly, Fig. 11 shows the chromatogram profile of expelled oil from the laminated altered tuffaceous shale HLP-708. Notably, in both samples, the C5–C14 components increase gradually with rising R_o , exhibiting a sharp increase after R_o reaches 1.1%, which indicates enhanced light hydrocarbon generation within this maturity window (Shi et al., 2023; Yang et al., 2023; Zou et al., 2023a).

The observed increase in light hydrocarbons (C5–C14) around $R_o = 1.1\%$ can be attributed to the thermal cracking of heavier hydrocarbon compounds and the decomposition of kerogen side chains. This maturity threshold represents a critical phase in the oil window where the rate of thermal degradation of C14+ compounds and polar components accelerates significantly, leading to enhanced generation of lighter fractions. The consistent behavior in both lithologies suggests that this phenomenon is primarily controlled by organic matter maturation rather than lithological differences (Zhang et al., 2009).

Fig. 12 illustrates the variations in expelled oil, retained oil, generated oil, and gas–oil ratio across different R_o values. A key observation is the distinct hydrocarbon generation–expulsion behaviors between massive organic-rich tuff and laminated

tuffaceous shale. Specifically, the massive tuff shows rapid expulsion of C5–C14 starting at $R_o = 0.7\%$, peaking at $R_o = 1.1\%$ (Fig. 12(a)), while C14+ compounds begin cracking at $R_o = 0.7\%$ and stabilize by $R_o = 1.1\%$. In contrast, the laminated tuffaceous shale exhibits a rapid increase in expelled oil after $R_o = 0.9\%$, with C5–C14 fractions rising markedly after $R_o = 1.1\%$. Retained oil reaches its maximum between $R_o = 1.0\%$ – 1.1% (Fig. 12(b)), and total generated oil peaks at $R_o = 1.1\%$ (Fig. 12(c)).

The earlier expulsion onset in massive tuff ($R_o = 0.7\%$) compared to laminated shale ($R_o = 0.9\%$) reflects fundamental differences in pore network connectivity and permeability anisotropy. Massive tuff typically exhibits well-connected inter-granular pores and microfractures that facilitate efficient hydrocarbon migration, while the laminated fabric of tuffaceous shale creates anisotropic flow pathways that retard expulsion until higher maturity stages (Yin et al., 2024). This lithology-dependent expulsion behavior has significant implications for predicting shale oil mobility and producibility in heterogeneous formations (Dang et al., 2022).

The total hydrocarbon yield in this experiment exceeds the original S1+S2 values from pyrolysis (Table 2), which we attribute to inhomogeneous distribution of residual oil within the sample column after heating. Oil migration toward the periphery and bottom resulted in higher oil content in the lower section, from which the ≈ 1000 mg subsample was taken—leading to measured yields higher than the whole-column average. Furthermore, the increasing gas–oil ratio with rising R_o (Fig. 12(d)) confirms ongoing cracking of crude oil into lighter components at higher maturities. The progressive increase in gas–oil ratio with maturation reflects the compositional evolution of generated hydrocarbons through secondary cracking reactions. At $R_o > 1.1\%$, the observed shift toward lighter products indicates the onset of condensate generation and gas formation through cracking of C14+ compounds and residual bitumen. This compositional evolution follows established reaction kinetics principles where C–C bond cleavage becomes increasingly favored at higher thermal stress (Lv et al., 2022).

Collectively, these results highlight notable differences in the evolution of retained oil: laminated tuffaceous shale begins to show decreased retention at approximately $R_o = 1.05\%$, whereas the massive tuff experiences a decline starting at $R_o = 0.7\%$ (Fig. 13). This suggests that the organic matter in massive tuff undergoes more advanced thermal evolution under the same conditions, which may reflect its distinct petrophysical properties and expulsion pathways. The differential retention behavior between lithologies can be explained by variations in adsorption capacity and pore structure (Wang et al., 2021). Laminated

