Upscaling of Pore Network and Permeability from Micron to Millimeter Scale in Organic-Pore Dominated Mudstones*

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Abstract

Pore-scale imaging and permeability modeling provide an important pathway to understanding mudstone pore structure and network, which is critical for efficient shale gas and oil production. The technique for 3D nm-scale pore characterization, FIB/SEM, however, can only address a very small volume, usually a few microns, of rock matrix. Given the known heterogeneities of mudstones, data from small volume FIB/SEM studies are not adequate for representative characterization of pore structure and pore-permeability relationships. We propose an improved strategy for upscaling pore networks and permeability in organic-pore dominated mudstones from the µm to mm scale.

The Barnett Shale, in which as much as 90% of the pore volume is developed in organic pores, was sampled and milled using a FIB/SEM as an example. 3D image analysis was then conducted to characterize the pores, including porosity, pore size, geometric tortuosity, and pore connectivity. The resulting porosity and pore size distribution are in good agreement with previous findings based on SEM image analysis. Permeability, which was calculated from pore-scale modeling, displays micro Darcy values for local pore clusters. Kerogen particle size, connectivity, and distribution were also analyzed using FIB/SEM images. These data show that large kerogen particles, with sizes > 0.5 µm, are more commonly connected than smaller particles, which are more likely to be isolated. Therefore, a 3D connected-kerogen-particle network (in mm-scale) can be defined with micro-CT analysis (at a resolution of 0.5 µm). The kerogen network is the fundamental framework of the connected organic pore network, since organic pores can be connected only if the surrounding organic matter (kerogen) is connected. The integration of nm-scale pore FIB/SEM analysis and mm-scale CT analysis can produce an organic pore network model that can form the basis for understanding and characterizing pore structure and permeability. This upscaling strategy has the potential to further the understanding of pore network and pore-permeability relationships of mudstones, facilitating more efficient shale oil and gas exploration and recovery.

Reference Cited

Loucks, R.G., R.M. Reed, S.C. Ruppel, and D.M. Jarvie, 2009, Morphology, genesis, and distribution of nanometer-scale pores in siliceous mudstones of the Mississippian Barnett Shale: JSR, v. 79/12, p. 848-861.

Up-scaling of Pore Network and Permeability from Micron to Millimeter Scale for Organic-Pore Dominated Mudstones

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Jijin Yang, Carl Zeiss Microscope
Xianghui Xiao, Advanced Photon Source
Bob Loucks, Steve Ruppel, Tongwei Zhang, BEG

Workflow

- Pore and Pore network characterization
  - Organic matter associated pore distribution using FIB/SEM: μm-scale;
  - Organic matter particle (OM) distribution using μCT: mm-scale;
  - Small pore imaging using Helium Ion Microscopy (HIM): nm-scale;

- Permeability estimation
  - A conceptual model based on image analysis and mercury intrusion;
  - Permeability simulation in μm-scale;
  - Up-scaled permeability estimation in mm-scale.

