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## Micro-occurrence of gas and water in shale nanopores

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The micro-occurrence of gas and water in shale nanopores under reservoir conditions, as well as the changes in their distribution following the infiltration of fracturing fluids, are critical for understanding the behavior and flow dynamics of gas and water during shale gas extraction. Small-angle and ultra-small-angle neutron scattering (SANS/USANS) experiments conducted under high-pressure and vacuum conditions have elucidated the occurrence state of methane within shale nanopores. Additionally, dynamic water vapor sorption measurements at varying temperatures have documented changes in water distribution within these nanopores.

To further investigate the process of fracturing fluid penetration into nanopores, a combination of field emission scanning electron microscopy (FE-SEM) and cryogenic scanning electron microscopy (cryo-SEM) was employed. These observations were performed following Wood's metal impregnation and spontaneous imbibition with deionized water. Furthermore, SANS/USANS experiments conducted under high-pressure conditions with coexisting gas and water revealed changes in micro-occurrence state of methane after fracturing fluid penetration.

SANS/USANS data collected under ambient and high-pressure conditions quantified the fraction of pores accessible to methane. The confinement effects observed indicated that the density of methane in pore spaces smaller than 20 nm under high pressure exceeded that of an ideal gas at the same pressure. This density further increased as pore sizes decreased, forming nanoscale methane clusters with densities several times higher than their inert state. Dynamic water vapor adsorption experiments demonstrated that as temperature increased, the affinity of monolayer water molecules to the shale surface diminished, reducing the formation of chemical bonds. Concurrently, the adsorption force between multilayer water vapor molecules decreased, with the maximum adsorption capacity primarily influenced by the nanopore structure.

FE-SEM observations revealed that molten Wood's metal penetrated the shale matrix from microfractures under high pressure. The extent of Wood's metal infiltration into matrix pores served as an indicator of the degree of microfracture development in the shale and the connectivity between matrix pores and microfractures. Cryo-SEM observations further demonstrated that fracturing fluid (water) infiltrated microfractures in a stratified manner due to spontaneous imbibition. Simultaneously, the fluid entered matrix nanopores in the form of water films or water bridges. Within organic matter nanopores, the fracturing fluid predominantly appeared as water films.

Based on SANS/USANS analysis, it was observed that when methane and water coexist in shale nanopores, the pores were predominantly occupied by water, which exhibits a stronger adsorption affinity than methane. Consequently, methane desorbs from the matrix pores and migrates into microfractures. This finding suggests that during hydraulic fracturing of shale gas reservoirs, a lower flowback rate of fracturing fluid is favorable for achieving higher gas production. Prolonging the borehole closure time facilitates displacement efficiency of gas and liquid within shale nanopores, thereby enhancing methane recovery rates.

### Country

China

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## **References**

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