Tracer-Guided Characterization of Dominant Pore Network and Implications on Permeability and Wettability in Shale*

Sheng Peng¹, Robert M. Reed¹, and Xianghui Xiao²

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Abstract

Pore network characterization is an important aspect in unconventional reservoir evaluation. A common technique is using the scanning electron microscope (SEM). While application of this technique brings substantial advances in pore characterization in shales, understanding the connected pore network that dominates flow in shale samples is limited by using SEM alone because of small fields-of-view and lack of views of connectivity in 3D. In this research, a new technique integrating tracer imbibition, micro-CT imaging, and SEM imaging was developed to provide a "real" solution for multiscale imaging in shale. Tracer imbibition indicates pore connectivity; micro-CT imaging after tracer imbibition thus provides an overview of the connected pore network at the millimeter scale. With guidance from micro-CT images after tracer imbibition, a more accurate and detailed characterization of pore systems and related mineralogy can be conducted using higher-resolution SEM. Therefore, this integrated method provides a way of simultaneous viewing of the overall framework of the dominant pore network and details of the pores. The method was applied to five samples from Wolfcamp and Eagle Ford Formations. Results reveal the effectiveness of the new method by showing different patterns of distribution of the dominant pore network and different controlling mineralogy. Dominant porosity, estimated from grayscale analyses, displays a good correlation with permeability. This result indicates that dominant porosity is more relevant to permeability than total porosity. Results from imbibition tests and micro-CT imaging are compared with that from contact angle measurement. Important implication for wettability can be obtained from the comparison. Contact angle measurements characterize the surface wettability and the results are consistent with the imbibition behavior in microfractures, whereas local pore wettability is more complicated and is affected by local surrounding mineral phases or organic matter.

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Peng, S., B. Ren, and M. Meng, 2019a, Quantifying the Influence of Fractures for More Accurate Laboratory Measurement of Shale Matrix Permeability using a Modified Gas Expansion Method: SPE Reservoir Evaluation & Engineering, Formation Evaluation, SPE-195570-PA, 12 p. doi.org/10.2118/195570-PA

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Peng, S., R.M. Reed, X. Xiao, Y. Yang, and Y. Liu, 2019b, Tracer-Guided Characterization of Dominant Pore Networks and Implications for Permeability and Wettability in Shale: Journal of Geophysical Research - Solid Earth, v. 124/2, p. 1459-1479. doi:10.1029/2018JB016103

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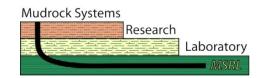
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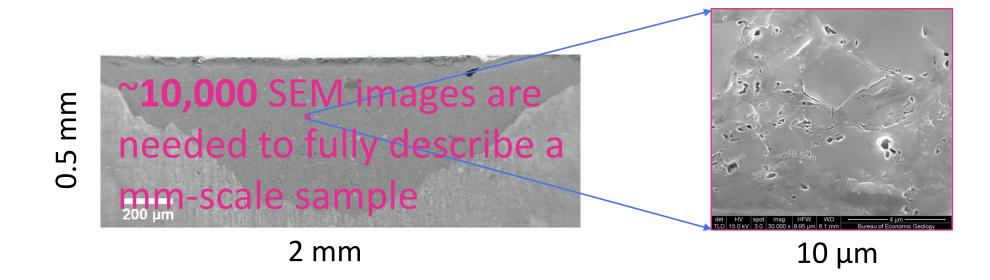






