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Progress and outlook of experimental techniques in lacustrine shale reservoir characterization

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Shale reservoirs, as a significant category of unconventional oil and gas resources, possess substantial potential. Compared to marine shale reservoirs, lacustrine shale reservoirs are characterized by pronounced heterogeneity, complex pore structures, and limited seepage capability. Precise characterization of these reservoirs and elucidation of the mechanisms of oil and gas enrichment within micron- to nano-scale pore systems are critical challenges. Recent advancements in experimental techniques, such as microscopic computed tomography (CT), scanning electron microscopy (SEM), focused ion beam scanning electron microscopy (FIB-SEM), and energy-dispersive X-ray spectroscopy (EDS), along with the rapid development of large-view imaging and micron- to nano-scale structure modeling, have considerably enhanced the characterization of lacustrine shale reservoirs. These technological improvements facilitate high-precision identification of mineral compositions and structures, detailed modeling of micron- to nano-scale pores and fractures, and improved characterization and prediction of natural fractures and fluid occurrence characteristics. This paper aims to provide a systematic review of the experimental technological advances in characterizing lacustrine shale reservoirs, identify the primary challenges, and propose a perspective on future development trends. It seeks to offer a reference for refining high-precision characterization and evaluation techniques for lacustrine shale reservoirs, thus supporting more effective and sustainable resource exploitation.

KEYWORDS

lacustrine shale, characterization, experimental techniques, mineral identification, pore structure, organic matter, basic physical property, fracturability

1 Introduction

As technology advances and the global energy landscape evolves, the exploration and development of shale oil and gas have transitioned from experimental efforts to commercial production, becoming a key strategic resource for numerous countries. Currently, global activities in shale oil and gas exploration and development are rapidly advancing, playing a pivotal role in the global oil and gas supply system. Inspired by the success in North America, China has recognized the resource potential and strategic significance of lacustrine shale oil and gas. Extensive exploration efforts have been undertaken, addressing key challenges and achieving significant breakthroughs in shale oil within organic-rich formations in the Junggar Basin, Bohai Bay Basin, and Songliao Basin. Similarly, notable advancements in shale gas exploration have been recorded in the Ordos Basin, Junggar Basin, and Songliao Basin, suggesting a promising future for the development of lacustrine shale oil and gas (Hu et al., 2020; Wang et al., 2022; Zhao X. et al., 2022). Scholars have extensively studied fundamental issues such as resource potential (Zou et al., 2021), primary classifications (Jin et al., 2023), and enrichment mechanisms of lacustrine shale (Jin et al., 2021). They have proposed critical technologies for the exploration and development of lacustrine shale oil and gas (Yuan et al., 2023; Zhao et al., 2023), thus establishing a robust scientific foundation for the efficient development and utilization of these resources.

Lacustrine shale oil and gas, a significant new resource in the oil and gas sector, still presents numerous unresolved issues in petroleum geology and engineering development (Zou et al., 2013). Shale reservoirs are characterized by unique features such as source-reservoir integration, self-generation, self-storage, and self-containment. Unlike conventional oil and gas reservoirs, shale reservoirs have finer grain sizes, more complex mineral compositions (Ross and Bustin, 2009), and smaller pore-throat systems (Loucks et al., 2009; Slatt and O'Brien, 2011), along with a substantial amount of organic matter. The development of shale reservoirs is affected by the interaction between inorganic mineral formation and organic hydrocarbon evolution, with hydrocarbon enrichment driven by the generation, migration, and retention of organic matter. Consequently, the mechanisms of pore formation and retention (Fishman et al., 2012), hydrocarbon accumulation (Chalmers and Bustin, 2007; Milliken et al., 2013), and the assessment of resource scale and recoverability in shale reservoirs significantly differ from those in conventional reservoirs. Furthermore, creating representative shale samples poses challenges, and traditional reservoir characterization and experimental evaluation methods often fail to meet the requirements for accurate industrial assessment of shale oil and gas (Kuang et al., 2021). In recent years, researchers have made significant strides in characterizing lacustrine shale reservoirs, particularly in accurately evaluating shale reservoir parameters from a laboratory perspective. This paper reviews the advancements in experimental techniques for lacustrine shale reservoir characterization and aims to identify potential challenges and future directions.

2 Current status of research on the characterization of lacustrine shale reservoirs

To provide a comprehensive and accurate understanding of the technological advancements and trends in characterizing lacustrine shale reservoirs, this paper employs bibliometric methods to analyze and statistically evaluate core documents published in recent years. Bibliometrics is extensively utilized to assess and quantify indicators from a significant body of academic literature, thereby aiding scholars in identifying and monitoring emerging research trends and critical issues within the field (Ninkov et al., 2022). This study used 'lacustrine shale' and 'characterization' as search terms to conduct a focused review of the related literature. A total of 262 articles were identified, employing software tools such as VOSviewer and Biblioshiny to analyze publishing trends and explore the evolution and interrelationships among research topics.

Based on the publication trend of relevant articles (Figure 1), research on the characterization of lacustrine basin shale reservoirs can be divided into three main phases. The initial phase, before 2014, was marked by a limited number of studies. The subsequent phase, from 2014 to 2019, experienced rapid development, and the volume of related research expanded significantly. The current phase, which began in 2019, has entered an accelerated development stage, evidenced by a marked increase in research activity. Overall, the characterization of lacustrine shale reservoirs has experienced a period of prosperous development in recent years. The active investment by China in lacustrine shale reservoirs is identified as the primary driving force behind this trend. Additionally, the metric MeanTCperYear, which measures the total number of publications and average total citations per year, shows two significant growth periods in the number of citations for relevant papers. This metric offers valuable insights into the impact of these publications. Despite these advancements, the overall citation count remains relatively low, indicating significant potential for further expansion within this research domain.

An analysis of the temporal changes in research topics (Figure 2) indicates that during the periods of low-speed and rapid development leading up to 2019, studies predominantly focused on "shale," "pyrolysis," "crude oil," and other subjects closely associated with source rock were prevalent. This sustained interest likely originates from scholars' incomplete understanding of the significance of lacustrine shale as a reservoir. In contrast, the accelerated development phase post-2019 has witnessed a significant increase in research focusing on topics such as "porosity," "pore structure," "adsorption," and "diagenesis," which are all critical to reservoir characterization. Additionally, research efforts are now primarily concentrated in key continental lacustrine basin shale development areas, including the Sichuan Basin, Ordos Basin, and Bohai Bay Basin.

The cluster analysis of prominent research terms in related articles (Figure 3) reveals that "pore structure," "organic matter," "porosity," "permeability," "lithofacies," "thermal maturity," and "geochemistry" constitute the core network of research hotspots, expanding into more specialized areas of investigation. Additionally, an examination of the temporal trends in these clustered terms indicates that the initial research focus was predominantly on geochemistry and sedimentary characteristics. Over time, the scope of research has broadened to include a significant focus on organic matter, thermal maturity, porosity, and fractal characteristics. In recent years, lithofacies and paleoenvironment have become new focal points, reflecting shifts in research priorities and emerging interests in the field.