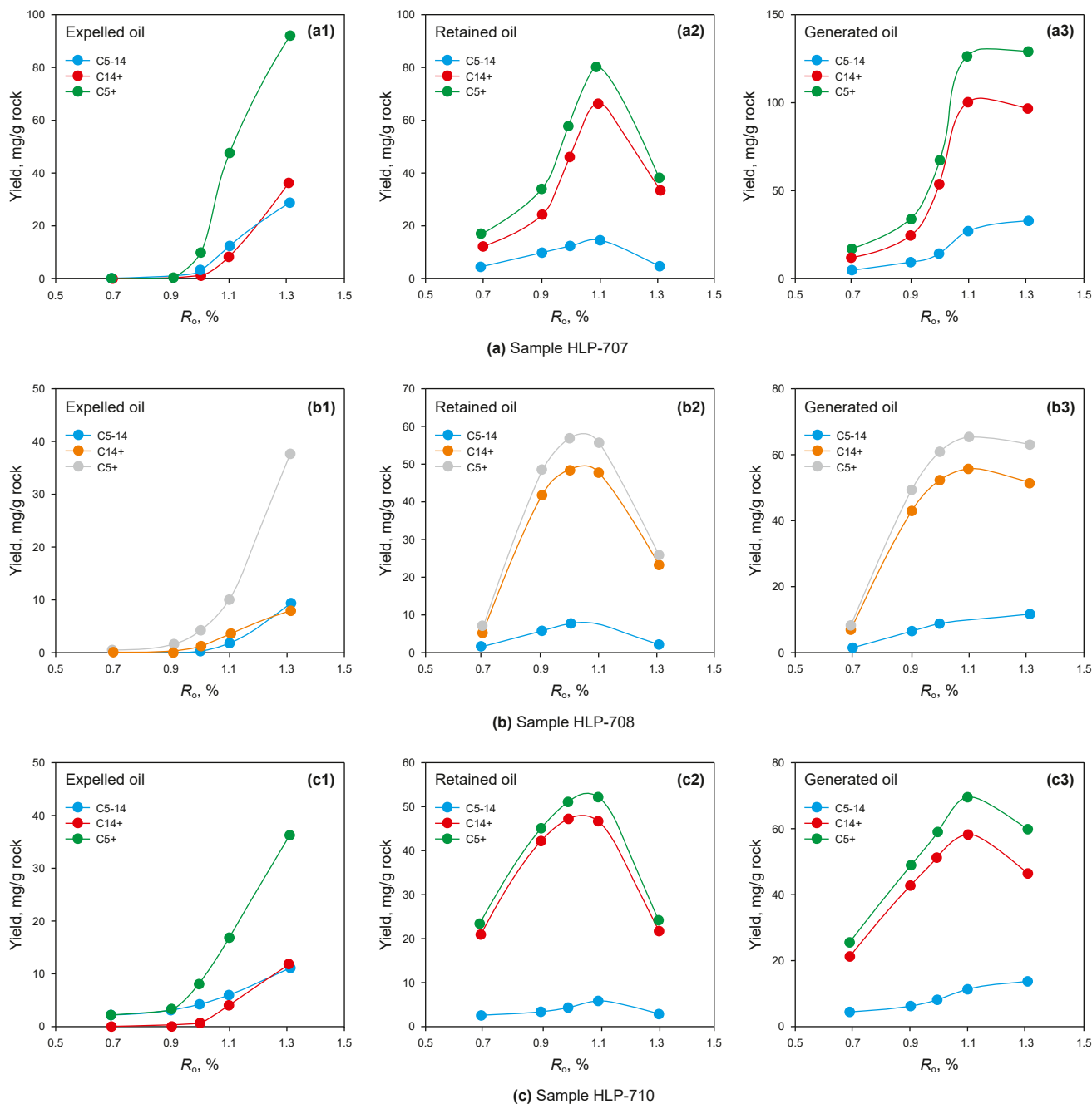


Fig. 8. Characteristics of expelled oil, retained oil, and total generated oil for the lamellar tuffaceous shale samples. **(a)** Sample HLP-707, **(b)** sample HLP-708 and **(c)** sample HLP-710.

tuffaceous shale, with its higher clay content and developed organic matter-hosted nanopores, provides greater adsorption surface area and capillary trapping effects, thereby retaining hydrocarbons until higher maturity stages (Li et al., 2022; Yin et al., 2025). In contrast, the more rigid framework and larger pore throats in massive tuff promote earlier and more efficient hydrocarbon expulsion.

These findings reinforce the central argument that tuffaceous lithologies exert a critical influence on hydrocarbon generation and retention patterns, supporting the thesis that volcanic-derived materials play a unique role in shale oil accumulation processes (Zhang et al., 2009, 2017; Zou et al., 2023b).

The mechanistic understanding provided by these experiments highlights the importance of lithology-specific kinetic models for

predicting hydrocarbon generation and expulsion in tuff-rich sequences. The distinct behaviors observed between massive and laminated tuffaceous rocks should be incorporated into basin models to improve accuracy in resource assessment and exploration targeting in volcanic-influenced lacustrine basins (Zheng et al., 2011; Zhang et al., 2022; Zou et al., 2023a).

5.2. Comparison of hydrocarbon expulsion efficiency

Hydrocarbon expulsion efficiency refers to the efficiency with which organic matter generates and expels oil and gas during thermal evolution (Zhang et al., 2009, 2017). The level of hydrocarbon expulsion efficiency significantly affects the accumulation of unconventional oil and gas. Considering both the efficiency of