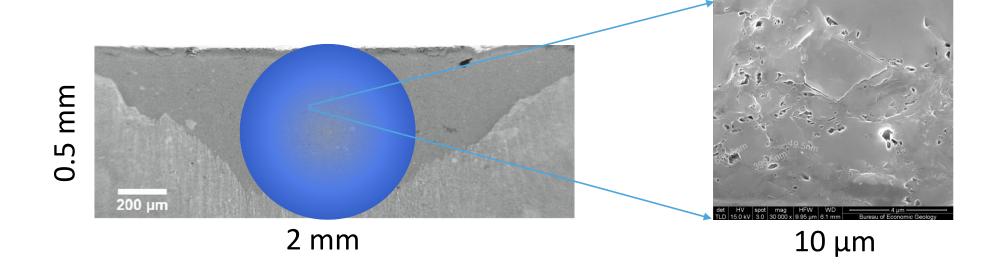
Motivation

- SEM is an important technique for pore network characterization
- However, it is challenging to evaluate the "big picture" of the pore network using SEM alone
- It is also difficult to evaluate pore connectivity using SEM

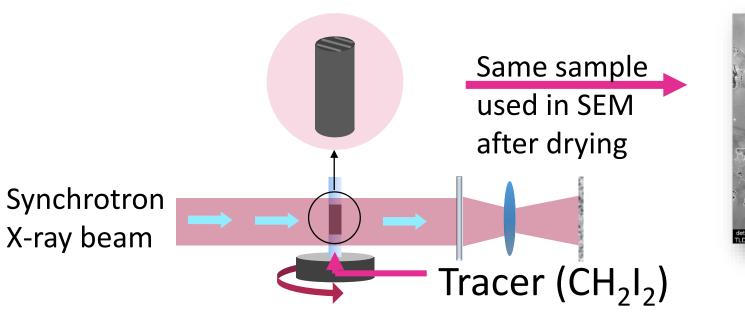


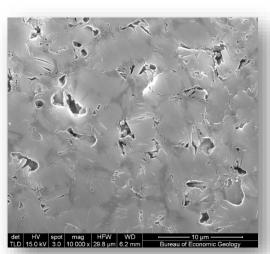
Motivation

- The sessile drop method measures contact angle of the sample surface
- The contact angle may not represent pore wettability
 - Pore surface ≠ sample surface



An integrated technique of tracer imbibition and multi-scale imaging





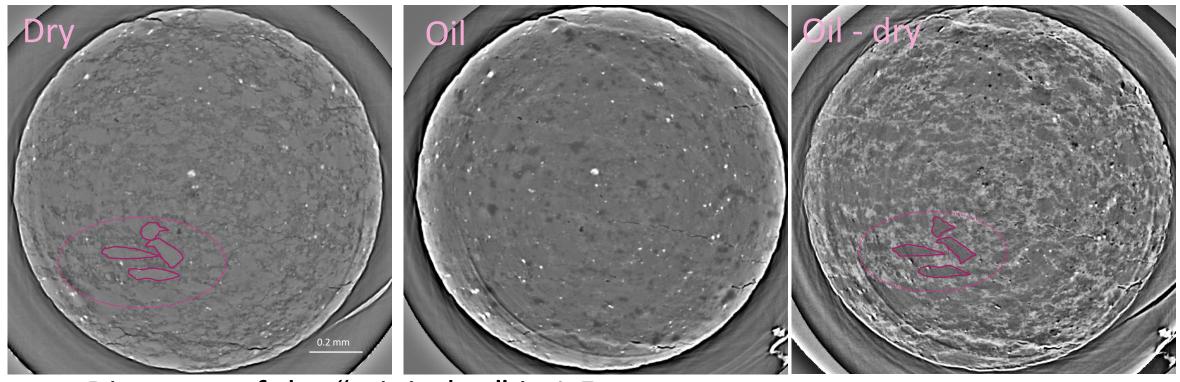
- Tracer (CH₂I₂) imbibition + micro-CT + SEM/EDS
- CH₂I₂: an oil-phase tracer, non-polar

Samples from Wolfcamp and Eagle Ford Shale

Sample	Formation	Depth (ft)	Mineralogy (XRD or XRF) facies
W1	Wolfcamp	10773	Calcareous mudstone
W2		10486	Siliceous mudstone
W3		10505	Siliceous mudstone
E1	Eagle Ford	12308	Calcareous mudrock (marl)
E2		11943	Calcite-rich argillaceous mudrock