3 Advancement in experimental techniques of lacustrine shale reservoirs' characterization

Lacustrine shale reservoirs are characterized by rich organic matter content, substantial clay composition, fine grain size, pronounced heterogeneity, extremely low porosity and permeability, nanoscale pore throats, high specific surface

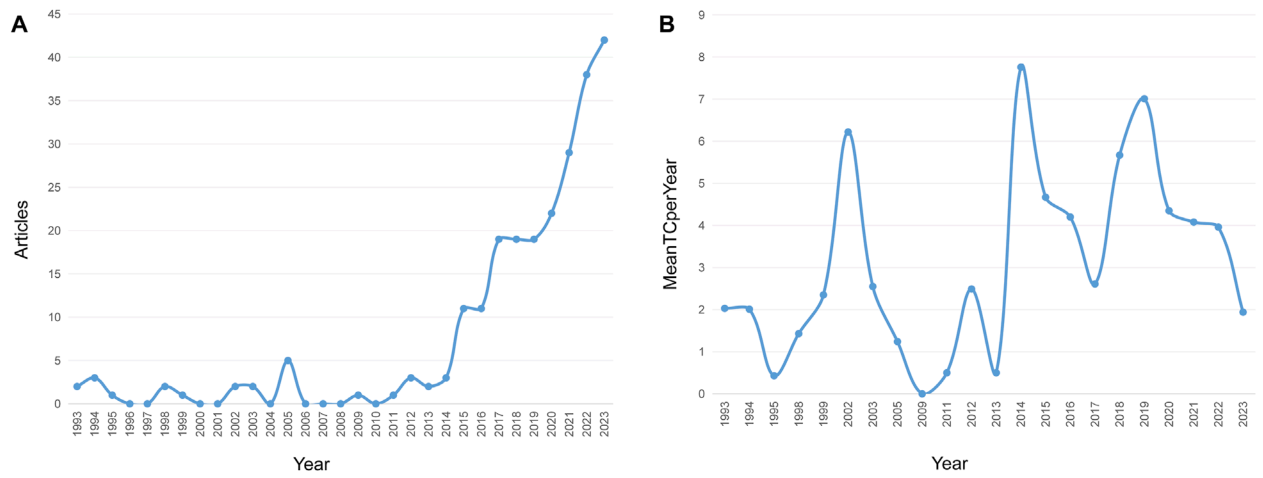

FIGURE 1
Trend in the number of published research papers related to the characterization of lacustrine shale reservoirs **(A)** and the MeanTCperYear trend of relevant research papers **(B)**.

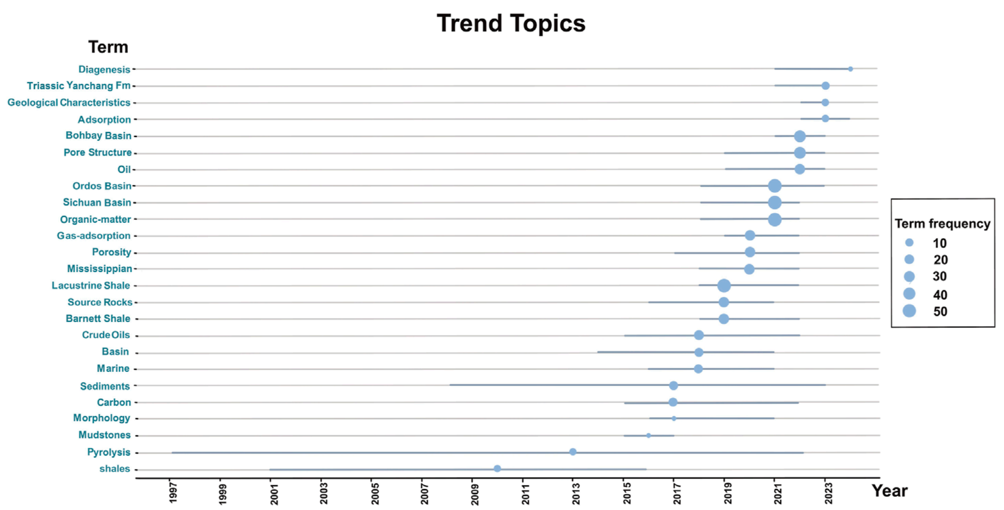

FIGURE 2
Trends in research topics of research papers related to the characterization of lacustrine shale reservoirs.

area, and complex diagenetic alterations coupled with varied hydrocarbon accumulation states (Bechtel et al., 2012; Wang et al., 2019; Zhao R. et al., 2022). These features necessitate distinct evaluation methods compared to conventional reservoirs, involving different content, methods, and techniques (Katz and Lin, 2014). The multi-scale accumulation space, mineral composition, fracture development, and flow production mechanisms in shale are currently focal topics within both academic and industrial spheres, with continuous advancements in experimental technologies driving breakthroughs in related theories and enhancing understanding (Mehmani et al., 2013; Semnani and Borja, 2017). Consequently, shale requires unique evaluation approaches compared to conventional reservoirs. Critical areas of focus include multi-scale storage capacity, mineral composition, fracture development, and flow mechanisms in shale. The growing demand for shale oil and gas exploration and development has propelled rapid advancements in experimental techniques for shale reservoir characterization, thereby establishing a robust foundation for the precise characterization of lacustrine shale reservoirs. Characterization techniques have evolved from optical microscopy to field emission scanning electron microscopy and low-temperature liquid nitrogen adsorption, refining precision from millimeter-micron to nanometer levels (Wu et al., 2018). The application of nano-CT scanning, focused ion beam scanning electron microscopy (FIB-SEM), and synchrotron radiation has transitioned reservoir characterization from two- to three-dimensional, facilitating the selection of optimal drilling sites, refining reservoir enhancement plans, and developing effective strategies (Hemes et al., 2015; Goral et al., 2019). This article aims to summarize and organize advancements in relevant