The Sample
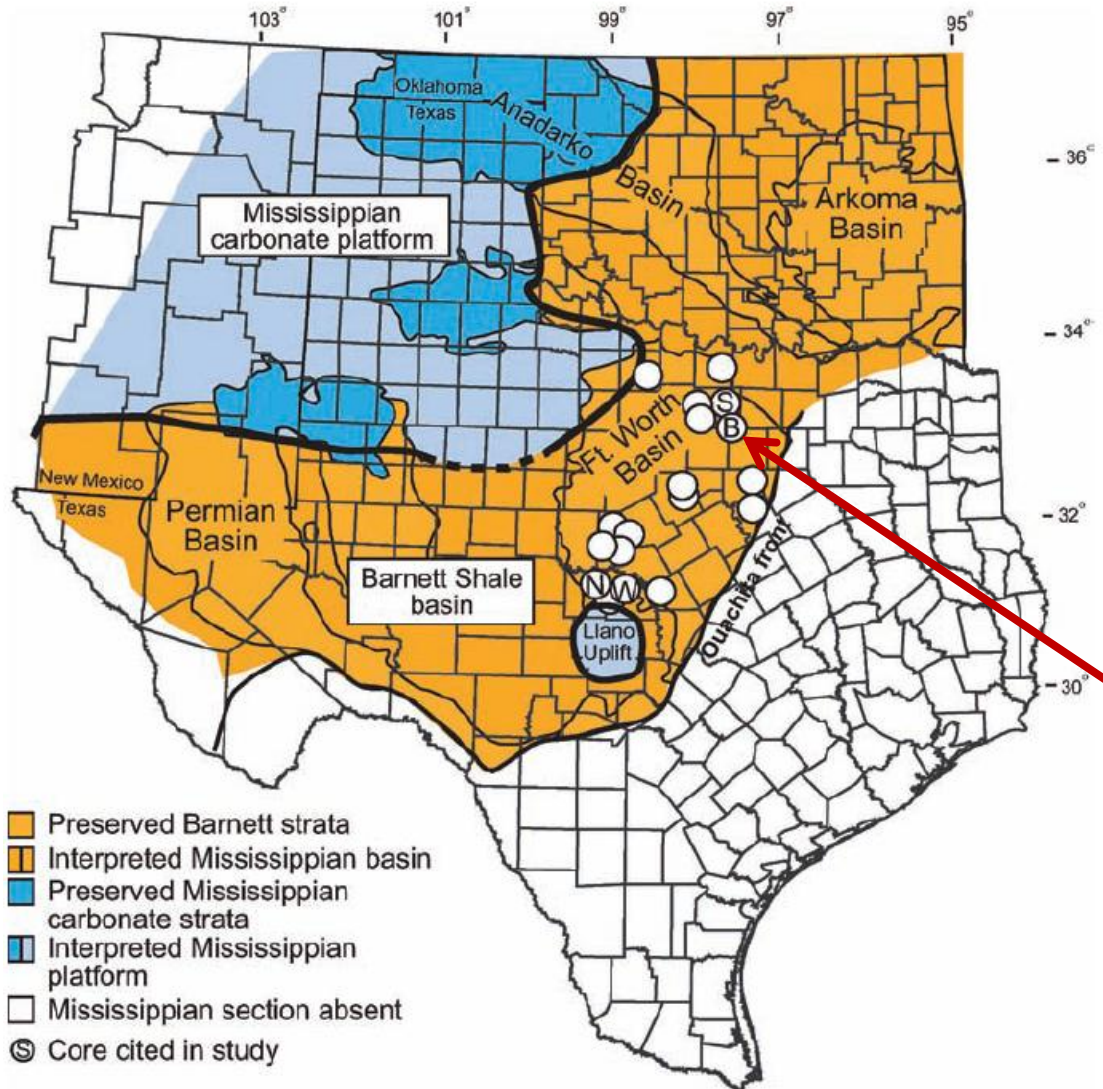
Barnett shale

Barnett: Blakely 7111' (2167 m)
TOC: 4.05% on average
Ro: ~1.35%
MICP porosity: 3.2%
Organic pore: > 90%