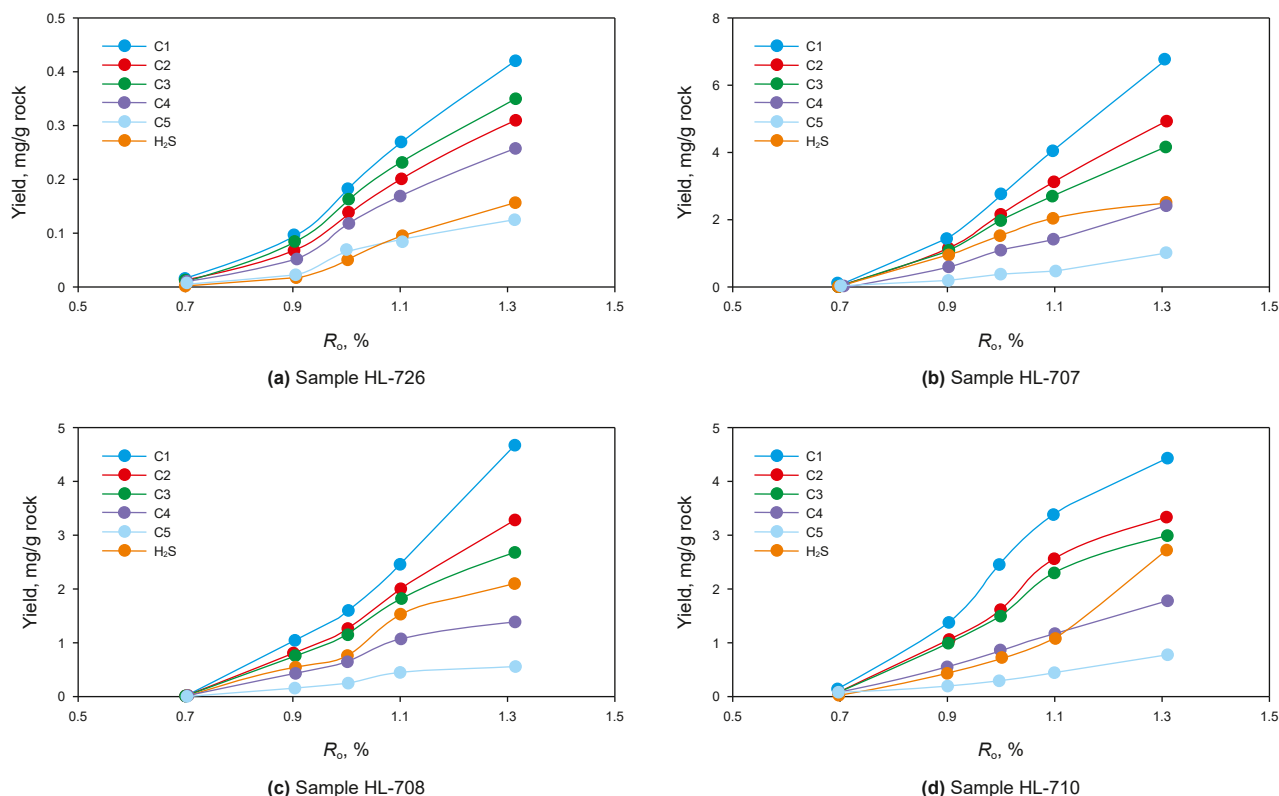


Fig. 9. Characteristics of hydrocarbon gas and non-hydrocarbon gas generation in thermal simulation samples. (a) Sample HLP-726, (b) Sample HLP-707, (c) sample HLP-708 and (d) sample HLP-710.

the experiment and the degree of sample reaction, this experiment employs a constant temperature thermal simulation method at 370 °C. This temperature is higher than the actual expulsion temperature in the strata, and as the thermal simulation progresses, heavy oil will transform into light oil, the viscosity of the petroleum will decrease, leading to increased mobility (Lv et al., 2022). Consequently, at higher R_o experimental points, the experimental results will show a higher expulsion efficiency than the actual expulsion efficiency in the strata. Furthermore, as oil and gas transform towards lighter qualities, the overall trend is an increase in the hydrogen-to-carbon ratio. Therefore, at the final experimental point, the total hydrocarbon content measured by

weight will be somewhat reduced (Wang et al., 2023; Yang et al., 2023).

Fig. 14 shows the characteristics of the change in expulsion efficiency of the four groups of samples under different R_o conditions. As R_o increases, the expulsion efficiency of the samples gradually increases. The expulsion efficiency of the massive organic-rich tuff is significantly higher than that of the laminar tuffaceous shale (Fig. 14). Further calculations of the S1/TOC values for each group of samples are presented in Table 3. It can be observed that the massive organic-rich tuff has a higher S1/TOC value compared to the laminar tuffaceous shale, with a value greater than 100, indicating it also has a high-quality reservoir

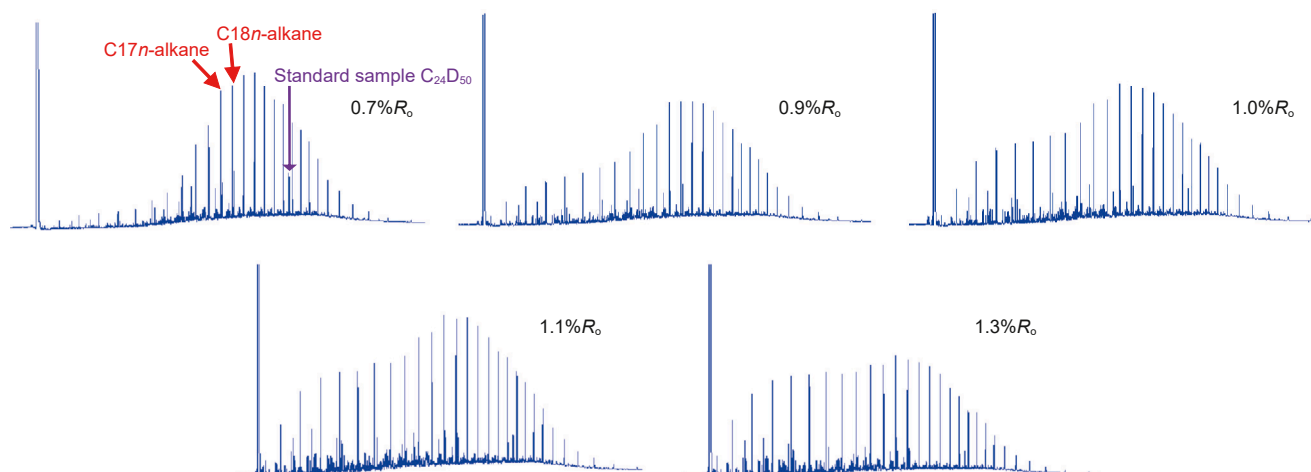


Fig. 10. Chromatographic characteristics of the expelled oil from the massive altered tuff (HLP-726).