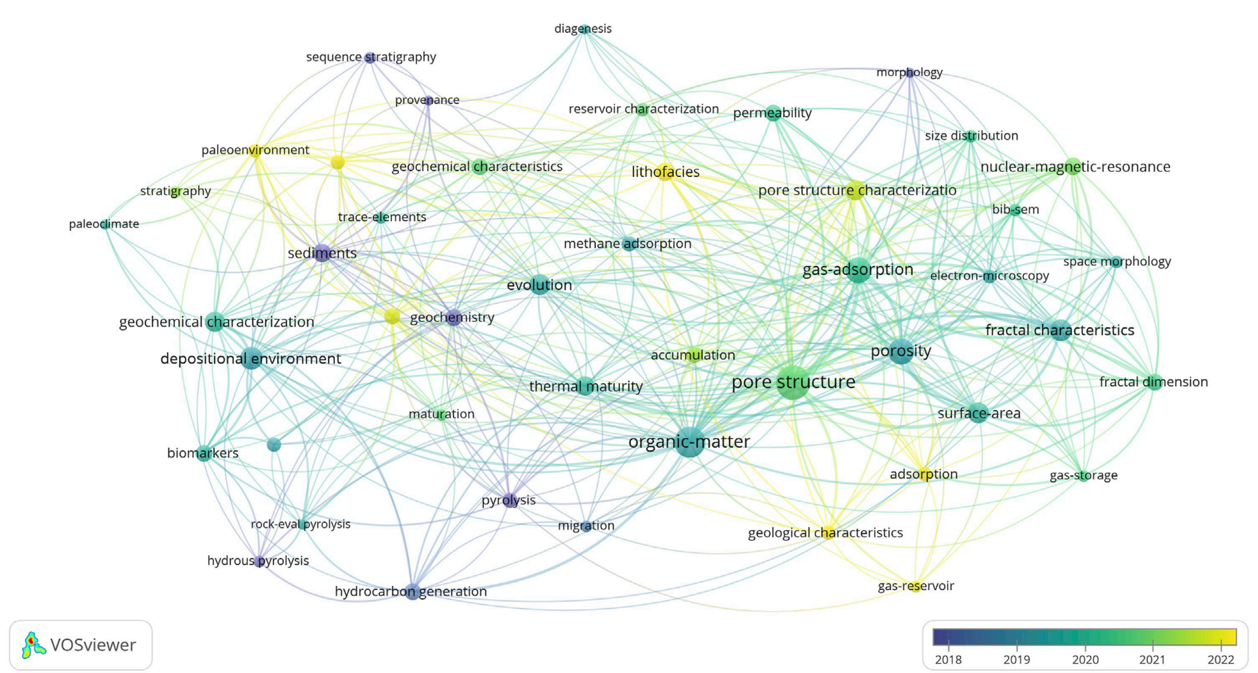

FIGURE 3
Cluster analysis results of research topics in papers related to the characterization of lacustrine shale reservoirs.

experimental technologies across five key aspects, namely, mineral identification, pore structure analysis and modeling, organic matter analysis, fundamental physical properties analysis, and fracturability characterization.

3.1 Mineral identification and quantitative analysis

The mineral composition of shale plays a crucial role in determining the quality of shale reservoirs. Since shale typically consists of tightly packed fine-grained minerals, accurately identifying its mineral composition has become a key research focus (Loucks and Ruppel, 2007; Macquaker et al., 2014). Currently, rock slice identification and X-ray diffraction (XRD) are the most effective methods for the qualitative and quantitative analysis of shale minerals (He et al., 2023). Although rock slice identification offers lower magnification, making it challenging to observe fine minerals such as clay, it still provides critical, rapid information on the shapes, contact relationships, and structural features of mineral particles within the rock. XRD, on the other hand, accurately quantifies the mineral content of shale, detailing the composition and relative proportions of minerals such as quartz, feldspar, clay, and carbonates (Raef et al., 2018).

The integration of scanning electron microscopy (SEM) with other high-resolution imaging technologies is crucial for identifying minerals in shale reservoirs. This approach provides superior resolution compared to earlier methods and allows for non-destructive observation of rock samples. SEM, with its backscattered imaging and large view field stitching method, when combined with X-ray energy dispersive spectroscopy (EDS) and advanced data and image processing from micro-area mineral analysis systems, enables precise nano-level characterization and quantitative analysis of mineral composition, particle size, and morphology (Figure 4). Additionally, cathodoluminescence (CL), which is used in conjunction with electron microscopy, helps determine the origin and formation processes of minerals, which supports the reconstruction of paleogeographic environments (Hackley et al., 2024). Raman spectroscopy, capable of identifying mineral components and assessing diagenetic processes based on crystal orientation, complements SEM in facilitating *in situ* micro-area analysis of mineral composition and rock structure (Hope et al., 2001). The electron microprobe offers a detailed analysis of trace elements in shale, providing essential insights into the chemical properties and formation environments of minerals (Orberger et al., 2003). X-ray fluorescence (XRF) spectroscopy can accurately analyze the elemental composition of minerals and calibrate the oxide proportions of primary elements. The results from these tests support the verification of mineral identification outcomes obtained through XRD (Hupp and Donovan, 2018). Moreover, the use of X-ray fluorescence for *in situ* elemental imaging and laser *in situ* micro-area elemental imaging technology enables accurate characterization and rapid assessment of rock components and structures through extensive scanning (Hua et al., 2022). Fourier-transform infrared spectroscopy (FTIR) is utilized by geologists to analyze both organic and inorganic materials in shale, facilitating a heterogeneous examination of mineral components (Mishra et al., 2018).

The experimental analysis of the mineral matrix effect is also a crucial issue for lacustrine shale reservoir characterization. During the stages of kerogen formation and organic matter preservation, the mineral matrix isolates organic matter via physical adsorption

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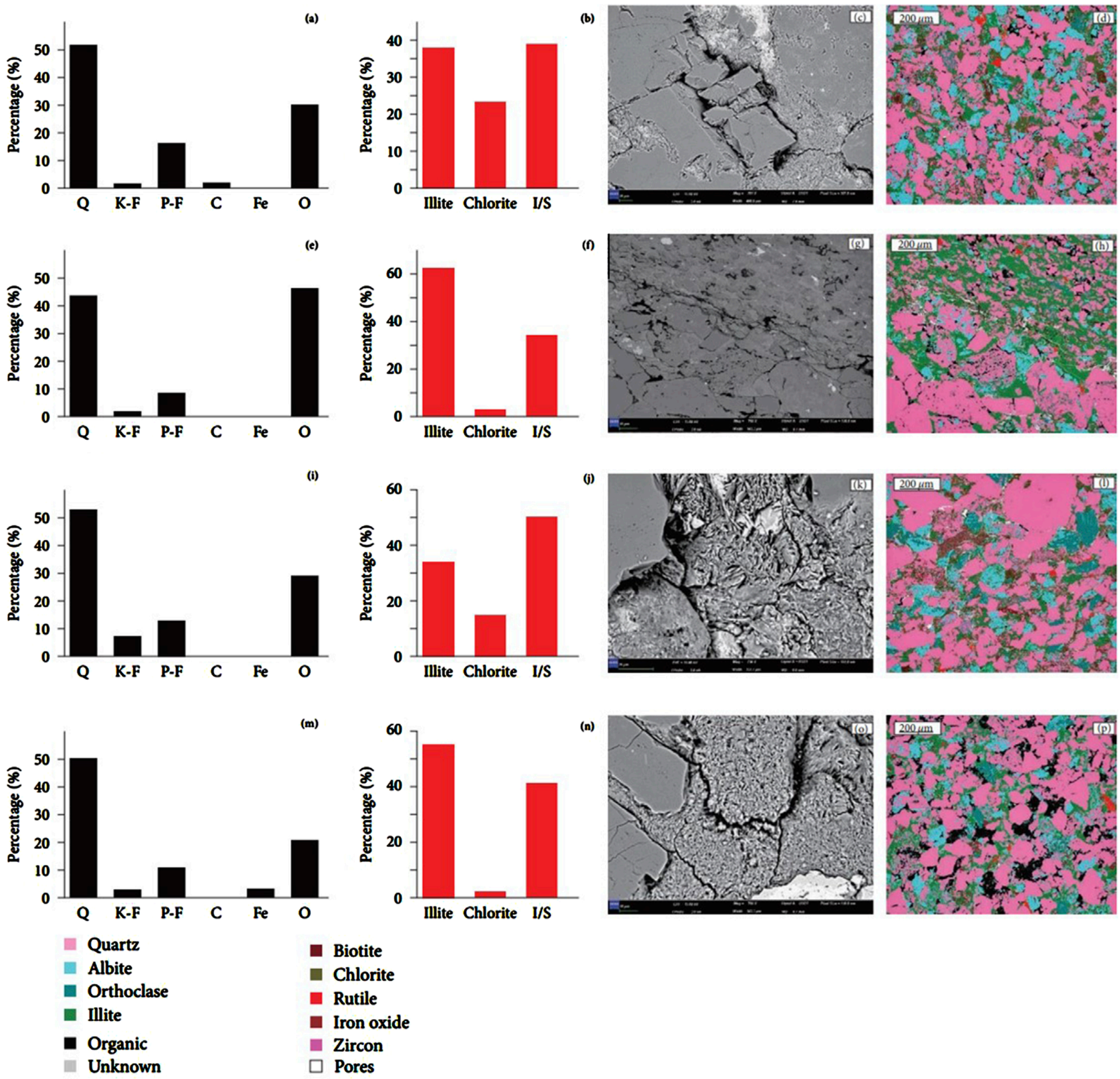