FIB/SEM 3D Volumes
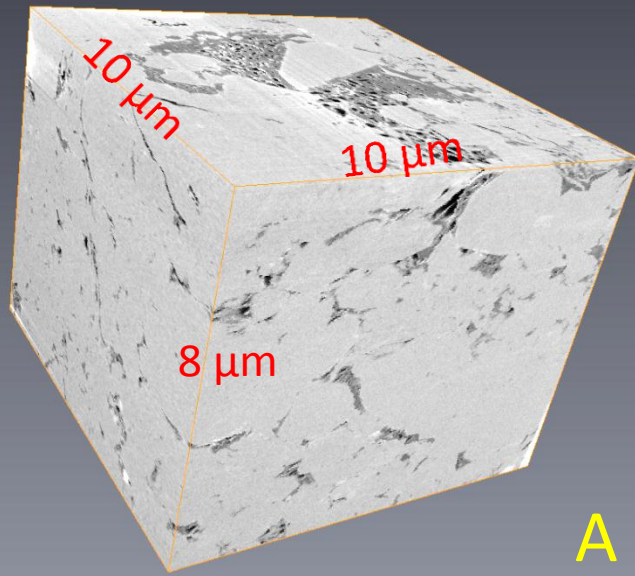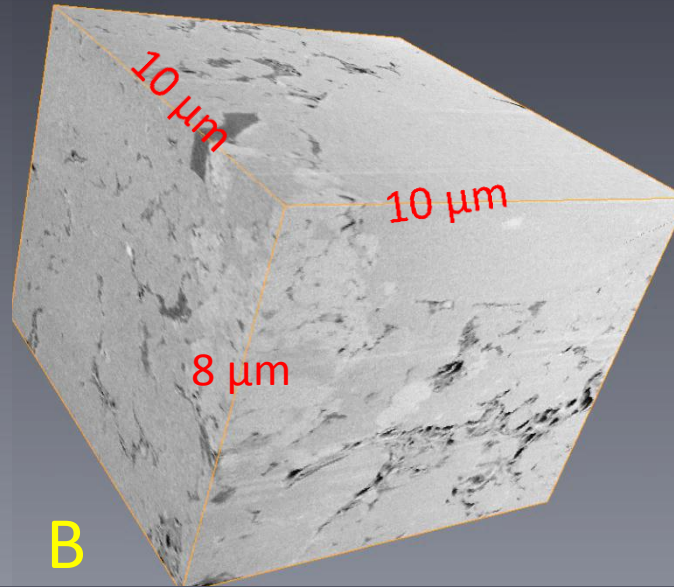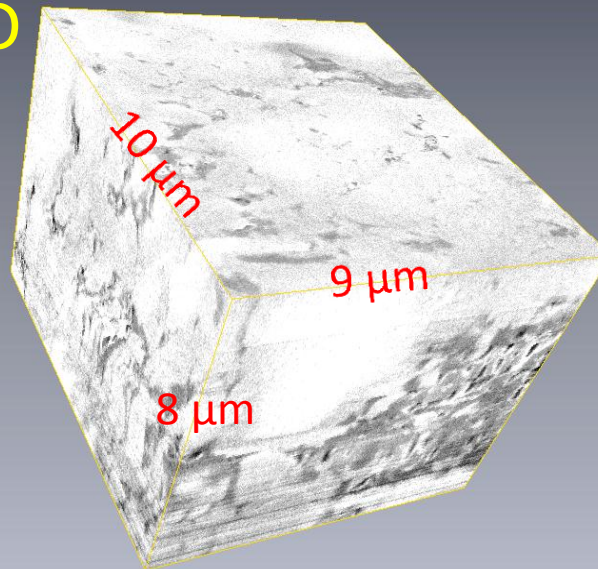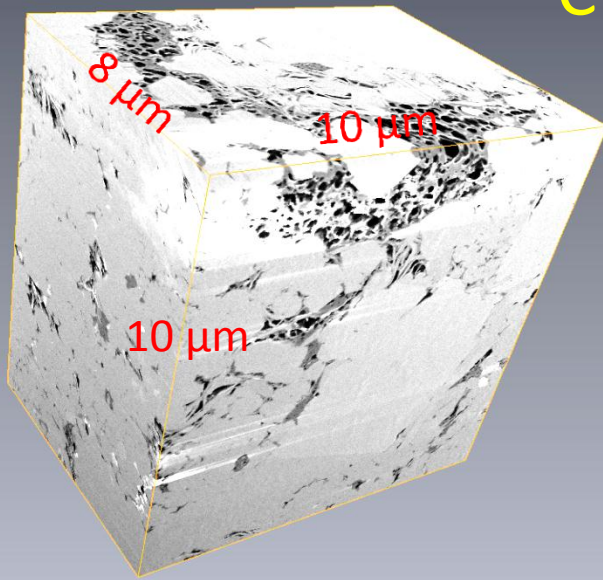
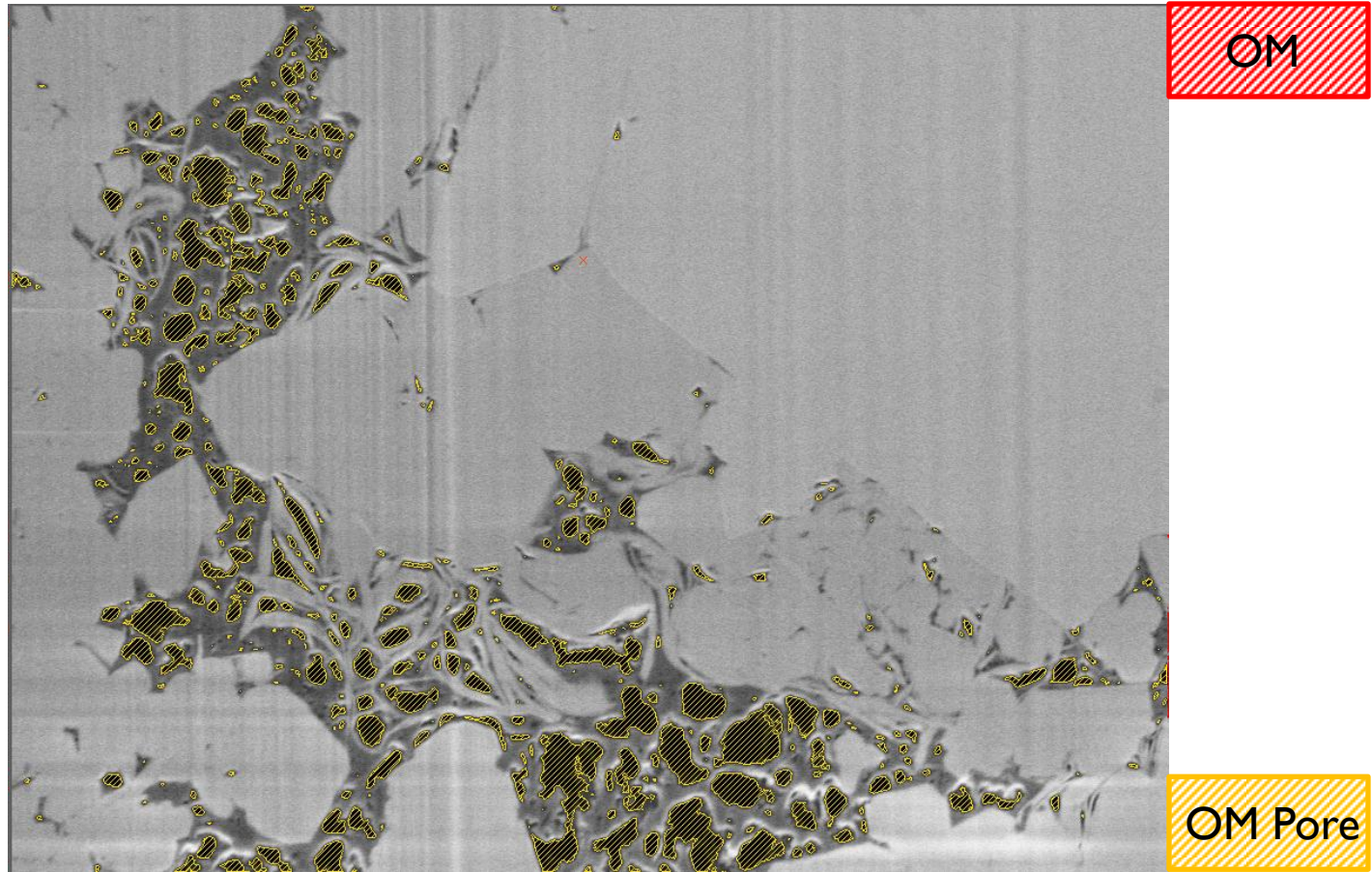4 volumes (sub-samples) from the same core sample;