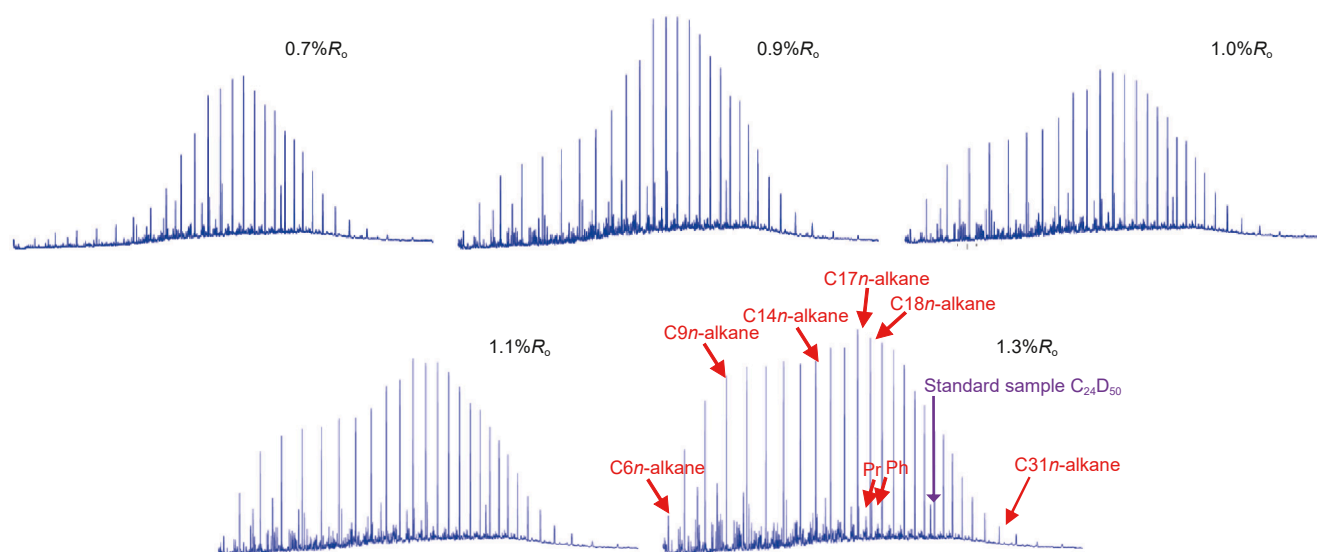


Fig. 11. Chromatographic characteristics of the expelled oil from the laminar altered tuffaceous shale (HLP-707).

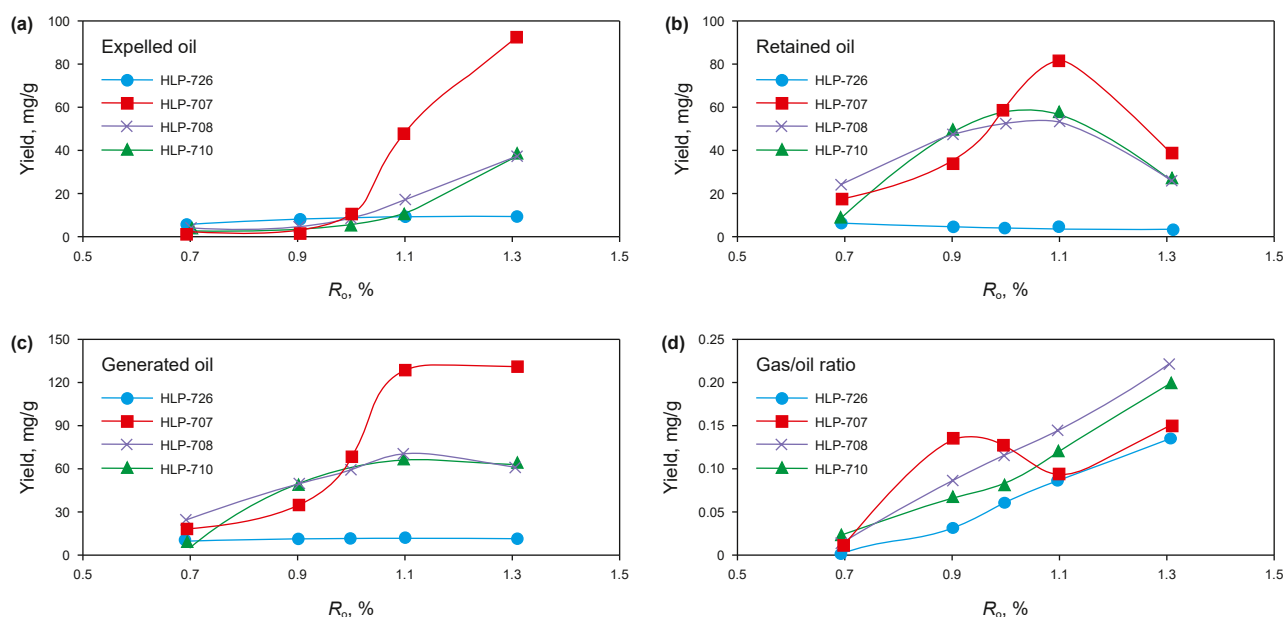


Fig. 12. Characteristics of changes in expelled oil, retained oil, generated oil, and oil-gas ratio under different R_o conditions for the samples. (a) Expelled oil; (b) retained oil; (c) generated oil; (d) gas/oil ratio.