FIGURE 4
Analysis of the mineral composition of unconventional reservoirs using SEM and EDS sample1, **(A–D)**; sample2, **(E–H)**; sample3, **(I–L)**; sample4, **(M–P)**; Q, quartz; K-F, potassium feldspar; P-F, plagioclase feldspar; C, calcite; Fe, siderite; O, others; and I/S, illite/smectite, cited from open access sources (Zhang et al., 2024).

and interlaminar stress, effectively protecting the organic matter. In the stage of organic matter pyrolysis, minerals such as metal oxides and carbonates exhibit catalytic properties, reducing the activation energy required for reactions and accelerating organic matter pyrolysis. The integration of equipment like SEM, TEM, and FTIR with geochemical analysis has elucidated the role of the mineral matrix in preserving organic matter in shale (Salmon et al., 2000). Studies utilizing XRD and FTIR have shown that the mineral matrix not only facilitates the oxidation of kerogen but also lowers the resistance to kerogen oxidation by generating inorganic pores (Yan et al., 2013).

3.2 Pore structure analysis and modeling

The pore structure in shale reservoirs is characterized by multi-scale features, with notable variations in connectivity and wettability across different pore types. These variations result in complex hydrocarbon flow patterns that deviate from those observed in traditional oil and gas migration (Chalmers et al., 2012). The micro–nano scale pore system plays a pivotal role in hydrocarbon accumulation within shale reservoirs, rendering its systematic characterization essential for the accurate assessment of shale oil (Ross and Bustin, 2009; Fink et al., 2018). As high-precision instruments increasingly pervade this field, the methods for characterizing shale pore structures are evolving toward comprehensive aperture coverage, quantification, and digitization, effectively creating a holistic evaluation system that addresses all scales, apertures, and dimensions. In recent years, the utilization of high-precision equipment such as micro-CT, SEM, FIB-SEM, atomic force microscopy (AFM), transmission electron microscopy (TEM), helium ion microscopy (HIM), and small-angle X-ray scattering (SAXS) has expanded from material research to characterize the microscopic pores of shale (Klaver et al., 2015; Liu W. et al., 2016).

Depending on the characterization dimensions, these methods can be classified into two categories: two-dimensional and three-dimensional characterization (Hou L. et al., 2023). For two-dimensional characterization, the combination of argon ion polishing and FE-SEM serves as the primary experimental method. This approach employs large-area stitching technology to produce high-precision images, achieving exceptional resolution and enabling comprehensive characterization across the entire sample, thereby minimizing the effects of reservoir heterogeneity on the results (Curtis et al., 2012; Wen et al., 2020). If there are stringent requirements for the accuracy of pore structure observations or if the conductivity of the sample fails to meet experimental standards, HIM and AFM can provide superior results. HIM can achieve resolutions exceeding five times that of SEM without requiring conductive treatment of the sample (Wang et al., 2020). Furthermore, atomic force microscopy can characterize ultra-small pores and assess the local elastic modulus of the samples (Javadpour et al., 2012). However, these two devices are relatively more expensive. Compared with two-dimensional methods that primarily utilize electron microscopy, three-dimensional characterization techniques offer a greater diversity of methods and cover a broader research scale. Micro-CT enables the three-dimensional characterization of pore structures across varying scales, achieving resolutions as fine as below 1 micron, making it a commonly used digital core technology (Garum et al., 2020). Utilizing its ability to achieve nanometer-level resolution, FIB can perform three-dimensional slice imaging of designated areas through nano-etching methods. This capability facilitates the creation of three-dimensional models from two-dimensional observations, aiding in the differentiation of organic and inorganic pores and enabling nanoscale three-dimensional characterization (Kelly et al., 2016). Additionally, FIB can be employed for TEM sample preparation, allowing for the precise characterization of sub-nanometer pores (Schulz et al., 2015). SAXS offers several advantages, including a wide measurement range from 0.5 nm to 20 μm, the ability to reflect pore connectivity, rapid detection, and non-destructive testing of samples, providing researchers with essential data on the specific surface area, pore size distribution (PSD), structural dimensions, and interface properties (Leu et al., 2016). Furthermore, some researchers have utilized SANS/USANS technology to evaluate the connectivity and heterogeneity of pores in shale (Chandra et al., 2020).

Each technique possesses distinct advantages and limitations: micro-CT scanning facilitates non-destructive sample analysis but offers relatively lower resolution, whereas FIB-SEM provides higher resolution but is limited to a smaller characterization range (10–20 μm) and is destructive to samples. Consequently, addressing the technical challenges associated with the comprehensive characterization of pore structures through the integration of various instruments is crucial for future research. As technology progresses, the digital core technology derived from these experimental methods is becoming increasingly vital for the quantitative characterization of micro-pores in shale. By precisely identifying and modeling rock pores, fractures, and other features in three dimensions, this approach effectively collects parameters such as porosity, pore size and distribution, pore density, and the proportion of different pore types, thereby facilitating a comprehensive transition from qualitative evaluations to quantitative evaluations (Figure 5).

3.3 Organic matter analysis

The distinctions in paleoenvironment and climatic conditions between lacustrine shale and marine shale lead to significant variations in the characteristics and types of organic matter (Xu et al., 2017; Liu et al., 2020). Key indicators, such as the formation mechanism, type, source, and maturity of organic matter, play crucial roles in determining the quality of shale reservoirs in lacustrine basins. Organic matter serves not only as the source of oil and gas generation but also as an essential type of reservoir space (Gao et al., 2016). The physical and chemical properties of organic matter, including porosity, permeability, total organic carbon (TOC), and the degree of thermal evolution, significantly influence the exploitation potential of lacustrine shale (Liu G. et al., 2016).

