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Perspective

Pore network characterization and fluid occurrence of shale reservoirs: State-of-the-art and future perspectives

Mengdi Sun[1]*, Jiajun Fu[1], Qiming Wang[2], Zhiye Gao[3]*

[1]National Key Laboratory of Continental Shale Oil, Northeast Petroleum University, Daqing 163318, P. R. China
[2]National Key Laboratory of Deep Oil and Gas, China University Petroleum (East China), Qingdao 266580, P. R. China
[3]National Key Laboratory of Petroleum Resources and Engineering, China University of Petroleum, Beijing 102249, P. R. China

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Abstract:
Due to the increasing energy consumption and the promoting of the carbon neutral target, the exploitation of shale oil and gas, as well as carbon dioxide sequestration and hydrogen storage using shale as caprock, has received enormous attention. As a foundation for these hotspots, the characterization of pore structure in shale reservoirs has been widely studied. In this paper, the application of fluid intrusion and radiation methods in the characterization of pore structure in shale reservoirs was systematically reviewed, and the merits and limitations of both methods were highlighted. Taking the Fengcheng shale as an example, a detailed investigation of the fluid occurrence state was conducted, indicating that the fluid occurrence state significantly impacts the exploitation of hydrocarbon from shale reservoirs. Furthermore, there needs to be a systematic of investigation of how the pore structure characteristics and inorganic components of shale reservoirs control the integrity and safety of CO_2 and H_2 storage. Moreover, confinement effect of nanopores in shale should be paid attention to in future research on carbon and hydrogen storage.

1. Introduction

Shale reservoirs have received a substantial attention as unconventional hydrocarbon reservoirs. Because of its ultra-low matrix permeability, complex micro- and nano-pore structure and mixed wettability caused by multiple components, shale reservoir faces many challenges in the study of hydrocarbon occurrence and development (Gao et al., 2023a; Yang et al., 2023). To effectively tackle these challenges, a variety of methods have been used to characterize the pore-fracture structure characteristics of shale reservoirs from multi-scale and multi-dimension (Fig. 1).

The in-depth study of the fluid occurrence in pore-fracture system is helpful to understand the enrichment mechanism of shale oil and gas and optimize the exploration plans. The fluid in shale reservoirs is mainly composed of methane, water, and multi-component liquid hydrocarbon, which changes with the evolution of shale maturity (Li et al., 2016). In overmature shale reservoirs, the fluid is mostly composed of methane and water in adsorbed and free states (Xie et al., 2023). For high mature shale oil reservoirs, light component and heavy component fluids are selectively distributed in the pore-fracture system, and the state of shale oil occurrence directly affects its mobility (Hu et al., 2021)

Underground depleted gas reservoirs for CO_2 sequestration or H_2 storage are usually sealed by shale formation to prevent upward fluid diffusion (Qin et al., 2023; Zhang et al., 2023). Due to the abundant nanoscale pores in shale, fluid migration will be restricted, which greatly enhances the sealing capacity of shale (Li et al., 2024). In addition, it is a focus of current research to improve the recovery of shale oil and gas and realize carbon storage by injecting CO_2 into shale reservoirs (Ma et al., 2021). Either way, pore network characterization and fluid occurrence of shale reservoirs will contribute to storage integrity during subsurface carbon or hydrogen storage.

This paper systematically describes the scale, advantages, and limitations of various analytical methods (fluid intrusion

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*Corresponding author.
E-mail address: sunmd@nepu.edu.cn (M. Sun); fujiajun0851@163.com (J. Fu); wangqm@upc.edu.cn (Q. Wang); gaozhiye@163.com (Z. Gao).