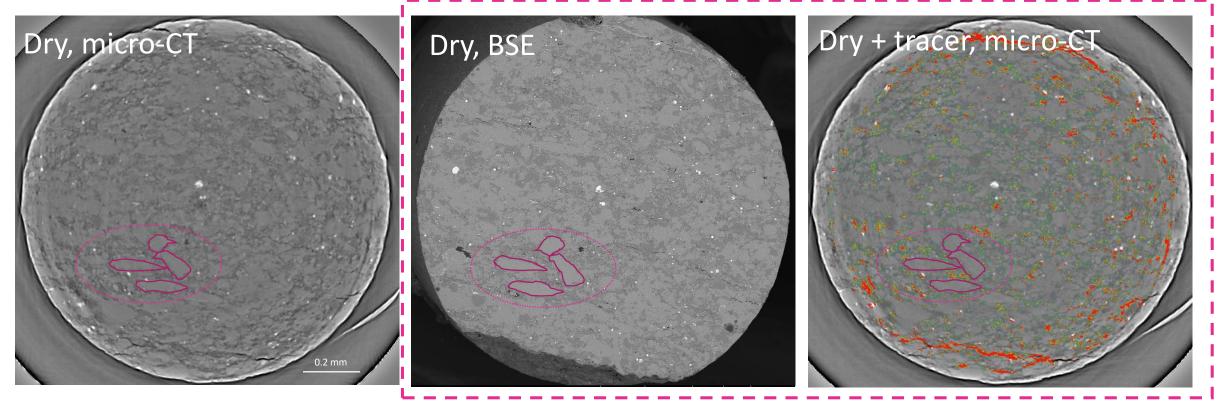
• Different or same lithofacies based on mineralogy

Results of Sample W1: Micro-CT



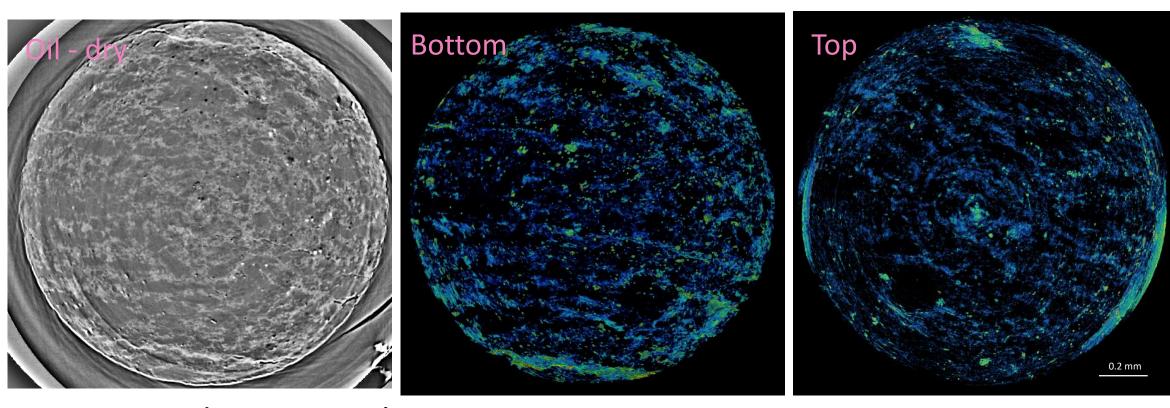
- Diameter of the "mini-plug" is 1.5 mm
- Micro-CT images have pixel resolution of 0.65 μm
- The difference image (oil dry) shows areas with tracer imbibition
 - Lighter areas indicate tracer imbibition
 - Darker areas indicate no tracer imbibition