Voxel resolution:

A, B: 24.5 x 24.5 x 29.5 nm

C: 10 x 10 x 10 nm

D: 20 x 20 x 45 nm

Avizo Fire (VSG)
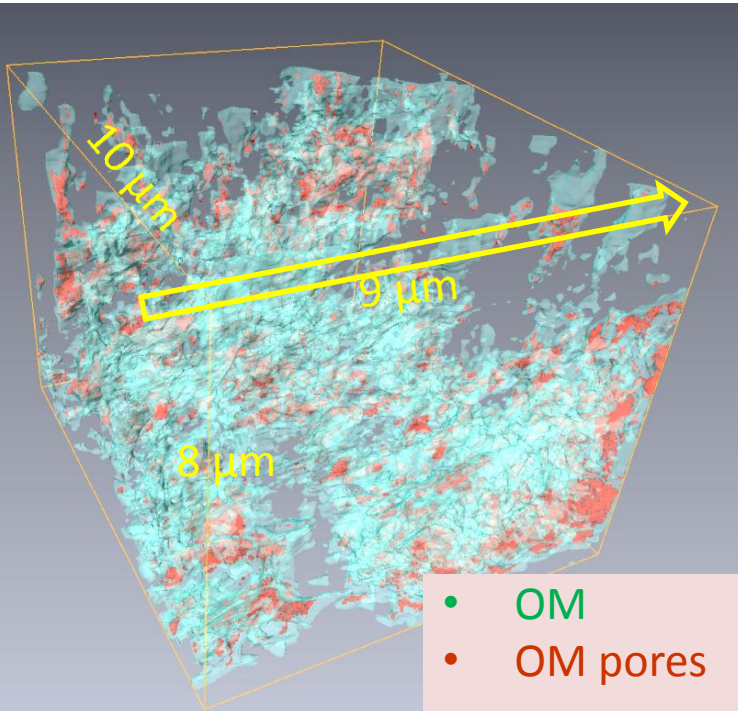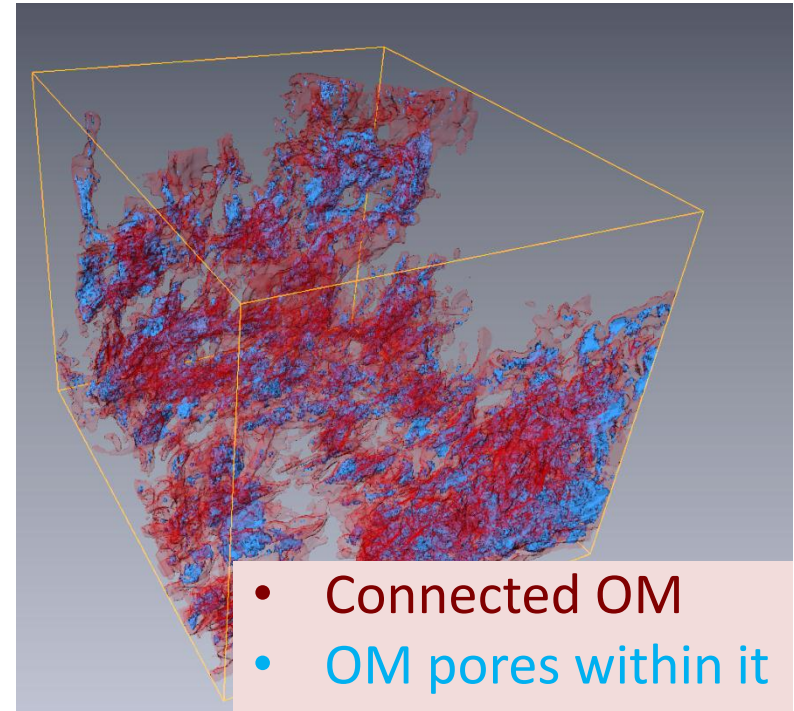
Organic Matter (OM) and OM Pore Segmentation