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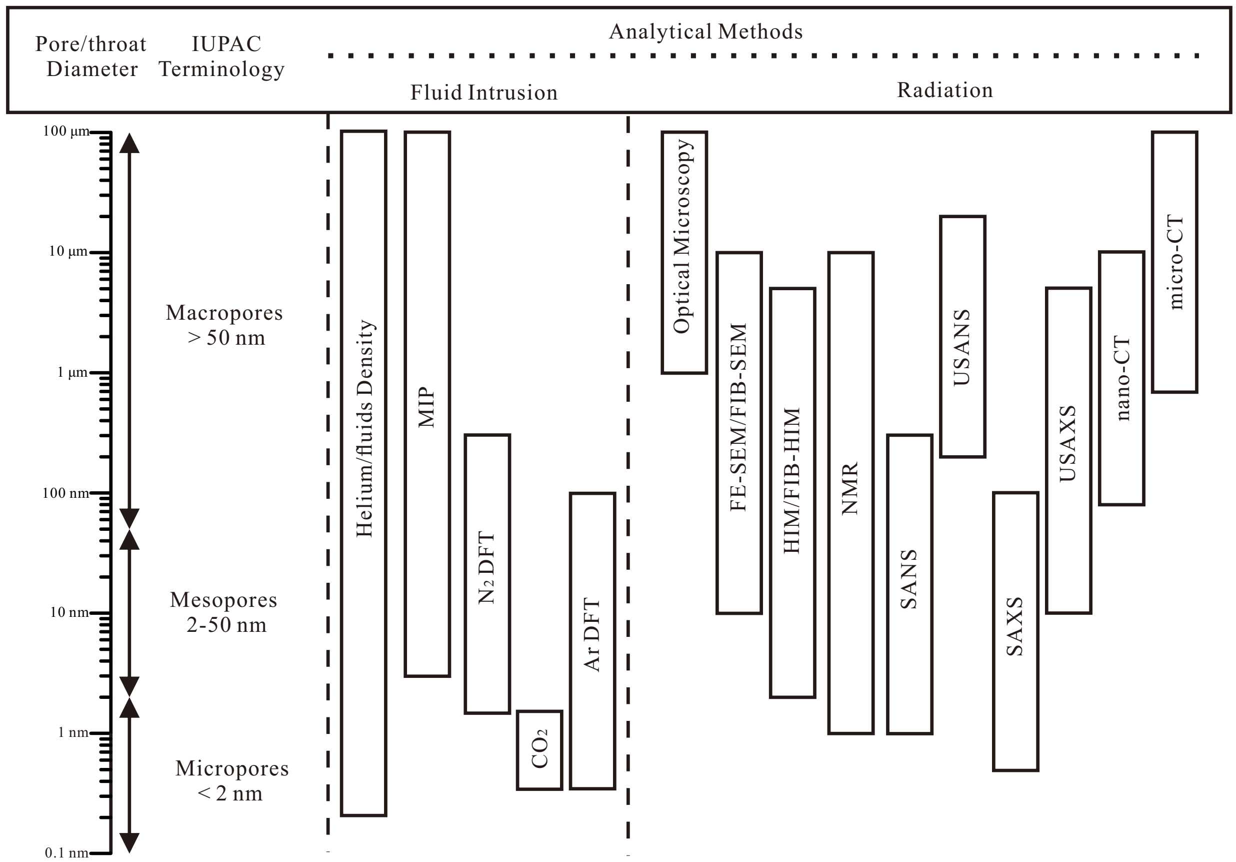

Fig. 1. Methods used to investigate pore characteristics in shale (modified from Sun et al., 2020).

and radiation method) for the characterization of pore structure in shale. Then, the occurrence state of fluid in shale pore-fracture network is analyzed by taking Fengcheng shale oil reservoir as an example. Finally, the important role of shale reservoir characterization in the future study of CO_2 sequestration and H_2 storage is briefly introduced. This work aims to provide references for the evaluation of shale oil and gas reservoirs and the future application of carbon neutrality in shale reservoirs.

2. Pore structure characterization of shale reservoirs

The pore structure of shale oil and gas reservoirs contains multiple information such as pore type, pore morphology, pore volume, surface area, pore/throat size distribution, tortuosity, connectivity (Glorioso and Rattia, 2012; Liu et al., 2022). Common characterization methods are exhibited in Fig. 1.

2.1 Fluid intrusion methods

Pore structure information can be quantitatively evaluated by fluid intrusion methods, which generally include helium pycnometry, fluid saturation, mercury intrusion, and gas physisorption (Anovitz and Cole, 2015; Sun et al., 2020). These methods are used to probe pore structure in shale reservoirs by injecting small molecular fluids such as helium, water, mercury, nitrogen, carbon dioxide, argon, etc.

