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Research Progress on Experimental Characterization Techniques of Pore Structures

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Abstract. The pore structure is considered the core carrier of hydrocarbon storage and migration in geological reservoirs, especially in unconventional oil and gas reservoirs. Accurate characterization of these pore structures is the premise of oil and gas exploration and development. This paper systematically establishes an experimental characterization system for pore structure, which contains three aspects: qualitative imaging, quantitative testing, and all-aperture integrated characterization. It analyzes the internal contradictions of the conventional techniques, for example, the trade-off between resolution and representativeness of the sample, and the inability to monitor dynamic evolution. It also describes the emerging techniques, including focused ion beam-nanotomography, coherent diffraction imaging, and atom probe tomography, which embody the significant advances being made in nanoscale resolution, in situ monitoring of dynamics, and element-pore correlation studies at the level of the atom. By combining various techniques (for example, micro/nano CT with mercury intrusion porosimetry, those involving pore network models (PNM)), and making use of intelligent algorithms (deep learning, Bayesian fusion), it becomes possible to span the gulf between "nanoscale pore morphology characterization" and "quantitative evaluation of full-scale flow properties". This study illustrates that the combination of the emerging and conventional techniques provides a new route to solving the problem of reservoir heterogeneity and multiscale pore characterization, affording much theoretical and practical significance for the efficient and effective development of unconventional oil and gas.

Keywords: pore structure, experimental characterization, resolution–representativeness, emerging technologies, multiscale integration

1. Introduction

The field of unconventional oil and gas has become the focus of attention for the sustainable development of the globalization of the petroleum industry. Compared with conventional oil and gas, the unconventional reservoirs represented by shale gas, tight sandstone and tight carbonate rocks have developed a morphology of pore structure and fluid migration mechanism to form, so that the large-scale exploration and development of such oil and gas is extremely difficult to implement. Thus, the accurate characterization of the pore structure of the reservoirs and the understanding of the mechanism of accumulation and migration of fluids, constitute the key issues

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to be dealt with in the geological research of oil and gas of unconventional origin [1]. In the last 10 years, the development of an effective means of looking at rocks by means of X-ray computer tomography (CT), nuclear magnetic resonance (NMR), field emission scanning electron micropscopy (FE-SEM), atomic force microscopy (AFM), gas adsoption etc. has made it possible to assess and quantitate (3D; cross-scale) the pore structure of tight cores and the movable fluids. There exciting scientific developments also have found their way into commercial application and have become basic techniques for geologists studying the interior structure of unconventional reservoirs and the effect on reservoir behaviour, especially in understanding the migration of hydro-carbons, thus giving important technical support it also has afforded the application of hydrocarbons rapid development of oil and gas of unconventional character [2].

The pore structure of a reservoir—namely, the pore morphology, throat distribution and their interconnectivity, all determine the hydrocarbon storage capacity and the ability to establish flow. In the case of unconventional reservoirs such as shale and tight sandstone, the nanoscale pores (<100 nm) can account for 60-90% of the total pore volume and hence high precision characterization is a basic prerequisite to determining sweet spots and optimising development plans [3,4]. The unconventional reservoirs are many and diverse, being mainly shales, tight sandstones, and tight carbonates, characterised by good development of micro- and nanoscale pore-throat systems and by low porosities and high degrees of heterogeneity [1]. The unconventional reservoirs possess also unique mineralogies and organic matter contents that cause them to differ significantly from conventional traps which have been thus distinguished. Subsequently, this will influence the type pore formation and preservation that occurs therein and will also affect the storage and enriching mechanisms of the fluids contained. Accordingly, the pore structure characterization of unconventional reservoirs needs to be considered in a systematic manner covering an integrated evaluation of the organic matter, minerals, pores, fractures and fluids [1].