Tracer-Guided Comparison: Micro-CT vs. BSE



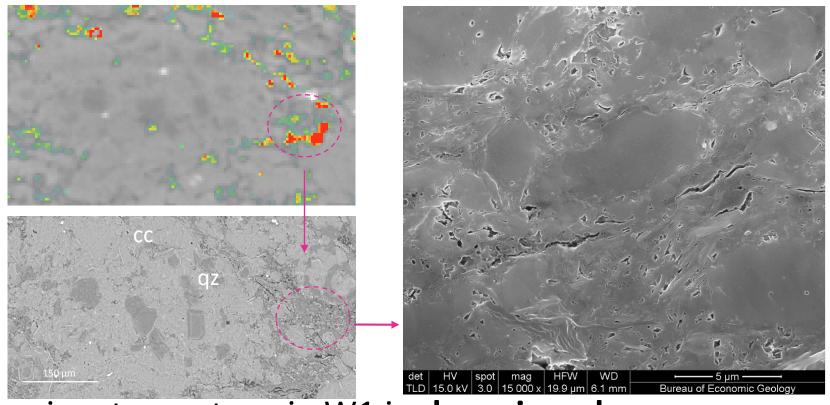
- Precisely aligned micro-CT and BSE images
- The registered micro-CT image (tracer + dry) provides a guidance for further characterization with SEM

Dominant Pore Network in W1



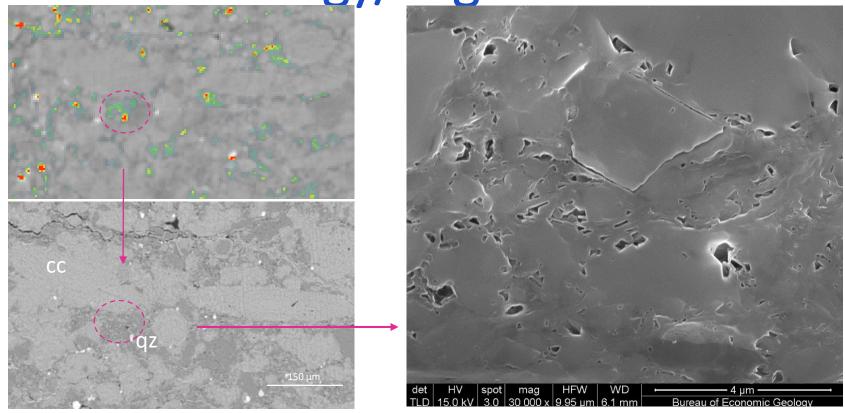
- Has evident grayscale contrast
- Has relatively uniform distribution in mm-scale
- Persists from bottom to top of the sample (5 mm)

Tracer-Guided Characterization of Pores and Local Mineralogy/Organic Matter



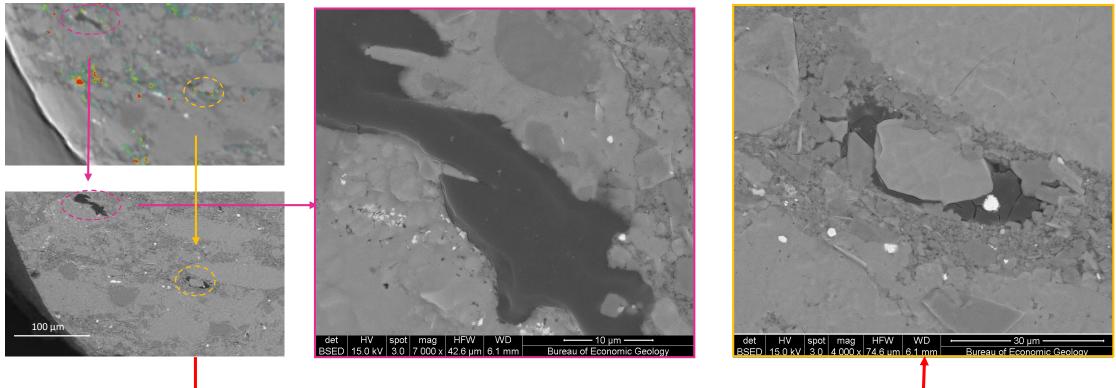
- Dominant pore type in W1 is clay mineral pores
- Which are distributed in "clay mineral clusters" between larger calcite and quartz grains