2.1.1 Fluid immersion porosimetry

Fluid immersion porosimetry is the most fundamental and effective method to determine the porosity (Wang and Cheng, 2023). It only requires the determination of the grain density and bulk density of the samples. Fluids such are helium, water, and organic fluid are first saturated into the edge-accessible pore spaces to determine the grain density (ρ_1). Bulk density (ρ_2) is determined based on Archimedes' principle or volume measurement. Finally, the porosity (φ) of the sample is obtained by:

$$\varphi = 1 - \frac{\rho_2}{\rho_1} \tag{1}$$

The fluid immersion porosimetry method can measure the effective porosity of shale but cannot obtain the distribution of pore volume with pore size.

2.1.2 Mercury intrusion porosimetry

Mercury intrusion porosimetry uses no-wetting mercury as a probe to measure porosity, and pore-throat diameter distribution to the edge-accessible pore spaces in the scale range of greater than 3 nm (Webb, 2001; Mastalerz et al., 2021). Beside the porosity and pore-throat size distribution measurement, mercury intrusion porosimetry can estimate permeability, surface area, touristy, and particle size (Gao and Hu, 2023). In addition, for shale reservoir, some scholars have developed repeated and directional mercury intrusion porosimetry experiments to quantitatively characterize pore connectivity and anisotropy of microfractures (Zhao et al., 2023). For the samples used in mercury intrusion porosimetry test, sandstone samples with good pore connectivity are usually prepared as 1-inch diameter core plugs, while shale samples with low pore connectivity are generally prepared as cubes (1 cm^3). However, mercury intrusion porosimetry experiment can only characterize connected pores and cannot detect closed pores. Moreover, the matrix compression of plastic minerals in shale under high pressure should be considered (Sun et al., 2023).

2.1.3 Gas physisorption

This involves the use of gas probes, such as argon, nitrogen, carbon dioxide, and water vapor (Thommes et al., 2015; Yang et al., 2020), to access the nanoscale pore structure present in shales. In Fig. 1, a range of pore sizes in shales can be characterized with gas adsorption. To determine pore diameter distribution density functional theory model, Barrett, Joyner and Halenda model, and Kalvin equation are applied. It is important to note that the result of gas adsorption is influenced by the particle size of shale samples.

2.2 Radiation methods

Radiation methods are commonly used for imaging observations through the interaction of light, electrons, ions, and X-ray with the surface or the whole of the shale. Because of the difference in the principle of observation, the characterization scale and field of view of the sample are different (Fig. 1). In addition, the pore network structure of shale can be characterized by neutron and X-ray scattering and nuclear magnetic resonance of hydrogen atoms (Hosseini et al., 2021).

2.2.1 Optical microscopy

Polarized and reflective light microscopy is usually used to observe sedimentary texture, mineral type, pore size, particle size, and roundness in sandstone, carbonate rocks, and igneous rocks. The micron scale microtextural and compositional attributes of shale are commonly analyzed by optical microscopy. However, due to the resolution of optical microscopy is in the micron scale (magnified 1,000 times), it cannot identify the nanoscale pores common in shale (Fig. 1).

2.2.2 Scanning electron microscopy imaging

Advanced field emission-scanning electron microscopy (FE-SEM) produce shale images with best resolution of 1 nm and provide shape, size, and contact relationship information of pores, minerals, and organic matters (Loucks et al., 2012; Milliken et al., 2013). However, to enhance the electrical conductivity of shale surface, carbon or gold with a thickness of approximately 10 nm is coated on the surface, resulting in the masking of sub-10 nm pores in shale. In addition to electron imaging techniques, helium ion microscopy has a higher resolution than FE-SEM, especially useful for observing pores less than 10 nm in size (Fig. 1). Moreover, combined with other instruments such as argon ion milling, focused ion beam milling, and energy dispersive X-ray spectroscopy, the functions of FE-SEM are greatly improved (Curtis et al., 2012).