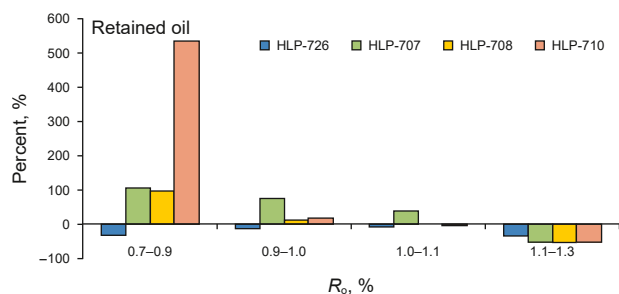


Fig. 13. Characteristics of changes in retained oil content during different stages of thermal evolution.

property. The expulsion efficiency of the massive tuff samples is not only higher than that of the laminar tuffaceous shale, but also has a larger initial expulsion efficiency, thus being capable of accepting oil influx from other nearby source rocks. Additionally, the hydrocarbon generation potential of the massive organic-rich tuff may not only be contributed by some kerogen but also from some heavy oil components. For the three groups of laminated tuffaceous shales, the expulsion efficiency increases with the increase in TOC content (Fig. 14). The higher the TOC content, the greater the hydrocarbon generation potential and expulsion efficiency.

The positive correlation between TOC content and expulsion efficiency in laminated shales reflects the crucial role of organic matter not only as hydrocarbon source but also as pore space contributor. Higher TOC content enhances both hydrocarbon

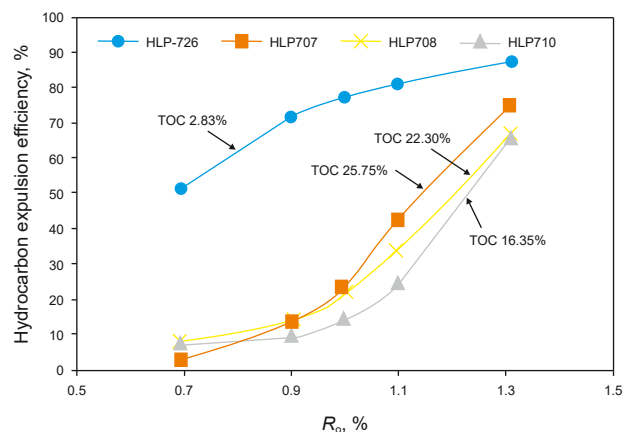


Fig. 14. Changes in hydrocarbon expulsion efficiency of the samples under different R_o conditions. Notes: Hydrocarbon expulsion efficiency is the ratio of the total amount of generated hydrocarbon gases and expelled oil to the total generated hydrocarbons.

generation potential and the development of organic-hosted pores, creating better connected pore networks that improve expulsion efficiency (Qi et al., 2019; Liu et al., 2021). However, this relationship exhibits a threshold effect—beyond a certain TOC content, the increased adsorption capacity and oil retention in organic pores may counterbalance the enhanced connectivity, leading to nonlinear changes in expulsion efficiency. This complex interplay between generation, retention, and expulsion mechanisms underscores the necessity of lithology-specific evaluation criteria for predicting shale oil mobility and producibility.

From a practical perspective, the higher S1/TOC ratios (>100) observed in massive tuff indicate superior fluid storage capacity and better pore connectivity, making such intervals potential targets for shale oil exploration. These geochemical characteristics, combined with the rock's higher brittleness, suggest that massive tuff intervals may respond more favorably to hydraulic stimulation techniques. The understanding of these lithology-controlled expulsion mechanisms provides critical insights for optimizing completion strategies and production forecasting in heterogeneous tuffaceous shale reservoirs (Qi et al., 2019).

5.3. Mechanisms influencing differences in hydrocarbon expulsion efficiency

Research indicates that massive organic-rich tuff exhibits higher hydrocarbon expulsion efficiency. This observation can be attributed to its unique geological structure and chemical composition. First, massive organic-rich tuff typically contains a higher proportion of volcanic glass and pores, which play a crucial role in hydrocarbon generation and storage (Zhao et al., 2023). Volcanic glass is prone to alteration during diagenesis, forming complex secondary pore networks (Schweitzer et al., 2019). These pore networks not only increase the rock's porosity but also provide multiple pathways for hydrocarbon migration. Additionally, the rigid framework structure of massive tuff effectively resists

compaction (Loucks et al., 2012), maintaining pore connectivity during deep burial, which is essential for sustaining efficient hydrocarbon migration. The stability of this structure allows hydrocarbons to maintain high expulsion efficiency over extended geological periods.