Tracer-Guided Characterization of Pores and Local Mineralogy/Organic Matter



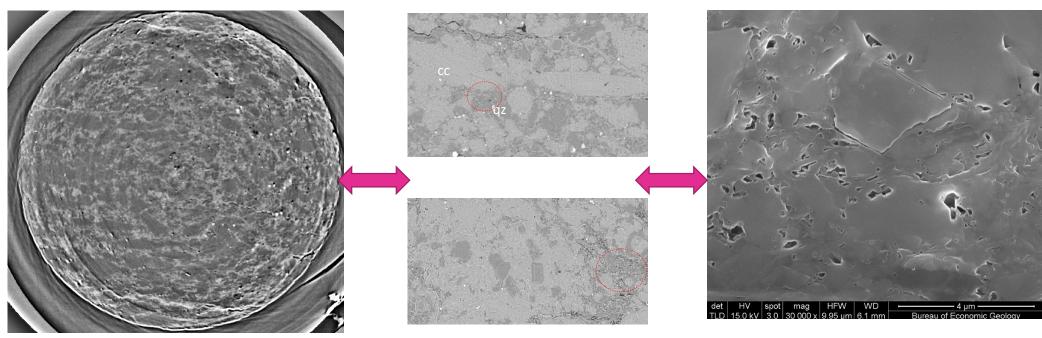
- Dominant pore type in W1 is clay mineral pores
- Which are distributed in "clay mineral clusters" between calcite and quartz grains

Tracer-Guided Characterization of Pores and Local Mineralogy/Organic Matter



- Kerogen with no pores shows no tracer imbibition
- Organic-matter pores are not important in W1

The Integrated Method Facilitates Multi-Scale Characterizations of the Dominant Pore Network

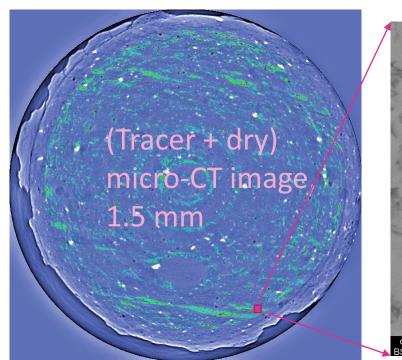


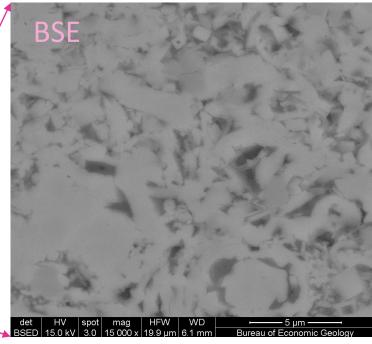
Dominant pore network in **mm-scale**

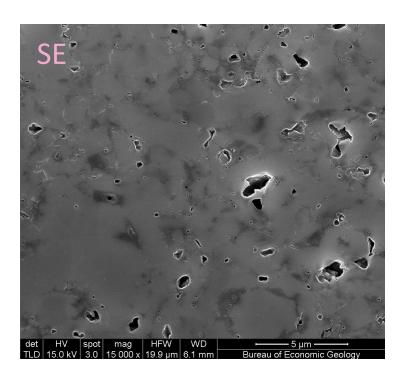
Local mineralogy
(µm-scale)
surrounding the
pores

Pores (nano-scale) within the dominant pore network

Results of An Eagle Ford Shale Sample (E1)