2.2.3 Nuclear magnetic resonance

The application of nuclear magnetic resonance to characterize shale includes the use of the T_2 spectrum to analyze the distribution of pores, and fluids (Liu et al., 2020; Elsayed et al., 2022) difficult for different wettability fluids to reach 100% saturation in mixed wettability shale, which will lead to errors in the characterization of pore structure. In addition, the nuclear magnetic resonance instrument can cooperate with other instruments such as freeze and thaw units and fluid flow displacement units with various temperatures and pressure to predict permeability and wettability (Lin et al., 2022). However, if the fluid does not fill the large pores, such as in the form of a water film, the measurement of its pore size can lead to a deviation, which increase the volume of the small pores.

2.2.4 Neutron/X-ray scattering

These are radiation-based twin methods that contain ultra-small angle scattering and small angle scattering (Xu, 2020; Hosseini et al., 2021). A combination of non-destructive ultra-small angle scattering and small angle scattering can extend the characterization range of pores in shale from subnanometer to submillimeter (0.5 nm - 20 μm) (Fig. 1). With high radiation perpetration ability, the scattering method can measure both closed and edge-accessible pores (Sun et al., 2020). X-ray scattering has the advantage of a short measurement duration of 3-5 minutes, whereas neutron scattering has the advantage of sensitivity to isotopic substitution (Wang et al., 2022). Therefore, contrast-matching fluids, such as H_2O and D_2O, C_7H_8 and C_7D_8, and CD_4 can be used to characterize the accessibility of various fluids in pores, thus evaluating the connectivity of shale pores and the occurrence of fluids under in-situ reservoir conditions.

2.2.5 Micro- and nano-CT

In the study of shale reservoir, micro-CT is mainly used to characterize micro-scale pores and fractures. For the sub-micron pores (< 1 μm) in shale, the spatial resolution is improved to 50 nm by nano-CT, but the corresponding sample size should be reduced to approximately 15 μm. When using CT to characterize the pore-fracture system of shale, some scholars will inject contrast agents (such as Wood's metal) to better visualize the pore-fracture network and evaluate the pore-fracture connectivity of shale (Zhao et al., 2020). In addition to X-ray CT, neutron CT, which is more sensitive to hydrogen and its isotopes and can provide complementary information for fluids in shale, is also being developed (Stavropoulou et al., 2019).

3. Fluid occurrence of shale reservoirs

The occurrence of fluid in shale reservoirs can be further studied based on the characteristics of pore-fracture system. The fluid in shale reservoirs is mostly composed of gas, oil, and water. The state of occurrence of shale oil affects its mobility, and its research is critical for understanding

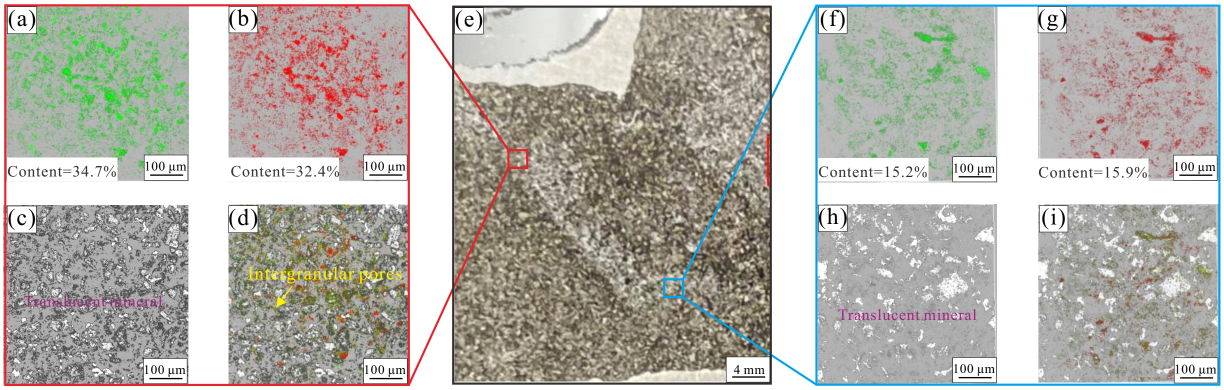

Fig. 2. Shale oil occurrence characteristics observed by LSCM images of the sandwich-type shale oil reservoirs. (a, f) Occurrence state of light components, (b, g) occurrence state of heavy components, (c, h) translucent mineral, (e)thin section photo of the sandwich-type shale oil reservoir sample, (d, i) distribution of shale oil components.