Moreover, massive tuff contains lower levels of clay minerals such as montmorillonite, reducing hydrocarbon adsorption and retention during migration (Xie et al., 2023; Zhang et al., 2025). The adsorption by clay minerals is a significant impediment to hydrocarbon migration, so lower clay content means hydrocarbons move more freely through the pores and are expelled more efficiently. Fig. 15 shows the scanning electron microscopy (SEM) images of sample HLP-726 under different R_o conditions. The original sample of HLP-726 ($R_o = 0.5\%$) exhibits relatively uniform pore development. After heating ($R_o = 0.7\%$), the number of pores in the sample increases, forming numerous slit-shaped and intercrystalline pores. From $R_o = 0.9\%$ to $R_o = 1.3\%$, neither the pore types nor their morphology undergo significant changes, indicating strong thermal stability of the minerals within the sample (Fig. 15). Furthermore, organic matter in massive tuff is dispersed, which not only shortens hydrocarbon migration pathways but also further decreases adsorption retention within the rock (Loucks et al., 2012; Lu et al., 2024; Zhang et al., 2025). The dispersed organic particles provide more generation sites for hydrocarbons, enabling their formation across a broader area and thereby enhancing overall expulsion efficiency.

In stark contrast, the organic matter occurrence characteristics of laminated tuffaceous shale are markedly different (Fig. 16). Sample HLP-708 primarily develops flat plate-shaped pores at $R_o = 0.5\%$ (Fig. 16). After heating ($R_o = 0.7\%$), the number of pores increases, with a significant formation of slit-shaped pores. When R_o reaches 0.9% – 1.0% , the slit-shaped pores further develop, resulting in enhanced pore connectivity and reduced heterogeneity. At $R_o = 1.1\%$, pore collapse occurs due to thermal stress and hydrocarbon generation, leading to deteriorated pore homogeneity. When R_o reaches 1.3% , the pore characteristics show little change compared to the previous stage, and pore homogeneity remains poor. The organic matter is primarily distributed in layers and tightly bound to clay minerals, a structural configuration that forces hydrocarbons to traverse numerous organic-inorganic interfaces during expulsion (Ritter et al., 2015). These complex interfacial interactions increase migration resistance, requiring hydrocarbons to overcome greater obstacles. Research by Loucks et al. (2012) indicates that the development of organic matter pores in shale is closely related to thermal maturity—at low maturity levels ($R_o < 0.6\%$), organic pores are scarcely developed. This suggests that during early geological history, the hydrocarbon generation and migration capacity of laminated tuffaceous shale were severely constrained. However, as thermal maturity increases, organic pores begin to develop extensively, creating more favorable conditions for hydrocarbon generation and migration (Loucks et al., 2012).

Notably, a higher TOC content in laminated tuffaceous shale is associated with an improved expulsion efficiency. This is because the increased organic matter facilitates the formation of a

Table 3
Comparison table of geochemical parameters of test samples.

Sample	Lithology	S1	TOC	S1/TOC
		mg/g	%	mg/g TOC
HLP-726	Altered tuff (massive)	3.29	2.83	116.25
HLP-707	Altered tuffaceous shale (lamellar)	6.01	25.75	23.34
HLP-708	Altered tuffaceous shale (lamellar)	5.60	22.30	25.11
HLP-710	Altered sedimentary tuffaceous tuff (lamellar)	3.94	16.35	24.10