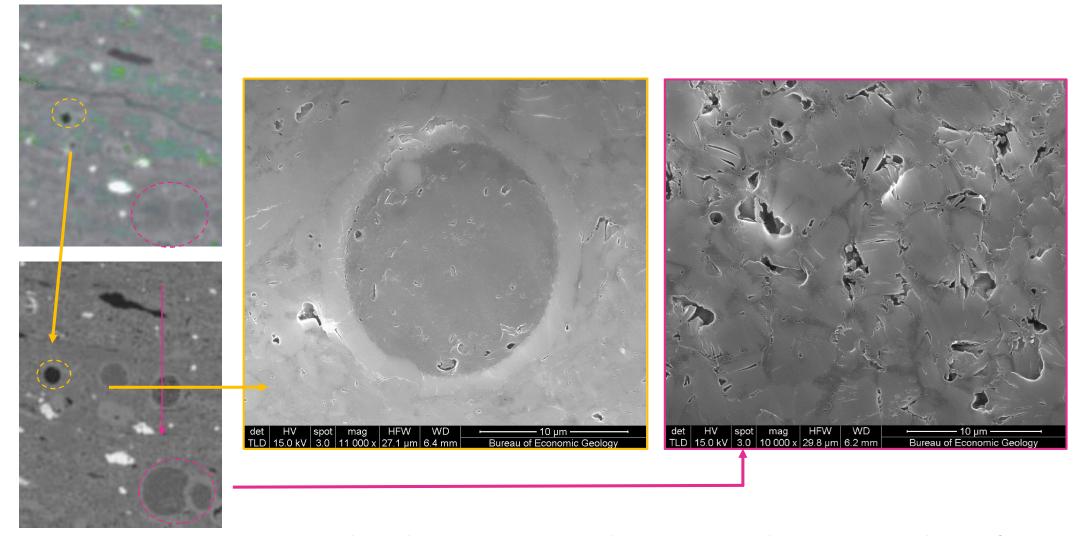






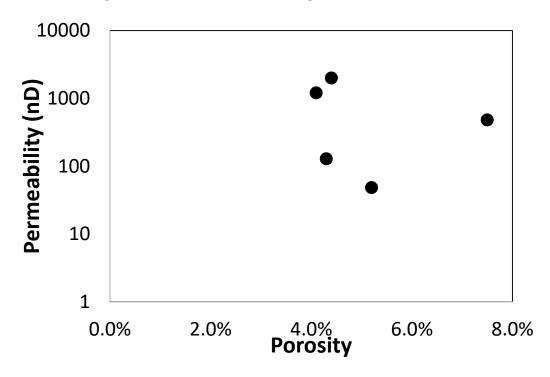
- Dominant pore network has layered distribution
- The layers are coccolith-rich lens
- Dominant pore type is interparticle pores with organic matter boundaries between coccolith pieces

Pores without Tracer Imbibition in E1



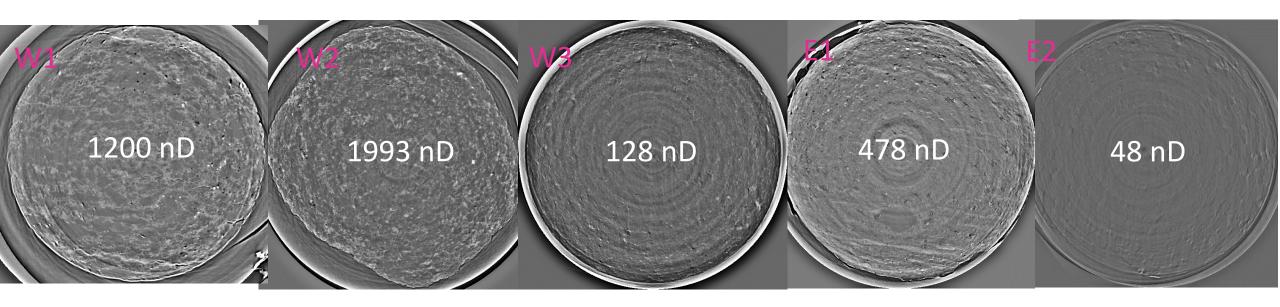
- Many pores exist within bitumen and between clay minerals in forams
- No (evident) imbibition indicates poor connectivity of these pores