OM

OM Pore

10 μm

- Multi-step segmentation + modification

3D Organic Matter (OM) and OM Pore Reconstruction

All OM

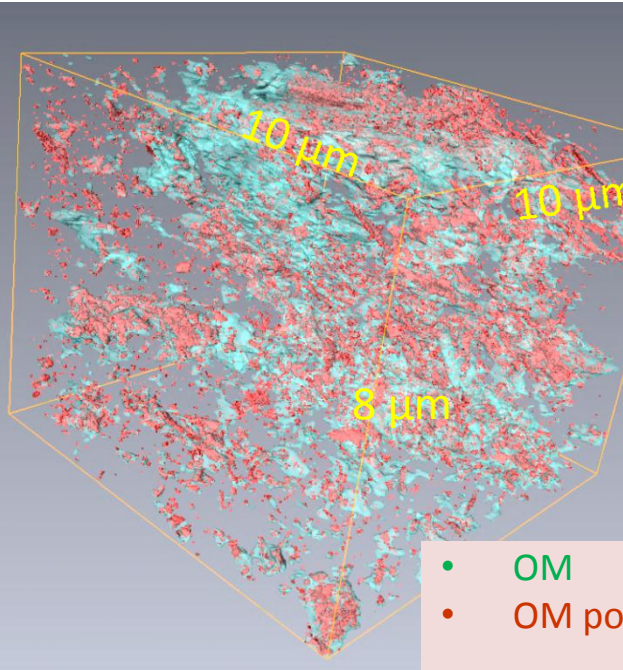- OM
- OM pores

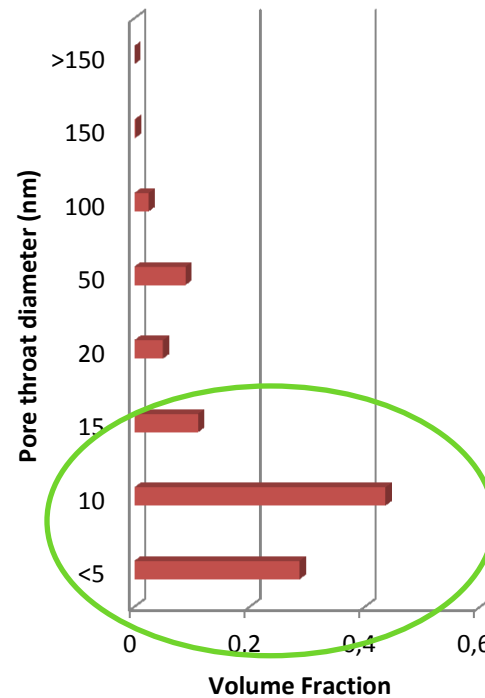## Connected OM

- Connected OM
- OM pores within it

- Connected organic matter exists
- Only locally connected pore network,
- but no throughout connected pore network…

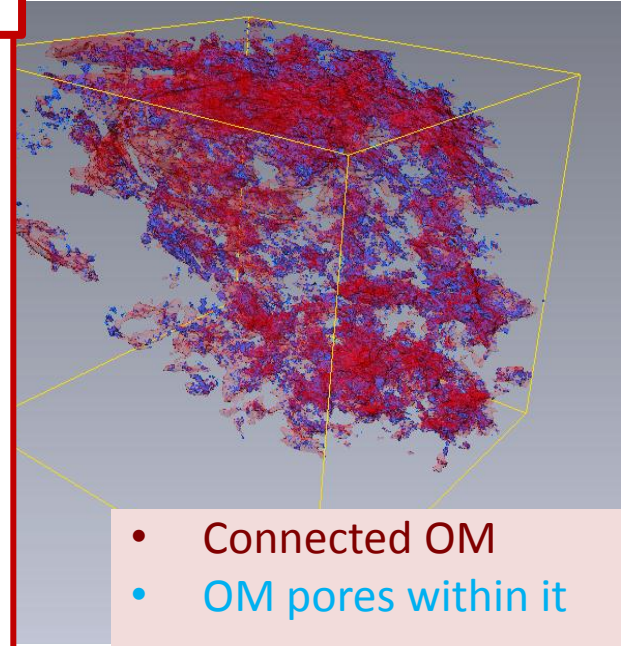# 3D Organic Matter (OM) and OM Pore Reconstruction

All OM

- OM
- OM po[res]

