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# Advanced 4D Imaging of Shales at Micro- to Nano-scale: Investigating the time-lapse evolution Under Subsurface Thermal, Hydrological, Mechanical, and Chemical Conditions

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Mudstones and shales are the source of unconventional oil and gas reservoirs, as well as the primary control on the sealing efficiency for subsurface storage applications (e.g. energy, CO2, H2, waste). However, their fine-grained and heterogeneous nature makes their full characterisation highly challenging. Here we demonstrate the multi-scale and dynamic imaging approaches that can help meet these challenges and discuss limitations and future opportunities.

Microstructures can be characterised at scales from sub- nm (<1 nm) to over 1 m, using multi-scale and multimodel imaging approaches [1-3], including X-ray tomography, Focused Ion Beam Scanning Electron Microscope and Transmission electron microscopy tomography. The majority of pores in mudstones/shales range from 0.2 nm to  $3\mu$ m, and we have documented 4 major types with 3 distinct size distributions [4]. Based on the REV analysis, pore sizes, types and distribution can be upscaled via three stages from sub-nm to cm-scale[5]. The permeability is pressure dependant, ranging from  $1.0 \times 10-17$  to  $1.0 \times 10-22m2$  [3, 5]. CO2 adsorption is 3-7 times higher than CH4 and over 10 times higher than H2 [6]. Image based modelling has demonstrated that the non-Darcy effects (e.g., slip flow and Knudsen diffusion). Adsorption/desorption and surface diffusion takes major controls over time after injection [7].

Dynamic imaging of mudstones/shale has provided the opportunity to characterise the thermo-hydro-mechanicalchemical (THMC) properties and the coupling mechanism in mudstones/shales to investigate the sealing ability under realistic reservoir conditions. These include high temperature (from less than 10 °C up to 1000 °C) [8], high pressure (e.g. confining pressure, indentation, torsion, deformation and fractures; up to 65 MPa) [9], fluids (e.g. diffusion, adsorption, flowing through, multi-phase flow) [10] and complex chemistry environment (brine and drilling fluids) [11]. Based on the dynamic behaviours observation and quantification, It can be concluded that mudstone/shales with horizontally thin-layered laminations, few fractures and less reactive minerals may act as the best caprocks.

Whilst the above has led to an improved understanding of shale/mudstone microstructure under static and dynamic conditions, significant challenges still remain regarding representivity and up-scaling, experimental analysis at subsurface-realistic temperatures, pressure and chemistry, accurate estimations of the long-term behaviours and the proper monitoring techniques.

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### References

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