Permeability-Porosity Relationship



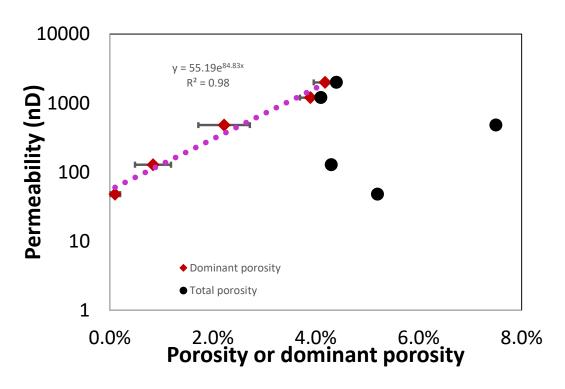
- Porosity and permeability were measured using a modified gas expansion method (Peng et al., 2019a)
- No correlation between matrix permeability and porosity

Consistency between Tracer Imbibition and Permeability



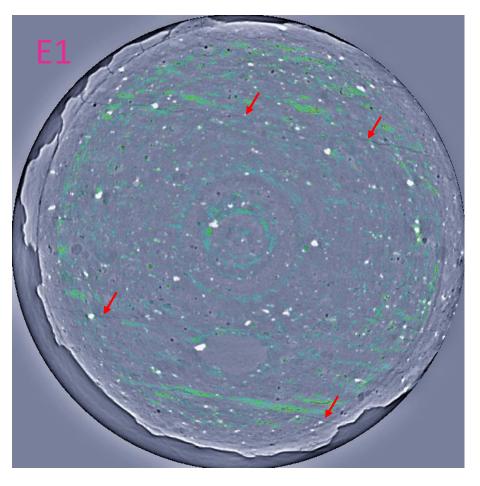
- Samples with higher permeabilities show more tracer imbibition
- **Dominant porosity** can be estimated based on grayscale contrast in the micro-CT images and the measured total porosity (Peng et al., 2019b)

Improved Permeability-Porosity Relationship



- A positive correlation exists between permeability and dominant porosity
- This indicates dominant pores (relatively larger and more denselydistributed) are more relevant to flow

Surface, Pore, and Microfracture Wettability



- Contact angle using the sessile drop method of the oil is 86°, meaning the sample surface is neutrally-wet to oil (CH₂I₂)
- Tracer imbibition occurred in pores within coccolith layers → pores are oil-wet
- The contradictory results indicate that surface wettability ≠ pore wettability in shale,
- which is because that the bulk mineralogy and surrounding mineralogy (or organic matter) of pores are different
- However, surface wettability can be consistent with microfracture wettability

Conclusions

- The integrated method of tracer imbibition and imaging provides a path for multiscale characterization of the dominant pore network in shale
 - The characterization provides new insights on <u>pore network</u> distribution in mm-scale, <u>local mineralogy surrounding pores</u> in <u>µm-scale</u>, and <u>details of the dominant pores in nm-scale</u>
- Dominant porosity is more relevant to permeability than total porosity
- Pore wettability is different from surface wettability in shale, whereas "fresh" microfracture wettability can be consistent with surface wettability

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- Peng, S., B. Ren, M. Meng, 2019a, Quantifying the Influence of Fractures for More Accurate Laboratory Measurement of Shale Matrix Permeability using a Modified Gas Expansion Method, SPE Reservoir Evaluation & Engineering-Formation Evaluation, SPE-195570-PA
- Peng, S., R.M. Reed, X. Xiao, 2019b, Tracer-guided characterization of dominant pore networks and implications for permeability and wettability in shale. Journal of Geophysical Research – Solid Earth, DOI:10.1029/2018JB016103
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Thank you for attention!

Questions?

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