Connected OM

- Connected OM
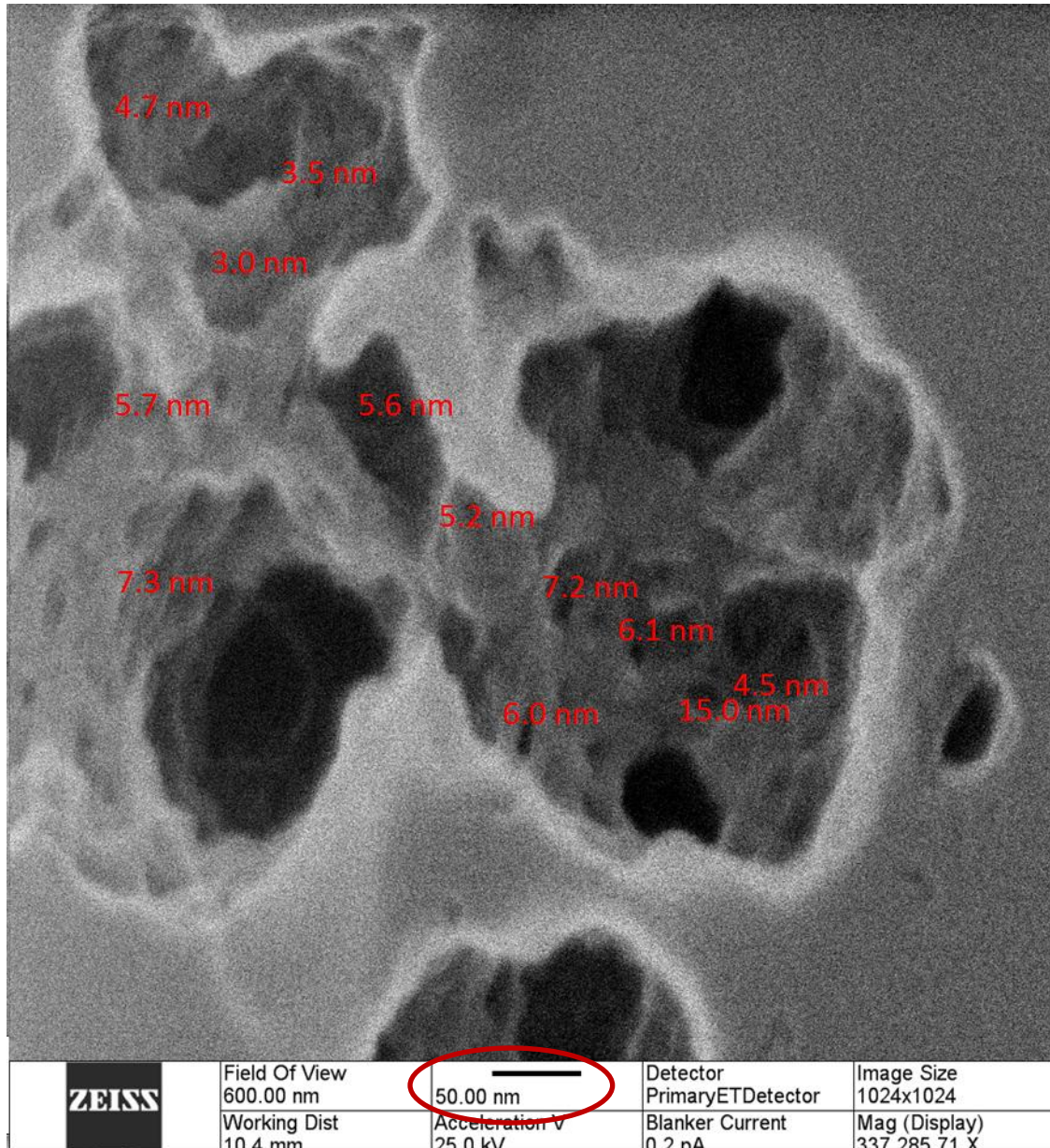- OM pores within it

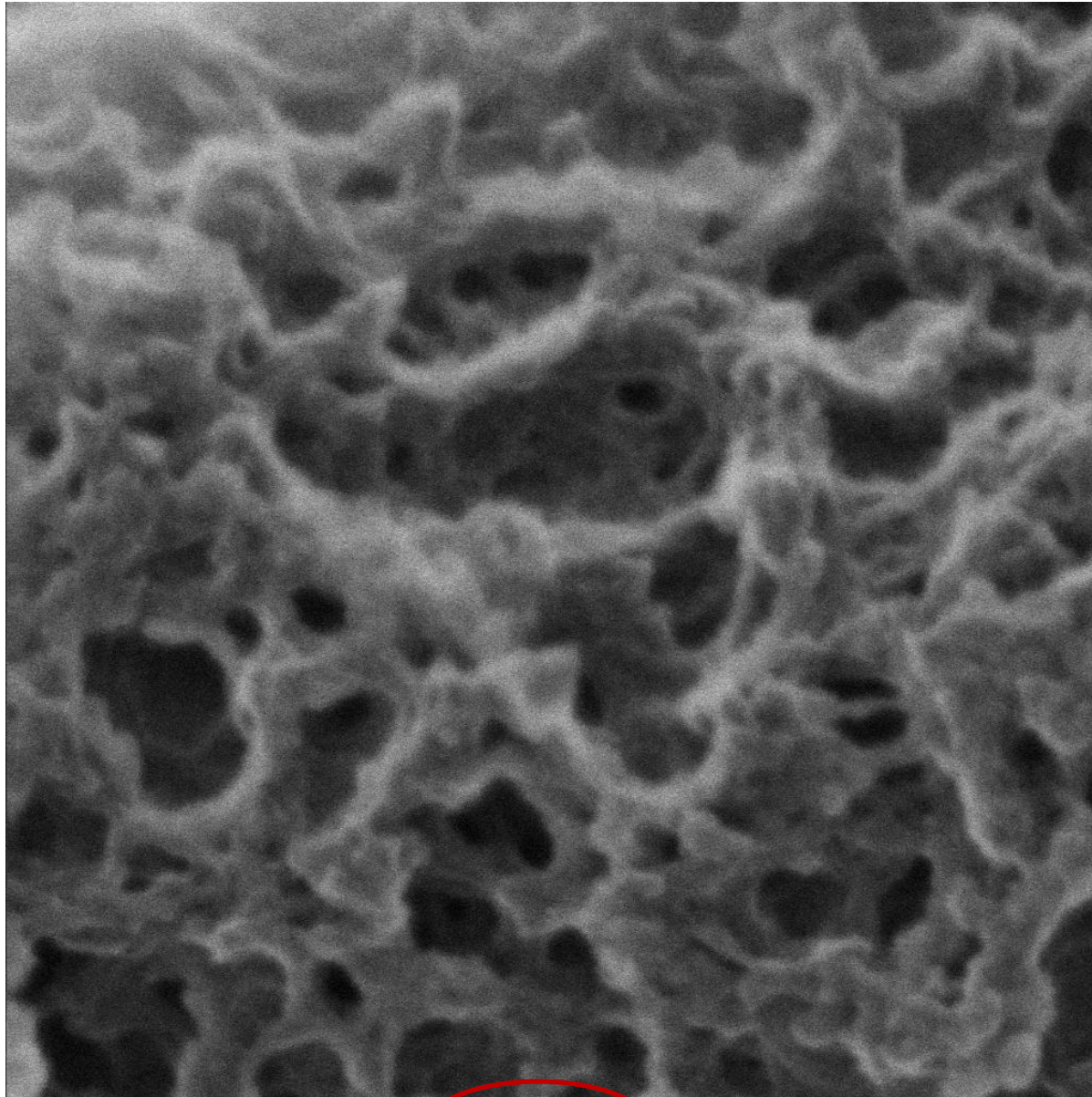### Mercury intrusion pore throat size distribution

