

In Situ Contact Angle Measurements of Supercritical CO₂, Brine, and Sandstone Cores Using Micro-CT Imaging

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Accurate descriptions of contact angles and fluid wettability are crucial to the understanding of multiphase behavior and storage efficiency factors for potential Geologic Carbon Storage (GCS) reservoirs. The contact angle is the angle between the fluid-fluid interfaces with a solid. Contact angle measurements on non-flat surfaces are challenging and not traditionally obtained. Tests are underway at the U.S. Department of Energy National Energy Technology Laboratory (NETL) building upon previous experiments that seek to utilize micro-CT scans to visually measure contact angles and determine how samples behave under various pressure, temperature, and geochemical settings. Before and after three-dimensional (3D) images of a sample at residual CO₂ saturation reveal the space occupied by supercritical CO₂ (scCO₂). These volumes of CO₂ are isolated and the contact angle of the CO₂ is shown to be variable within this space due to additional forces acting upon the trapped bubbles. Careful selection of a region of interest for measuring contact angles is of utmost importance to extrapolate a representative population of contact angles from a single volume [1]. This research incorporates conditions more representative of those found in a GCS reservoir to determine accurate contact angle measurements.

Two tests were completed, one with a Berea sandstone core and the second with a hybrid rock system. The hybrid was comprised of a three layered system with sand from the CO₂ injection zone of the Mount Simon formation (Illinois Basin–Decatur Project) loaded between two Berea cores. The focus of the second experiment was to determine differences/similarities in how scCO₂ interacts with this sand compared to the Berea sandstone. The core was initially saturated with a 10% KI brine within a beryllium pressure vessel mounted within the XRadia Micro-CT scanner. The system inside the CT scanner was elevated to the representative *in situ* pressure of 1800 psi at room temperature (73°F). Four pore volumes of liquid CO₂ and a second brine flood of four pore volumes were injected (flow rate of 0.05 ml/min) to trap and isolate CO₂ bubbles in the pore space. After the second brine flood, the temperature within the core was raised from room temperature to 115°F to attain *in situ* temperature conditions. Subsequently, two scans of the trapped scCO₂ bubbles in the pore were completed. Next, the temperature of the system was lowered back to room temperature to image the same core regions now containing liquid CO₂. All scans were completed at 2.36 μm resolution allowing a direct comparison between the scCO₂ and liquid CO₂ phases within the rock matrix.

Results show scCO₂ at *in situ*, static conditions continued to migrate over a three day period, further saturating the pore space with time (**Figure 1**). When the temperature was lowered, returning the scCO₂ to a liquid CO₂ phase, additional fluid migration was observed (**Figure 2**).

Ten Regions of Interest (ROI) were obtained for image analysis. Basic two-dimensional (2D) angle measurements were performed on each scCO₂ ROI bubble using the ImageJ Angle Tool (**Figure 3**). The preliminary results show a contact angle of 40.8° ± 5.0° for the scCO₂-brine-Berea interface. For more

extensive analysis, a 3D surface/directional tensor will be obtained and used to measure the angle from the surface line of the void. This will be contrasted to a known surface area or volume to compare to processed data.

References:

[1] Tudek *et al*, *In situ* contact angle measurements of liquid CO₂, brine, and Mount Simon sandstone core using Micro X-ray CT imaging, Sessile Drop, and Lattice Boltzmann Modeling: Journal of Petroleum Science and Engineering special issue (2017), p. 1.

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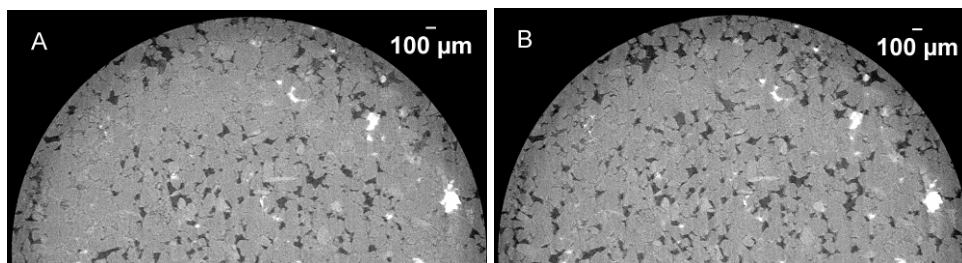


Figure 1. 2D 16-bit Grayscale Images of a Berea Sandstone Core with A) scCO₂ scanned on day 1 and B) scCO₂ scanned on day 3. scCO₂ movement was observed between the imaging of A and B. During this time, no flow was applied, only constant pressure at the elevated temperature.

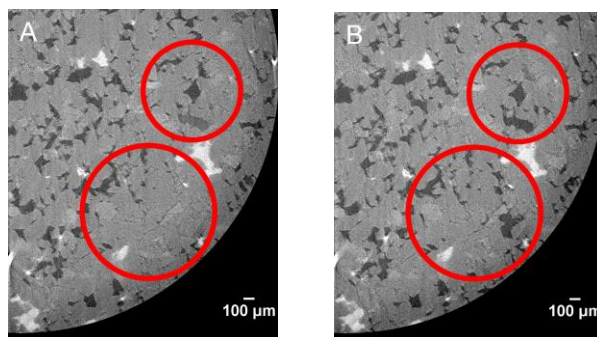


Figure 2. 2D 16-bit Grayscale Images of a Berea Sandstone Core with A) scCO₂ and B) liquid CO₂. The movement of the CO₂ can be observed between A to B, where the liquid CO₂ migrated after the temperature was lowered.

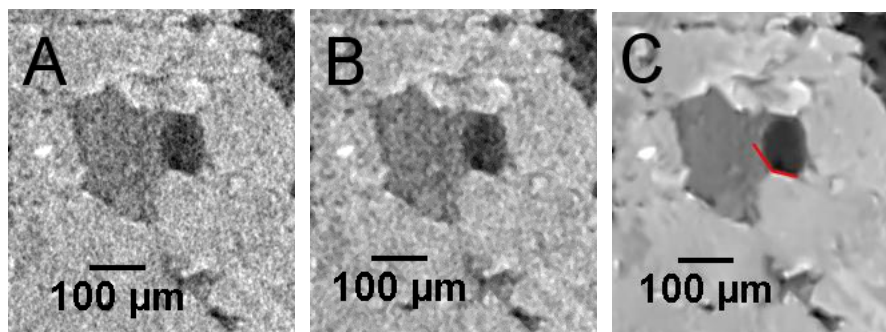


Figure 3. Contact Angle Analysis: A) Original Image, B) Despeckle, and C) Edge-Preserving Non-Local Means Filter.