The quantity of organic matter and the presence of light hydrocarbon fluids in shale are critical indicators for evaluating the potential of shale oil and gas development (Saberi and Hosseini-Barzi, 2024). Traditional geochemical properties such as TOC, free hydrocarbons (S1), and extractable compounds provide only partial insights into these indicators. Moreover, acquiring these properties typically requires destructive sample treatment, which restricts the ability to perform multiple analyses on the same sample. The inherent heterogeneity of lacustrine shale reservoirs complicates the selection of comparable samples, potentially increasing analytical errors. Utilizing nuclear magnetic resonance (NMR) to assess organic matter abundance and light hydrocarbon content in shale preserves sample integrity during pretreatment and enables comprehensive joint testing and analysis on a single sample. It also allows for the dynamic analysis of changes in the chemical composition of kerogen as its thermal maturity evolves (Lee et al., 2020). MALDI-TOF (Matrix-Assisted Laser Desorption/Ionization Time-of-Flight) imaging mass spectrometry can be utilized to elucidate the complex molecular structure of organic matter in shale, thereby assisting researchers in understanding the effects of thermal maturation on the molecular architecture of kerogen (Rudyk et al., 2023). Thermal gravimetric analyzer (TGA) can quantitatively analyze the organic matter in a sample by examining the weight loss intervals, peak positions, and shapes of the thermogram, which facilitates the determination of kerogen composition, TOC content, and thermal maturity (Williams and Ahmad, 2000). Raman band separation (RBS) achieved through Raman spectroscopy exhibits a strong correlation with equivalent vitrinite reflectance, making it a reliable proxy for assessing shale thermal maturity (Figure 6). Additionally, the combined

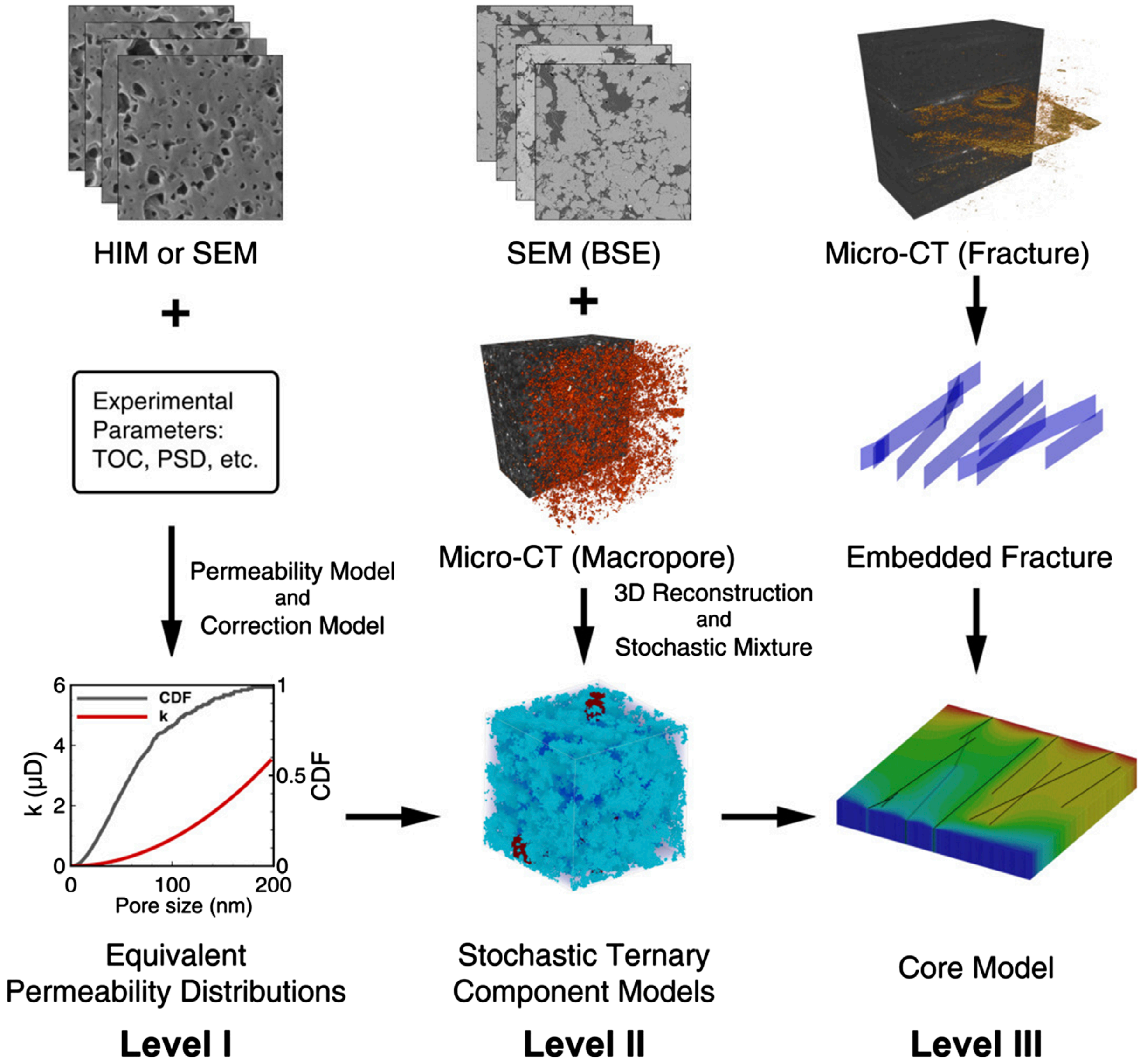

FIGURE 5
Proposed workflow of the multiscale pore structure characterization of shale by comprehensively using different technical means, cited from open access sources (Wu et al., 2017).

analysis of confocal laser scanning microscopy (CLSM) and fluorescence spectroscopy can quantitatively assess the distribution of organic matter and its influence on the potential for shale gas (Liu et al., 2020).

During diagenesis, organic matter in shale undergoes thermal evolution, transforming into hydrocarbons and generating numerous pores that serve as essential storage for shale oil and gas. To explore the formation and evolution of shale pores, thermal simulation experiments are indispensable. These experiments can be classified into closed, semi-open, and open systems based on their closure characteristics (Yin and Wei, 2019). Closed systems are tailored for specific research objectives, such as examining the effects of temperature, pressure, water medium, and minerals on hydrocarbon generation through pyrolysis. In contrast, open systems focus on analyzing the relationship between pyrolysis temperature and product composition. Semi-closed systems, which can replicate the temperature and pressure conditions of geological strata and collect pyrolysis products, are particularly suited for isothermal pyrolysis simulations and pore evolution.