- Small pores not shown up under the FIB/SEM resolution can play an important role in connection
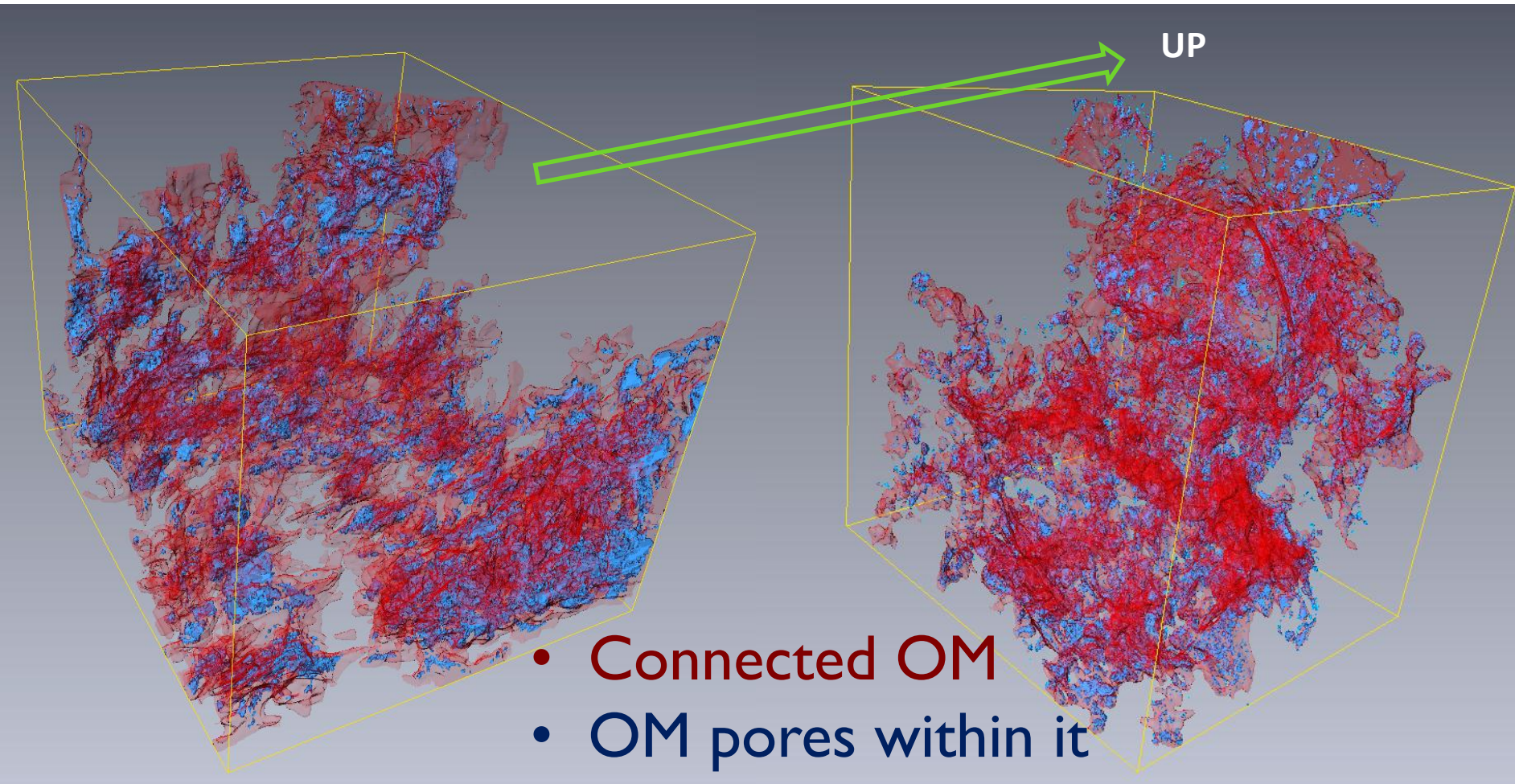
Helium Ion Microscope Image (No Coating)