The pore characterization methods currently used, such as thin section petrography and mercury intrusion, have the limitations of scale and resolution which often prevent a complete characterization of the pore structure for all pore sizes throughout the range of nanometers to centimeters. These methods are generally static and lack the ability to simulate the changes which take place in pore structure with time as a result of temperature, pressure, and fluid interactions in the subsurface environment. Nevertheless, with the exploration and development of unconventional oil and gas resources, the pore size of reservoirs has gradually moved to the smaller sizes, which has made the unconventional sources the most optimal in oil and gas resource development. These reservoirs generally have poor physical properties, with porosity less than 10 per cent, permeability less than 0.1 M.D, pore throat sizes generally smaller than 1μ, and a predominately micro to nano pore structure with complex throat structure [2]. The results of the different characterization techniques vary greatly as far as their principles, accuracy and applicability, while each method is applied so as to be generally only adverse for certain pore sizes, which cut down on their degrees of usefulness [2]. The recent changes in synchrotron radiation imaging, monitoring in situ, atom level characterization methods and intelligent algorithms provide a possible road to the overcoming of the limitations of the traditional techniques. The paper presented systematically the research and development in the area of experimental pore characterization from the standpoints of the construction of the technical systems employed, the limitations of each technique analyzed in a critique of their application, the advances made in the field of the emerging technologies and the field of application, and their future directions.

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2. Experimental characterization system for pore structures

2.1. Qualitative imaging techniques

A qualitative imaging approach is taken to intuitively present the pore morphology, spatial distribution, and mineral associations. Observation can be either two-dimensional (2D), or three-dimensional (3D) reconstruction. Method selection depends on the desired scale and resolution of the investigation.

2.1.1. Two-dimensional imaging techniques

Thin sectioning combined with scanning electron microscopy (SEM) is the classical method for 2D characterization. The preparation of thin sections consists of filling the rock pores with colored epoxy resin (for example, blue epoxy) using vacuum infusion, and polishing sections of 30 μm thickness for optical microscopy observations. This allows an intuitive representation of the micron-scale pores (being >1 μm) such as those formed by dissolution and intergranular pores, but with a limit of resolution of 200 nm and an error source of 10%–15% in the interpretation due to manual means [5]. Field emission scanning electronic microscopy (FESEM), using the very fine electronic beam (1–5 nm diameter) achieves high resolution (0.5–6 nm) and can adequately identify the organic matter pores and intergranular mineral pores in shales within the range 2–50 nm [3]. Environmental scanning electron microscopy (ESEM) overcomes the limits imposed by vacuum, but in conjunction with energy-dispersive spectroscopy (EDS) the in situ identification of the occurrence of hydrocarbons in pores can be achieved under humid conditions. Thus, an ESEM-EDS investigation of tuffaceous rocks in the Tiaohu Formation of the Santanghu Basin has shown that micron-scale feldspar dissolution pores (50 μm) can contain C/O atomic ratios of 1.2–1.5 which infers the presence of residual oil the pores.

Atomic force microscopy (AFM) is an important method for 2D nanoscale pore characterization. Using the forces of atomic interaction between the probe and the sample, AFM can obtain lateral resolutions of 0.1 nm and vertical resolutions of 0.01 nm, sufficiently small for the characterization of 2–50 nm nanoscale pores. In organic-rich regions of the Longmaxi Formation shale in the Sichuan Basin, surface roughness (Ra) values ranged from 12–18 nm, significantly higher than in inorganic mineral regions (3–5 nm). Ra values were positively correlated with specific surface area (SSA > 20 m^2/g when Ra > 10 nm), directly influencing shale gas adsorption capacity. However, AFM scans cover small areas (<100 μm^2), requiring repeated scanning of 5–10 representative regions to ensure statistical reliability. Additionally, probe wear (radius > 10 nm) can introduce pore size measurement deviations of 10%–20% [6].

2.1.2. Three-dimensional reconstruction techniques

Micro/nano-computed tomography (micro-CT and nano-CT) and focused ion beam–nanotomography (FIB-nt) are central to achieving 3D visualization of pore structures. Micro-CT (μ-CT) has a resolution of approximately 1 μm, suitable for analyzing the connectivity of pores larger than 1 μm, while nano-CT reaches 50 nm resolution and can characterize nanoscale pores from 50 nm to 5 μm. Multiscale CT scans of tight sandstones in the Chang 7 Member of the Ordos Basin show that micron-scale dissolution pores (2–10 μm) and nanoscale intergranular pores (50–500 nm) are connected via throats (0.5–2 μm), forming a hierarchical “macro–micro–nano” network. However, the effective field of view of nano-CT is limited to 50–100 μm^3, requiring multiple scans

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to cover heterogeneous regions, and the experimental cost is high (approximately 20,000 RMB per synchrotron CT scan).