3.4 Basic physical property analysis

Shale is characterized by its low porosity and permeability, making the accurate measurement of these properties a central focus and challenge in shale gas exploration and development. Recently, a variety of new technologies, methods, and interdisciplinary approaches have been applied to characterize the physical properties of shale reservoirs (Handwerger et al., 2011; Bohacs et al., 2014;

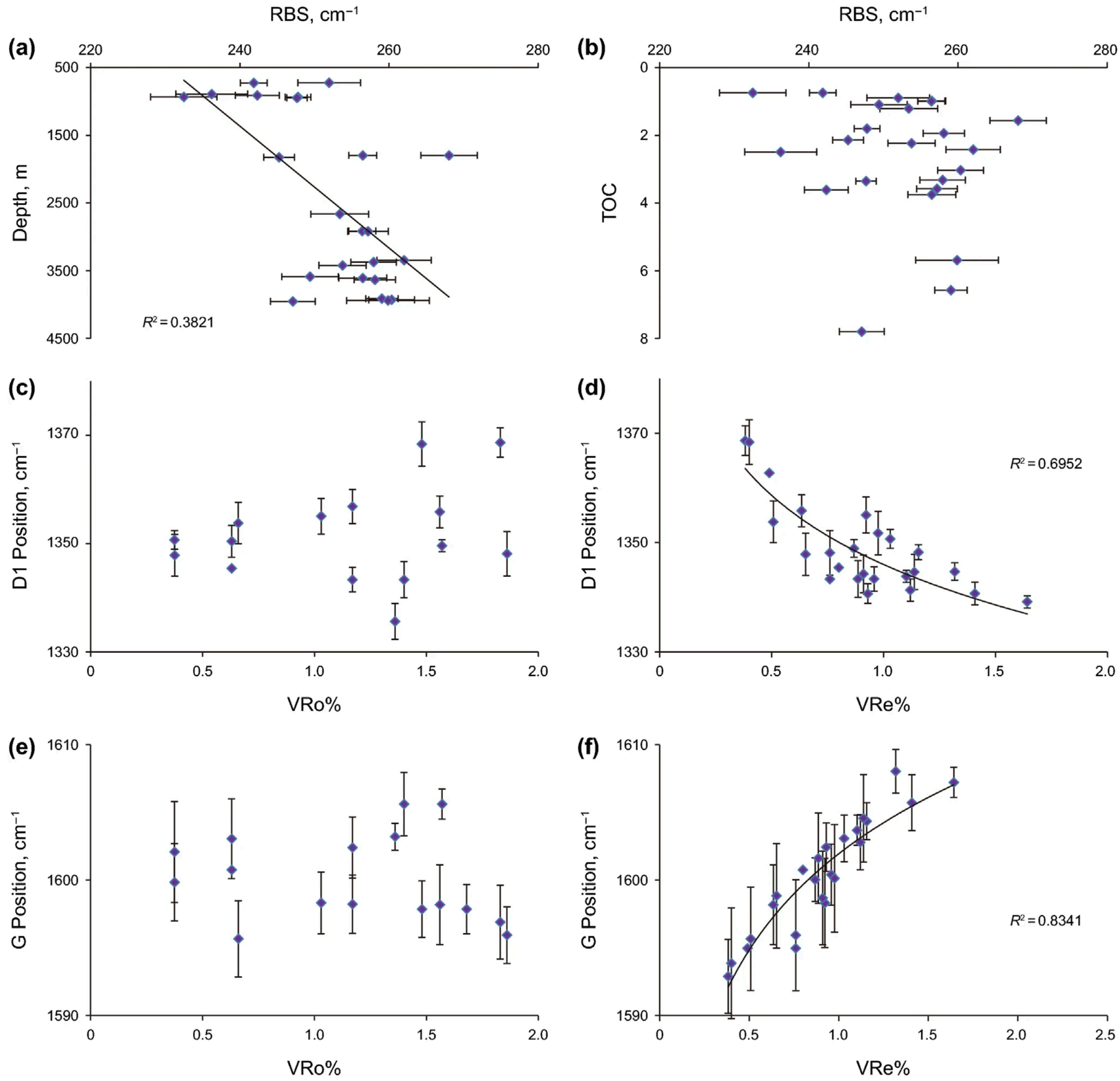

FIGURE 6
Raman spectroscopy test shale samples from different regions of North America; the experimental results showed that RBS has a high correlation with equivalent vitrinite reflectance (VRe%) **(A)** RBS distance versus depth; **(B)** RBS distance versus TOC; **(C)** VRo% versus D1 position; **(D)** VRe% versus D1 position; **(E)** VRo% versus G position; **(F)** VRe% versus G position, cited from open access sources (Kibria et al., 2020).

Goral et al., 2020). The primary experimental methods for assessing basic physical properties include gas adsorption analysis using N_2 and CO_2, high-pressure mercury intrusion analysis, nuclear magnetic resonance (NMR) analysis, and helium porosity analysis. Currently, gas adsorption, helium porosity, and nuclear magnetic resonance analyses are commonly employed to assess effective storage capacity, with results from these methods generally showing consistency (Hou L. et al., 2023).

Porosity testing in shale typically entails measuring bulk volume, particle volume, and pore volume. Laser 3D scanning has increasingly supplanted traditional caliper measurements and Archimedes' immersion method for assessing bulk volume. This advanced technique generates a three-dimensional surface map by scanning the sample with low-frequency pulse waves, thus circumventing limitations related to shape and contamination. Particle volume is commonly determined using a dual-chamber method based on Boyle's law. However, this method's susceptibility to environmental conditions and instrument precision can lead to variability in results across different laboratories. Pore volume measurement is performed using either the single-chamber method based on Boyle's law or a more precise liquid saturation method (He et al., 2023). Moreover, techniques such as NMR and digital core analysis are employed to assess porosity, although their results might be influenced by signal interference and limited observation areas.

The extremely low permeability of lacustrine shale reservoirs renders conventional testing methods inadequate for meeting accuracy requirements. The pulse decay method is currently the most widely used and effective approach for measuring permeability. Additionally, NMR has been employed to assess shale permeability (Zhou et al., 2016). In scenarios where obtaining complete core samples is challenging, a pressure decay method using crushed samples can be utilized.

3.5 Fracturability characterization

Fracturability refers to the ability of shale reservoirs to be effectively fractured to enhance production capacity. The brittleness of shale significantly influences its fracturability; typically, higher brittleness correlates with better fracturability. The brittleness index is the primary method for assessing the fracturability of shale reservoirs, and it typically utilizes two key indicators, namely, the elastic mechanical parameters of the rock and the content of brittle minerals. These indicators are primarily derived from geophysical and mechanical testing methods, which include full stress–strain testing instruments, conventional logging data, elemental capture spectrum (ECS), and radioactivity spectrum logging (Jarvie et al., 2007; Rickman et al., 2008; Li et al., 2012). In addition to evaluating mineral components and elastic parameters, researchers have explored calculating the brittleness index through laboratory indentation tests, fracture surface characteristics, rock hardness, and complete stress–strain curves. As research methodologies have diversified, the focus has expanded from analyzing individual brittle minerals, primarily quartz, to include combinations of minerals such as quartz, feldspar, and carbonates (Zhao et al., 2015). Rock mechanics analysis often involves true triaxial or pseudo-triaxial stress experiments, facilitating a comprehensive assessment of shale oil reservoir compressibility based on stress–strain curves, Young's modulus, and Poisson's ratio. Micro CT aids researchers in obtaining the distribution patterns of fractures in shale, and combined with fractal calculation methods, three-dimensional modeling, and other technologies, it can provide an accurate visual representation of the results of shale fracture expansion (Figure 7). For samples subjected to fracturing tests, the integration of *in situ* stress loading systems with high-resolution CT scanning enables real-time monitoring of fracture growth (Wu S. et al., 2020).