the enrichment mechanism of shale oil and optimizing the exploration plans. Shale oil mainly exists in the reservoir space in free, adsorbed, and dissolved states. For instance, in the Fengcheng Formation, shale oil occurs as adsorbed oil and free oil, and the occurrence space is mainly microfractures, dissolution pores and intergranular pores (Jiang et al., 2023). In addition, Gao et al. (2023b) innovatively used laser scanning confocal microscopy observations to posit that within the formation, shale oil occurs in a bound state and free state (Figs. 2(a), 2(b), 2(e), 2(f) and 2(g)). The bound oil is adsorbed on mineral surfaces in the form of disseminated adhesion film, attached to the surface of mineral particles in the form of particle adsorption, partially retained in a narrow throat, with a large amount of free oil enriched along microfractures (Figs. 2(c), 2(d), 2(h) and 2(i)).

Shale oil is also concentrated in clusters, gathered in intragranular pores within minerals in the laminae, occurring within the pore space via an intergranular adsorption mechanism. Gao et al. (2023b) also observed a higher shale oil enrichment in lipophilic samples, with the occurrence of heavy components controlled by wettability and total organic carbon content. The intergranular pores formed between alkaline minerals provide a certain storage space for shale oil. Shale oil enrichment is more obvious in the lamina sedimentary structure, with light and heavy components respectively distributed in the bright lamina and dark lamina. The felsic lamina is the source rock of shale oil, and the siltstone interlayer is the high-quality reservoir of shale oil.

Shale oil reservoirs are characterized by a heterogeneity in source-reservoir configurations, and oil migration scales. Gong et al. (2023) used a hydrocarbon potential, reservoir physical characteristics, and oil-bearing properties basis to categorize shale oil reservoirs of Fengcheng Formation into sandwich-type and laminated-type. The pore volume of sandwich-type shale oil reservoir is larger than that of laminated-type shale oil reservoir, and macropores are more developed in sandwich-type shale oil reservoir. Light components of sandwich-type shale oil reservoir mainly occur in a contiguous free state within the intergranular pores and microfractures and are also distributed in bright laminae of laminated-type shale oil reservoir. Heavy components are distributed on organic matter and translucent mineral surfaces in an adsorbed state.

4. The role of shale reservoirs in CO_2 sequestration and H_2 storage

Since CO_2 has a 0.33 nm kinetic diameter which is smaller than that of CH_4 (0.38 nm), has stronger adsorption capacity in nanopores, CO_2 injection into shale reservoirs helps to improve the desorption and flow capacity of residual gas and oil, to enhance the gas/oil recovery (EGR/EOR). In addition, the caprock is essential for the preventing the leakage of injected CO_2 (Shaffer, 2010). Hu et al. (2023) posited that the temperature and pressure conditions of typical geological reservoirs, the density of CO_2 is less than that of brine, which will migrate upward under the action of buoyancy. Due to ultra-low permeability, shales are suitable caprocks for the long-term effective storage of CO_2 (Zou et al., 2024). Fluid migration within the shale matrix is inhibited by the confinement effect of nanopores. Hu and Wang (2003) found that the intense movement of gas in the nanopores will reduce the diffusion rate of the gas in the porous medium. Through the CO_2 adsorption experiment, Schaef et al. (2014) posited that CO_2 molecules would form clusters in the nanopores under specific pressure (Fig. 3), with the adsorption energy between the CO_2-shale matrix increasing and changing gaseous CO_2 into a supercritical state. The viscosity of supercritical CO_2 is much higher than that of gaseous CO_2, and its diffusion rate decreases due to its liquid-like property, inhibits the leakage of CO_2 from the shale caprock. The study of confinement effect of shale nanopores is helpful to realize long-term CO_2 sequestration, especially considering the safety of underground storage of CO_2.