FIB-nt achieves 3D reconstruction through "focused ion beam (FIB) slicing + SEM imaging," reaching resolutions of 10 nm and accurately depicting the spatial distribution of organic matter pores in shales. Curtis et al. analyzed Woodford shale using FIB-nt and found that at an organic maturity of Ro = 2.0%, organic matter pores accounted for 60%–70% of total pore volume, with pore-throat connectivity increasing significantly with higher Ro [4]. Nevertheless, the analyzed volume is very small (<10 μm × 10 μm × 2 μm), experiments are time-consuming (4–12 h), and the milling process can generate thermal damage (local temperatures >300 ℃) leading to nanoscale pore closure. Pre-experiment low-temperature cooling (−50 ℃) is required to minimize such damage.

2.2. Quantitative testing techniques

Quantitative testing techniques aim to obtain pore-throat parameters (e.g., pore size distribution, pore-throat ratio, specific surface area) and fluid occurrence information. Cross-validation among multiple techniques is necessary to reduce errors and ensure data reliability.

2.2.1. Mercury Intrusion Porosimetry (MIP)

Mercury intrusion porosimetry is based on the Washburn equation:

$$r = \frac{2\gamma cos\theta}{P} \tag{1}$$

MIP is classified into high-pressure mercury intrusion (HPMI) and rate-controlled porosimetry (RCP). HPMI can reach maximum intrusion pressures of 414 MPa, covering a theoretical pore size range of 3 nm–300 μm, from mesopores to macropores. However, high pressures may fracture samples (e.g., tight sandstones develop artificial fractures above 200 MPa, overestimating the proportion of large pore throats by 10%–15%). RCP maintains a quasi-static process with extremely low mercury injection rates (0.00005 mL/min). Pressure fluctuations are used to distinguish pores (pressure stable, rapid mercury uptake) from throats (pressure spikes, slow mercury uptake), enabling direct measurement of pore-throat ratios (typically 3–6). Due to equipment pressure limitations, nano-sized pore throats <0.12 μm cannot be measured. Combined MIP–CT analysis of Berea sandstone shows that the pore-throat radius distribution measured by MIP (main peak 1–10 μm) deviates by less than 10% from CT-extracted throat radii (main peak 2–8 μm). Accuracy is significantly improved after threshold correction [6].

2.2.2. Nuclear Magnetic Resonance (NMR) and gas adsorption

NMR: NMR characterizes pore structures via the transverse relaxation time (T_2) distribution of hydrogen-containing fluids. The core principle is that fluid in small pores is strongly bound (T_2 < 10 ms, bound fluid), whereas fluid in large pores is freer (T_2 > 100 ms, movable fluid). NMR analysis of tight sandstones in the Fuyu oil layer of the Daqing Oilfield showed that when movable fluid saturation (proportion of the right-peak area) is 25%–30%, daily oil production exceeds 1 t, consistent with test production results. Combined with diffusion-weighted imaging (DWI), pore tortuosity can be calculated (shale tortuosity generally >5). Absolute pore sizes can be inverted using

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a T_2-to-pore radius conversion ($r = C \times T_2$, with C calibrated via mercury intrusion), minimizing distortion of T_2 spectra caused by magnetic minerals such as pyrite (post-correction error <5%).

Gas Adsorption: Gas adsorption techniques derive nanoscale pore parameters from adsorption–desorption isotherms. Nitrogen adsorption at 77 K targets mesopores of 2–200 nm, while CO_2 adsorption at 273 K complements the <2 nm micropore range. Nitrogen adsorption of Longmaxi Formation shale in the Sichuan Basin revealed a specific surface area of 20–35 m^2/g, with pores primarily concentrated in the 10–50 nm range (ink-bottle shaped, H4-type hysteresis). Combined CO_2 adsorption and FIB-nt analysis of Opalinus clay showed that micropores (<2 nm) account for 30%–40% of total pore volume, positively correlating with the number of organic-matter micropores observed by FIB-nt (R^2 = 0.82), confirming the critical role of micropores in shale gas adsorption [6].