Factors influencing the fracturability of shale reservoirs encompass not only intrinsic properties but also the geological environment, indicating that brittleness evaluation is only one aspect of assessing fracturability. Additional key indicators include the extent of natural fracture development, diagenetic alterations, water saturation, and micro-fracture structures (Fan et al., 2023; Li P. et al., 2023). With advancements in micromechanical methods, technologies such as micro-indentation and nano-indentation have been employed to investigate the fracturability of shale reservoirs. Micro-indentation facilitates the evaluation of the mechanical characteristics of shale in its natural state and aligns with macroscopic mechanical methods, effectively complementing each other (Kasyap and Senetakis, 2022). Additionally, nano-indentation allows for the assessment of mechanical and fracture characteristics of shale across varying temperatures and bedding orientations (Zeng et al., 2022).

4 Challenges and issues of the experimental techniques in lacustrine shale reservoirs' characterization

Recent advancements in the exploration and development of lacustrine shale oil and gas have led to significant improvements in evaluation and experimental technologies. However, as these resources enter a new phase of comprehensive development, there is an increasing demand for enhanced experimental methods and fundamental research approaches. As a representative of "in-source" resources, the evaluation of shale oil and gas must evolve from assessing source rock to evaluating reservoirs, shifting focus from hydrocarbon generation capacity to hydrocarbon storage capacity and from hydrocarbon expulsion capacity to hydrocarbon production capacity.

4.1 Cross-scale characterization of shale reservoirs

Lacustrine shale, characterized as a fine-grained sedimentary system, has small particle sizes and is rich in organic matter. The formation model of shale reservoirs significantly diverges from that of conventional sandstone reservoirs. These reservoirs display pronounced vertical and lateral heterogeneity, with scale differences spanning 12 orders of magnitude from micro to macro levels, which complicates their characterization and evaluation (Wu Y. et al., 2020). Consequently, achieving a comprehensive cross-scale evaluation of lacustrine shale reservoirs and establishing connections among minerals, nano-pores, rock types, and geophysical responses, particularly in addressing data coarsening and integrating diverse data types, has emerged as a critical research area that demands significant breakthroughs.

4.2 Characterization of the evolutionary patterns of shale reservoirs

Unlike conventional sandstone and carbonate reservoirs, shale reservoirs are subject to complex interactions between organic and inorganic processes. The generation and expulsion of hydrocarbons from organic matter, coupled with the thermal evolution of inorganic matter during diagenesis, collectively influence hydrocarbon generation, accumulation, enrichment, and production in shale. The behavior of fluids within the micro–nano scale pore-fracture system of shale reservoirs is influenced by factors such as pore diameter, mineral composition, and complex wettability (Li G. et al., 2023). Current experimental evaluations face challenges in fully replicating this intricate process, which impedes a deeper understanding of the patterns of hydrocarbon generation, storage, and enrichment in lacustrine shale reservoirs.

4.3 *In situ* characterization of shale reservoirs

Laboratory conditions cannot fully replicate the underground geological environment, resulting in experimental results that may

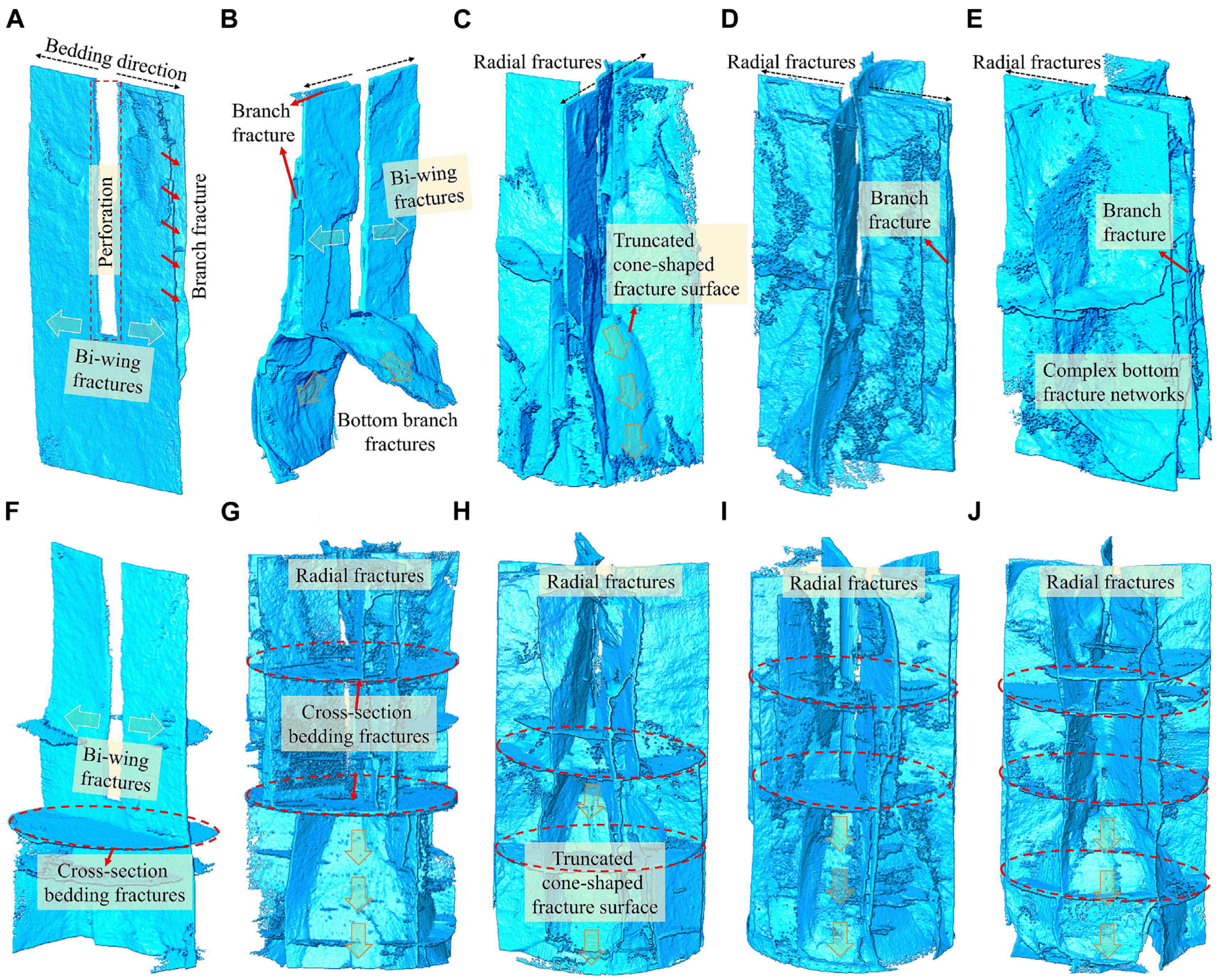

FIGURE 7
Micro CT is commonly utilized to characterize the three-dimensional distribution and morphology of cracks resulting from the experimental results of methane *in situ* explosion fracturing, cited from open access sources (Wang et al., 2024). **(A)** P-1. **(B)** P-2. **(C)** P-3. **(D)** P-4. **(E)** P-5. **(F)** V-1. **(G)** V-2. **(H)** V-3. **(I)** V-4. **(J)** V-5.

not accurately reflect the true geological situation. For instance, the measurement of porosity and oil content in shale oil reservoirs is complicated by the rich fluid content under formation conditions, which include high temperature, pressure, and a three-dimensional stress field. While temperature and pressure can be simulated in laboratory experiments, replicating fluid characteristics and the three-dimensional stress field environment remains a significant challenge (Tang et al., 2025). Additionally, field operations can alter the original temperature, pressure, and fluid composition of the formation, significantly impacting the characterization and evaluation of oil and gas content in the reservoir.