The upward migration of hydrogen is also inhibited by low permeable shale caprocks (Al-Yaseri et al., 2023). The confinement effect from shale nanopores accelerates the adsorption of H_2 and aids the formation of H_2 clusters in nanopores (Fig.

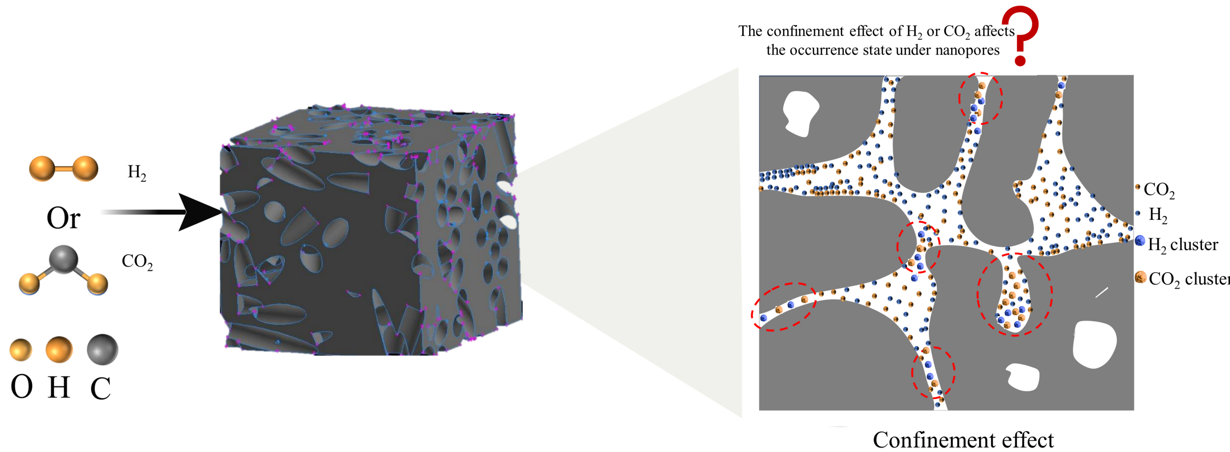

Fig. 3. CO_2 or H_2 is injected into the nanopores of shale to achieve CO_2 sequestration and hydrogen storage.

3) (Froudakis, 2011). In addition, the interaction between H_2 and nanoscale pore surfaces inhibits the diffusion of H_2 and aids long-term effective H_2 storage (Aghaei et al., 2023). The geochemical reaction and interaction between H_2 and shale with different lithofacies as caprock were investigated, and the escaping amount of H_2 on a 30-year time scale was evaluated (Zeng et al., 2023). The results showed that shale as a caprock has high stability and inhibits the upward diffusion of H_2.

5. Summary

Due to the advancements of various technologies, the pore structure of shale reservoir can be multi-scale and multi-dimensional characterized. However, the characterization of pore structure under reservoir conditions still faces challenges. It is difficult to determine whether some fractures and pores exist underground or are formed by pressure release after reaching the surface. In addition, the analysis of the state of fluid occurrence in shale pore-fracture networks under the reservoir conditions is another significant challenge, which is directly related to the evaluation of geological sweet spots. Bridging the gap between pore structure and fluid occurrence requires not only advanced techniques but also the development of modeling approaches under reservoir conditions to fully evaluate shale reservoir characteristics.

With the pursuit of decarbonization and net-zero carbon emissions, large-scale geological carbon sequestration and hydrogen storage will attract more attention. Because of its ultra-low permeability, shale is preferred as the caprock for CO_2 and H_2 storage. However, the integrity and safety of CO_2 and H_2 storage have not been systematically evaluated by considering shale pore characteristics and mineral components. In addition, the confinement effect caused by abundant nanoscale pores in shale reservoir will greatly affect the occurrence state and migration behavior of fluid in shale. Therefore, the confinement effect of nanopores in shale should be paid attention to in future research on carbon and hydrogen storage.

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Conflict of interest

The authors declare no competing interest.

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