2.3. Integrated full-aperture characterization techniques

Pore radii in tight oil reservoirs span 10^{-9}–10^{-4} m. No single technique can cover this full scale due to inherent advantages and limitations (Table 1). Complete characterization requires the combined application of "qualitative–quantitative" and "microscopic–macroscopic" techniques. Xiao et al. integrated low-temperature nitrogen adsorption (1–50 nm), high-pressure mercury intrusion (3 nm–300 μm), rate-controlled porosimetry (0.12–300 μm), and centrifugal nuclear magnetic resonance to establish a method for full-scale pore size distribution in tight cores. In typical domestic tight oil areas (Daqing, Changqing, Sichuan, Dagang), pores around 100 nm account for the largest proportion (40%–50%) in tight sandstones, representing the primary target for field development [7]. By embedding pore-throat parameters obtained from integrated characterization (e.g., pore-throat radius distribution, coordination number) into pore network models (PNM), reservoir permeability can be predicted with errors <15%, providing parameter support for the optimization of development plans [6].

Table 1. Comparison of pore structure characterization techniques (integrating traditional and some emerging methods)

Technique Type	Optimal Pore Size Range	Resolution	Measured Content	Advantages	Limitations
Thin Section	>1 μm	200 nm	Pore morphology, connectivity	Intuitive, low cost	Cannot characterize nanoscale pores; large manual errors
FESEM	5 nm–10 μm	0.5 nm	Pore morphology, mineral properties	High resolution; can combine with EDS	2D analysis; requires conductive coating
Nano-CT	50 nm–5 μm	50 nm	3D pore network	Non-destructive; nanoscale resolution	Small field of view (<100 μm^3); high cost
High-Pressure Mercury Intrusion	3 nm–300 μm	–	Full-aperture pore-throat distribution	Wide coverage	High-pressure may damage samples; overestimates large pore throats
Nitrogen Adsorption	2–200 nm	–	Specific surface area, mesopore distribution	Sensitive to nanoscale pores	Cannot simulate subsurface T-P conditions; only reflects open pores

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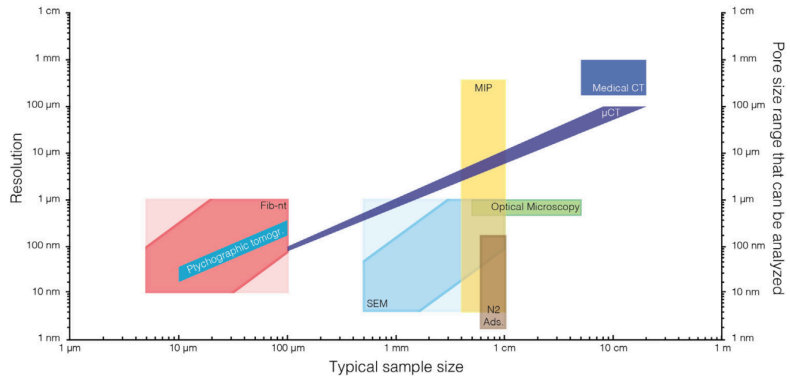


Figure 1. Relationship between resolution and sample size for different pore characterization techniques (modified from Bultreys et al. [6])

3. Limitations of traditional techniques and breakthroughs in emerging technologies

3.1. Core contradictions of traditional techniques

Bultreys et al.'s "resolution–sample size relationship diagram" (Figure 1) vividly illustrates this contradiction. High-resolution techniques such as FIB-nt and AFM allow nanoscale pore observation but have extremely limited fields of view (FIB-nt <100 µm³, AFM <100 µm²), making it impossible to reflect macroscopic reservoir heterogeneity. Large-scale techniques (e.g., medical CT with sample sizes on the order of 10 cm) can cover centimeter-scale cores, but their resolution is only at the micrometer level, missing 60%–90% of nanoscale pores [6]. For shale reservoirs, FIB-nt scans of a single region (<10 µm³) yield porosities of 8%–10%, while whole-rock measured porosity is 4%–5%, a deviation of 30%–40%. Repeated scanning of more than 10 heterogeneous regions is needed, consuming 20–40 h in total. µCT underestimates porosity by 15%–20% due to missed nanoscale pores, insufficient for detailed evaluation of unconventional reservoirs [8].