4.4 Analysis of the mineral matrix effect in hydrocarbon retention

The composition of the mineral matrix in lacustrine shale reservoirs is extremely complex and plays essential roles in hydrocarbon generation and preservation. The interaction mechanisms between various minerals and organic matter represent a significant area of study. Experimental analysis and simulations in this field not only require experimental techniques such as *in situ* characterization and the integration of multiple technologies but also necessitate the development of experimental methods capable of dynamically analyzing various evolutionary stages. These methods should aim to comprehensively assess the impact of critical environmental factors like temperature and pressure on mineral matrix across different geological epochs.

5 Outlook on the development trends of the experimental techniques in lacustrine shale reservoirs' characterization

As the exploration and development of lacustrine shale oil and gas resources continue to advance, the importance of characterization and evaluation technologies becomes increasingly critical. These technologies are essential not only for understanding the complex geological and physical properties of reservoirs but also for optimizing their large-scale development and efficient utilization. Accurate and comprehensive characterization enables better resource assessment, enhances recovery strategies, and ultimately supports sustainable and economically viable extraction processes.

5.1 Development toward high precision, multi-method, and cross-scale integration

The precise and quantitative characterization of nano-micro pores in terrestrial shale reservoirs is essential for reservoir evaluation. Nano-scientific techniques and evaluation methods, such as helium ion microscopy, transmission electron microscopy, and nano-indentation have proven effective in various fields and are promising for the characterization of shale reservoirs. However, integrating micro and macro scales poses significant challenges as different technical methods are limited by their applicable scales. No single experimental method can seamlessly transition from precise micro–nano scale to reservoir-scale. Future efforts should focus on enhancing cross-scale experimental devices and integrating multiple methods. This integration should include optical microscopy, electron microscopy, micro-area mineral analysis, laser Raman spectroscopy, and CT scanning to comprehensively characterize minerals, pores, fractures, and fluids.

5.2 Enabling dynamic evolutionary evaluation of key attributes

The nanoscale pore system in shale reservoirs, combined with their complex mineral composition and hydrocarbon storage states, necessitates the dynamic evaluation of geological evolution. Experimentally simulating geological evolution can be used in reconstructing geological history, identifying key controlling elements, revealing the dynamics and thermodynamics of geological evolution, and predicting the distribution of favorable geological bodies and sweet spots. Future research directions should focus on simulating fine-grained sedimentation and organic matter enrichment, dynamic hydrocarbon generation and drainage processes, mineral matrix effect, micro- and nano-pore evolution, and the growth of artificial fractures.

5.3 Enhanced evaluation of shale reservoir characterization for *in situ* conditions

Shale oil and gas reservoirs are highly sensitive to stress release, weathering, and other surface conditions, which complicates the accuracy of laboratory evaluations in reflecting true *in situ* reservoir characteristics. Developing experimental techniques that mimic the *in situ* conditions can significantly enhance the accuracy and precision of key parameter characterization in shale reservoirs. Consequently, direct or indirect *in situ* evaluation technologies are essential for accurately characterizing the true state of subsurface shale reservoirs and represent a crucial direction for future development. The advancement of *in situ* testing technologies should be prioritized, including tests for shale physical properties under formation temperature, pressure, and fluid storage conditions, isothermal adsorption tests, multiphase fluid seepage and drive-off experiments, and competitive gas adsorption experiments.

5.4 Big data and artificial intelligence will play key roles

In the future, the characterization and evaluation of shale reservoirs will generate vast amounts of data across various scales and methods, fostering the integration of intelligent technologies such as big data, artificial intelligence, and cloud computing for the oil and gas industry. The adoption of these technologies will streamline automated data acquisition, processing, and analysis, providing essential support for comprehensive multi-factor experimental evaluations. Machine learning techniques, for example, are employed to analyze the lithofacies characteristics of shale reservoirs based on well logging curves (Hou M. et al., 2023). Techniques such as principal component analysis (PCA) and K-means clustering are utilized to classify geochemical characteristics (Li et al., 2024), offering efficient data analysis solutions to tackle complex mechanistic challenges in shale gas exploration and development.

6 Conclusion

1) Advancements of experimental techniques in lacustrine shale reservoir characterization. The unique characteristics of lacustrine shale, including its micro- and nano-porosity systems, strong heterogeneity, and complex evolutionary processes, necessitate advanced reservoir characterization and evaluation technologies. Although research in this field has significantly increased in recent years, the citation index of core articles still offers room for improvement. These technologies, particularly experimental methods, are crucial for guiding the efficient exploration and development of shale oil and gas. Recent advancements have substantially enhanced the accuracy of lacustrine shale reservoir characterization across scales, from millimeter to micrometer and even nanometer levels. Additionally, the dimensionality of characterization has progressed from two-dimensional to three-dimensional, facilitated by cutting-edge technologies such as nano-CT scanning and FIB-SEM. These technological advancements lay a solid foundation for identifying sweet spots, optimizing reservoir reforming strategies, and formulating effective development policies. The key aspects of shale reservoir characterization include mineral identification, organic matter analysis, pore structure evaluation, fundamental physical properties, and fracturability assessment.
2) Challenges and issues. As exploration and development of lacustrine shale oil and gas intensify, the demand for more sophisticated evaluation methods and fundamental research tools is increasing. The related evaluation techniques must address several key challenges, including the need for cross-scale characterization, dynamic evolution characterization, and *in situ* parameter characterization. Overcoming these challenges requires the integration of multiple methods and

the development of new technologies to ensure accurate and comprehensive assessments.

3) Development trends outlook. Future characterization technologies for lacustrine shale reservoirs should emphasize high precision, multi-method integration, and cross-scale analysis. There will be a focus on the dynamic evaluation of key attributes and the characterization of *in situ* parameters. Big data and artificial intelligence will play a crucial role in these advancements, enabling more accurate and efficient data acquisition, processing, and analysis. By leveraging these technologies, researchers and industry professionals will gain a better understanding of the complex mechanisms underlying lacustrine shale reservoirs, leading to more effective and sustainable exploitation of these resources.

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