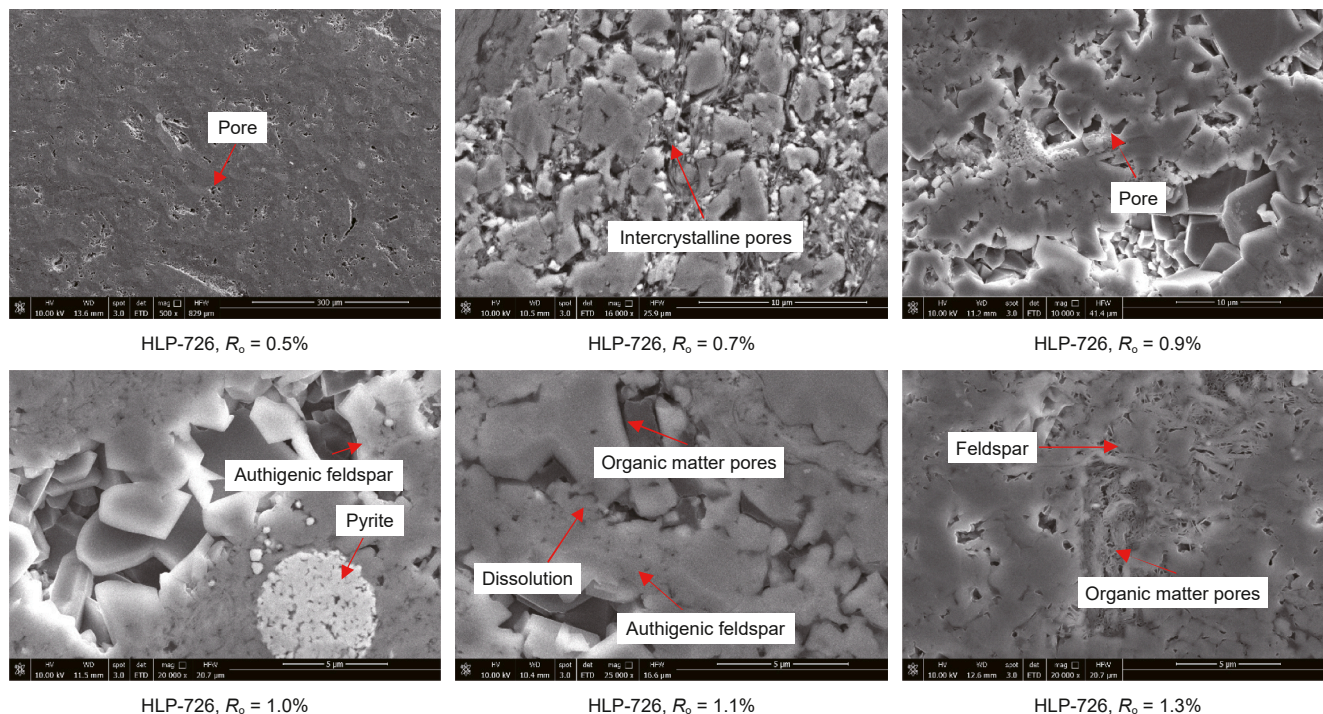


Fig. 15. SEM characteristics of sample HLP-726 (massive altered tuff) under different R_0 conditions during thermal simulation experiments.

continuous organic network, providing pathways for hydrocarbon migration (Ritter et al., 2015). Such a network effectively connects dispersed pores, allowing hydrocarbons to flow more smoothly within the rock. Additionally, high TOC shale is more prone to developing overpressure systems, which can drive hydrocarbon expulsion and further enhance efficiency (Ritter et al., 2015). This effect becomes more pronounced with increasing maturity—once

R_0 exceeds 0.6%, organic pores develop extensively, not only increasing hydrocarbon generation but also optimizing pore structure for more efficient migration. The presence of volcanic material can accelerate the thermal evolution of organic matter, further improving pore structure and significantly boosting hydrocarbon expulsion efficiency (Schweitzer et al., 2019). The thermal and chemical reactivity of volcanic material plays a crucial

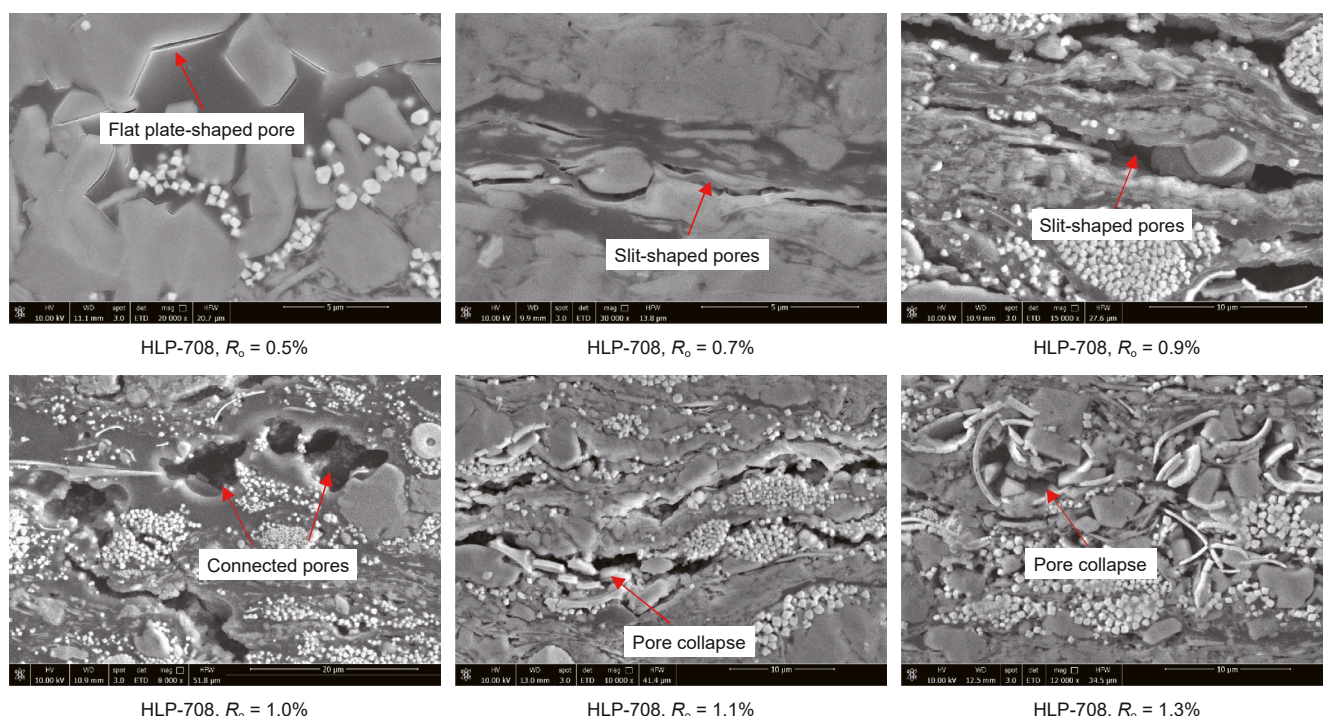


Fig. 16. SEM characteristics of sample HLP-708 (altered tuffaceous shale, lamellar) under different R_0 conditions during thermal simulation experiments.

role in organic matter transformation, enabling hydrocarbons to generate and expel more rapidly.