3.1.1. Insufficient dynamic evolution characterization

Traditional techniques provide largely "static snapshots" and cannot simulate the dynamic changes of pores during geological processes such as diagenesis, fracturing, and production. High-resolution devices (e.g., FESEM, nano-CT) have delicate sample chambers that cannot withstand actual subsurface temperature and pressure conditions (>150 ℃ , >30 MPa). When simulating shale thermal evolution, the formation of organic matter pores (Ro = 0.9%–2.5%) may deviate significantly from natural geological conditions. Equipment capable of high-temperature and high-pressure loading (e.g., high-pressure autoclaves) has imaging resolutions >1 µm, insufficient to observe nanoscale pore evolution, such as pore blockage caused by fracturing fluids reacting with clay minerals. Furthermore, static characterization cannot distinguish the causes of pore changes; for example, an increase in pore volume from CO_2 injection cannot be easily attributed to either dissolution (chemical process) or pressure variation (physical process) [9].

3.2. Breakthroughs in emerging technologies

Emerging technologies overcome traditional limitations along three dimensions: "resolution enhancement," "dynamic monitoring," and "atomic-level correlation," providing new approaches for detailed pore structure characterization. The significant developments are summarized below.

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3.2.1. Synchrotron radiation and coherent imaging

Coherent diffraction imaging (CDI) uses the coherence of X-rays to reconstruct the electron density distribution of a sample from scattering patterns, without the need for physical lenses, to exceed the physical limits of resolution (necessarily less than about 10 nm) which exist in conventional imaging systems and is also well suited for low-contrast samples of materials such as organic matter which have wide distributions of small pores in their structure. Dierolf et al. used laminography CT (a form of CDI) to get down to a few nm (usually less than 104 poisons) in the characterization of pores of nanoscale dimensions. Analysis of Woodford shale revealed that organic matter pores are honeycomb-like and that pore-throat connectivity increases significantly with maturity [10]. Soft X-ray tomography, using soft X-rays (wavelength 2.4–4.4 nm), exploits absorption differences of light elements such as C and O, achieving ~30 nm resolution. It allows in situ imaging of fluid-containing samples (avoiding pore deformation caused by drying) and is suitable for dynamic monitoring of fluid-filled pores. For example, in Barnett shale, 3D quantitative analysis shows that organic matter pores account for 65%–75% of total pore volume, representing the main storage space for shale gas.

3.2.2. In situ dynamic monitoring

In situ high-temperature and high-pressure Raman-AFM coupling simultaneously acquires chemical and structural information. Raman spectroscopy monitors gases (e.g., CH_4, CO_2) generated from organic matter pyrolysis, while AFM observes real-time pore morphology changes, such as pore enlargement and new pore formation. In shale thermal evolution experiments simulating 150 °C and 30 MPa conditions, the formation rate of organic matter pores ranged up to 0.1 nm/s as Ro increased from 0.9% to 2.5%, and the proportion of aromatic carbon correlated positively with pore development [11], providing a microscopic mechanism for the effect of thermal maturity on pores. In situ liquid environmental transmission electron microscopy (In-situ Liquid TEM) introduces fluids (water, oil, fracturing fluid) into the TEM sample holder, achieving ~1 nm resolution. It allows observing the dynamic interrelations of fluids and pores. Thus, during the waterflooding of a porous medium, the rupturing and reforming of oil films in the underground throats (50 nm) give direct information for optimizing enhanced oil recovery.

3.2.3. Atomic-level characterization

With the atom probe microscope (APT) is involved the techniques of field evaporation which evaporate in one facet after another the atoms of the sample, and mass spectral analysis with the time of flight detects the kind of atom as also his spatial position. With 0.1 nm resolution, APT can locate nanoscale pores and analyze elemental distributions. Zhang et al. found that C and H atoms are enriched around organic matter pores, and the π-electron state of aromatic carbon positively correlates with methane adsorption, providing atomic-level evidence for shale gas adsorption mechanisms [12]. Scanning transmission electron microscopy (STEM) combined with electron energy loss spectroscopy (EELS) integrates high-resolution imaging and chemical analysis. STEM localizes nanoscale pores, and EELS analyzes the electronic structure around pores (e.g., π-electron state of organic matter pores). Research on Longmaxi Formation shale in the Sichuan Basin shows that when the aromatic carbon proportion of organic matter pores exceeds 70%, methane adsorption reaches 3.5 cm^3/g, guiding the prediction of shale gas sweet spots.

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4. Technological development directions and prospects

4.1. Multiscale data integration and intelligent empowerment

A core direction is the integration of multi-source data using machine learning algorithms to construct a "pore–core–reservoir" multiscale model. First, image super-resolution reconstruction employs convolutional neural networks (CNNs) to train macroscopic CT images (10 μm resolution), enhancing resolution to 50 nm and achieving "cm-scale field of view + nm-scale resolution," thus resolving the conflict between high resolution and large field of view [6]. Second, multi-technique data correlation assigns weights to different datasets—mercury intrusion (pore-throat radius), NMR (fluid occurrence), and FIB-nt (pore morphology)—using a Bayesian fusion algorithm (CT 40%, MIP 30%, adsorption 30%), constructing a probabilistic pore model and reducing porosity fusion error from 12% to 5%. Third, laboratory–well log integration applies random forest algorithms to link core pore parameters (porosity, permeability) with well log curves (sonic transit time, density), enabling full-reservoir pore structure prediction. For example, Zhao et al. achieved prediction accuracy exceeding 85% for the Chang 7 Formation in the Ordos Basin [13].

4.2. In situ real-time monitoring devices and multiphysics coupling

Overcoming the conflict between "high resolution" and "high temperature/high pressure" is key to in situ monitoring. Three core devices need to be developed: First, an NMR in situ loading apparatus with a high-temperature and high-pressure sample chamber (200 ℃, 50 MPa), combined with diffusion-weighted imaging to monitor fluid migration in real time (e.g., organic matter pore formation rate during shale thermal evolution). Second, a high-resolution X-ray in situ device, where synchrotron facilities are equipped with high-temperature/high-pressure core holders, achieving dynamic imaging at <50 nm resolution (e.g., pore evolution induced by CO_2 dissolution). Third, an FESEM in situ loading device, with an optimized sample stage to maintain 1 nm resolution at 150 ℃ and 30 MPa, allowing direct observation of fluid occurrence and flow in nanoscale pores. These devices provide data that more realistically replicate subsurface geological conditions, crucial for elucidating pore evolution mechanisms.

4.3. Integrated imaging–modeling and digital twins

Promoting deep coupling between experimental data and pore-scale modeling achieves a closed loop of "characterization–simulation–prediction." First, dynamic updating of digital rock models uses in situ CT time-series data to continuously update digital cores, simulating multiphase flow evolution (e.g., fluid movement in fracture–pore networks after hydraulic fracturing). Second, automatic extraction of PNM parameters develops AI algorithms (e.g., U-Net, Mask R-CNN) to extract pore-throat parameters (coordination number, shape factor) from FIB-nt/CT images, reducing manual intervention (error <8%). Third, multiphysics coupled simulations introduce temperature, pressure, and chemical fields into PNM, simulating complex geological processes such as pore evolution during diagenesis, providing more comprehensive theoretical support for reservoir development plan optimization [14].

5. Conclusions

1. Experimental techniques for pore structure characterization have formed a complete system of "qualitative imaging—quantitative testing—full-pore-size integration." The limitations of traditional

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methods in resolution, sample scale, and dynamic monitoring have driven the development of emerging technologies such as synchrotron imaging, in situ monitoring, and atomic-level characterization, providing new approaches for the detailed evaluation of unconventional reservoirs.

2.Emerging technologies—including coherent diffraction imaging, in situ high-temperature/high-pressure coupling, and atom probe tomography—overcome traditional technical bottlenecks along three dimensions: nanoscale resolution (~10 nm), dynamic evolution monitoring (real-time capture of fluid–rock interactions), and atomic-level element-pore correlation (~0.1 nm), revealing the microscopic mechanisms of pore formation and evolution.

3. In future research, the core direction is multiscale data integration, that is, machine learning-driven multi-source data fusion, research and development of in situ monitoring devices, that is, high-resolution high-temperature/high-pressure sensors, integrated imaging and modeling, that is, dynamic digital rock models combined with multiphysics coupled PNM. Transition is from nanoscale pore morphology description to quantitative assessment of the full-scale flow properties, to provide both theoretical and technical support for the efficient development of unconventional reservoirs such as shale and tight sandstones.

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