In summary, the differences in hydrocarbon expulsion efficiency between massive organic-rich tuff and laminated tuffaceous shale primarily stem from variations in pore structure, organic matter distribution, and thermal maturity. The high volcanic glass content, low clay mineral content, and dispersed organic matter in massive tuff provide highly efficient hydrocarbon migration conditions, whereas laminated tuffaceous shale requires higher thermal maturity and TOC content to achieve comparable expulsion efficiency.

The core argument of the text is that the geological architecture of the rock—whether it is a brittle, glass-rich, clay-poor massive tuff with a rigid pore framework, or a compactable, clay-rich laminated shale—is the first-order control. This architecture dictates the pore network's connectivity and stability, which in turn governs the ease with which hydrocarbons can migrate and be expelled (Qi et al., 2019; Liu et al., 2021). All other factors (TOC, maturity) enhance or enable expulsion but operate within the constraints set by this primary geological framework. Therefore, the most controlling factor is the rock type and its resultant pore structure.

Through this study, we have conducted a comprehensive comparison of the differences in hydrocarbon generation intensity and expulsion efficiency between the organic-rich tuff and tuffaceous shale of the Chang 7 Member. The results of the study have certain guiding significance for the exploration and development of unconventional shale oil in the Chang 7 Member.

6. Conclusions

- (1) This study conducted integrated geochemical, petrographic, and hydrocarbon generation thermal simulation experiments on massive organic-rich tuff and laminated tuffaceous shale samples from Well Z40 in the Chang 7 Member. The results indicate that the samples originate from both airfall and water-transported volcanoclastic deposition. Airfall-derived tuffs are characterized by abundant vitric pyroclasts, including accretionary pellets (AP1), while water-transported deposits are dominated by feldspar crystal pyroclasts.
- (2) Significant differences in hydrocarbon generation behavior were identified between massive tuff and laminated tuffaceous shale. The expelled oil from the lamellar tuffaceous shale increases rapidly after 0.9% R_o , and the C5–C14 component increases rapidly after 1.1% R_o ; the retained oil is maximum between 1.0 and 1.1% R_o ; the total generated oil reaches a maximum at 1.1% R_o ; for the massive organic-rich tuff, C5–C14 is rapidly expelled at 0.7% R_o and reaches the highest value at 1.1% R_o ; C14+ begins to crack at 0.7% R_o and reaches stability at 1.1% R_o .
- (3) Massive tuff demonstrates significantly higher hydrocarbon expulsion efficiency compared to laminated shale, with greater initial expulsion capacity. The hydrocarbon potential of massive tuff derives from both kerogen cracking and heavy oil components, while laminated shale shows positive correlation between expulsion efficiency and TOC content. Retention characteristics also differ substantially: retained oil in laminated shale begins declining at approximately $R_o = 1.05\%$, while massive tuff shows decline initiation at $R_o = 0.7\%$, indicating advanced thermal evolution of organic matter in tuffaceous rocks under identical conditions.
- (4) The fundamental controls on expulsion efficiency differences are attributed to contrasting pore network architectures and organic matter distribution patterns. Massive tuff's high volcanic glass content, well-developed interconnected pore system, and rigid framework facilitate

efficient hydrocarbon migration and early expulsion. Conversely, laminated shale's organic-clay complexes and anisotropic flow pathways require higher thermal maturity ($R_o > 0.6\%$) and TOC content to develop continuous organic networks enabling effective expulsion.

- (5) These findings establish that lithological architecture—whether brittle, glass-rich massive tuff or clay-rich laminated shale—constitutes the primary control on hydrocarbon mobility, with thermal maturity and TOC content operating within this geological framework. The superior expulsion characteristics and higher S1/TOC ratios (>100) of massive tuff identify such intervals as priority targets for shale oil exploration and development in the Chang 7 Member.
- (6) This study provides a mechanistic understanding of hydrocarbon generation and expulsion processes in volcanic-influenced lacustrine basins, emphasizing the necessity of lithology-specific evaluation criteria for predicting shale oil producibility and optimizing exploration strategies in heterogeneous tuffaceous sequences.

CRediT authorship contribution statement

Han-Lin Liu: Writing – original draft, Methodology, Investigation. **Shuai Yin:** Methodology, Investigation. **Qun Zhao:** Methodology, Investigation. **Guo-Sheng Zhang:** Investigation, Methodology. **Yu-Jie Li:** Software, Resources, Investigation. **Yan-Peng Chen:** Investigation, Formal analysis. **Zhen Qiu:** Investigation, Formal analysis. **Zhi Yang:** Investigation, Formal analysis. **Song-Tao Wu:** Investigation, Formal analysis. **Shi-Xiang Li:** Formal analysis. **Cai-Neng Zou:** Methodology.

Conflict of interest

There's no conflict of interest of this paper.

Acknowledgements

This study was supported by the National Natural Science Foundation of China (Nos. 42222209, 72140005 and 42302167), the China Petroleum Exploration and Development Research Institute Project (Nos. R2PED-2023-JS-1447, 2021yj02), the Strategic Research and Consulting Project of the Chinese Academy of Engineering (2024-XZ-88), the State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation (No. CDUT-PLC2025004) and the CNPC Innovation Found (No. 2024DQ02-0109).

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