Small pores (2-10 nm) in large pores

Freshly broken sample: small pore connection

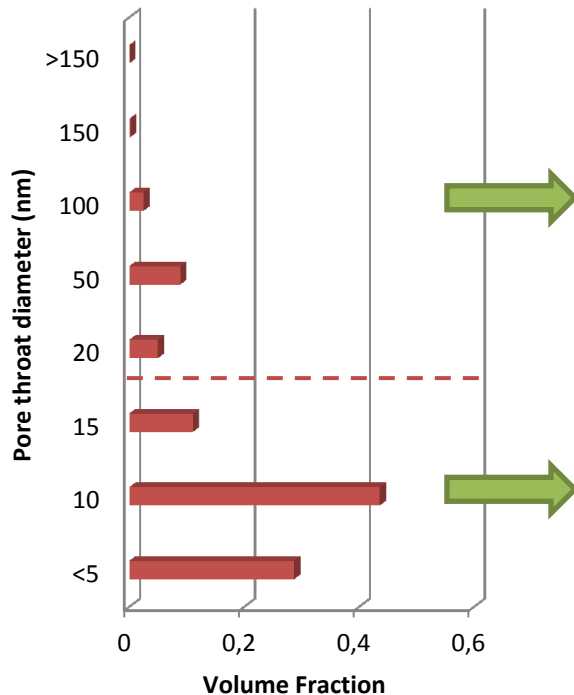| Field Of View 300.00 nm | 20.00 nm | Working Dist 7.8 mm | Blanker Current 0.0 pA |
| Mag (4x5 Polaroid) 381,000.00 X | Image Size 1024x1024 | Tilt Angle 0.0 deg | Barnett Shale |

Connected OM and OM Pore within it

UP

- Connected OM
- OM pores within it
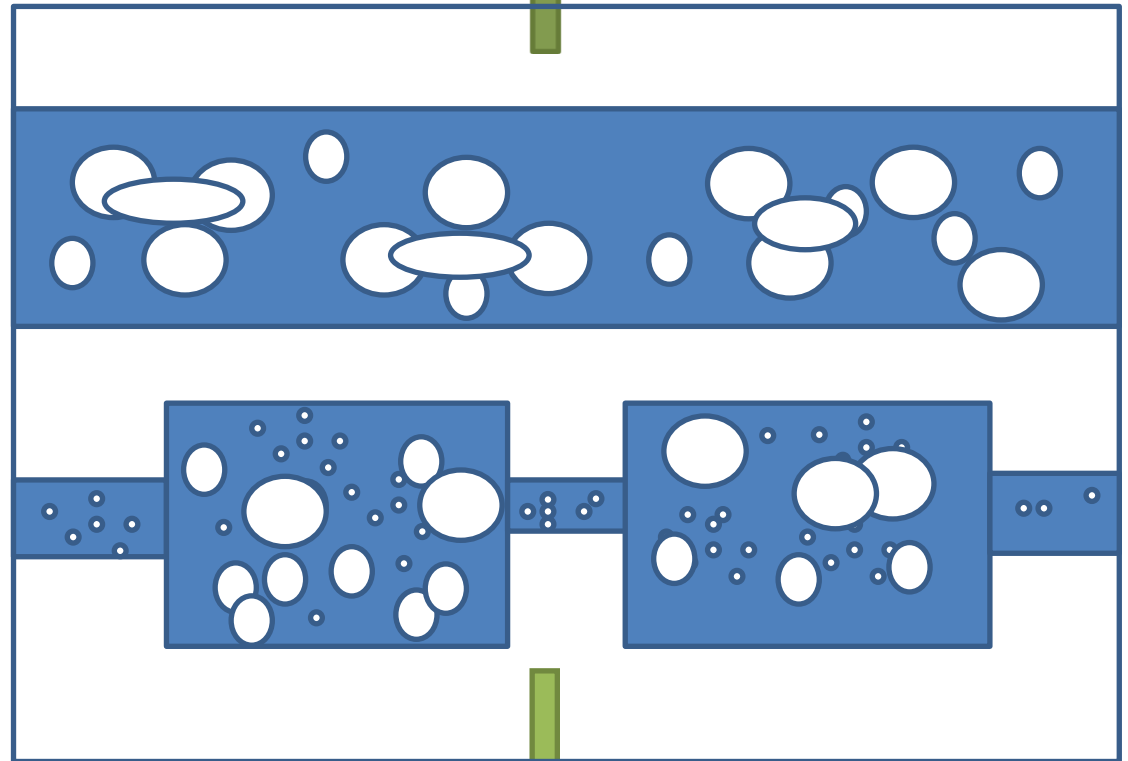
- Adding of small pores (< 10-20 nm) can lead to a percolating pore network

A Conceptual Model based on Mercury Intrusion and Image Analysis

Mercury intrusion pore throat size distribution

Large pore (> 20 nm) connected network
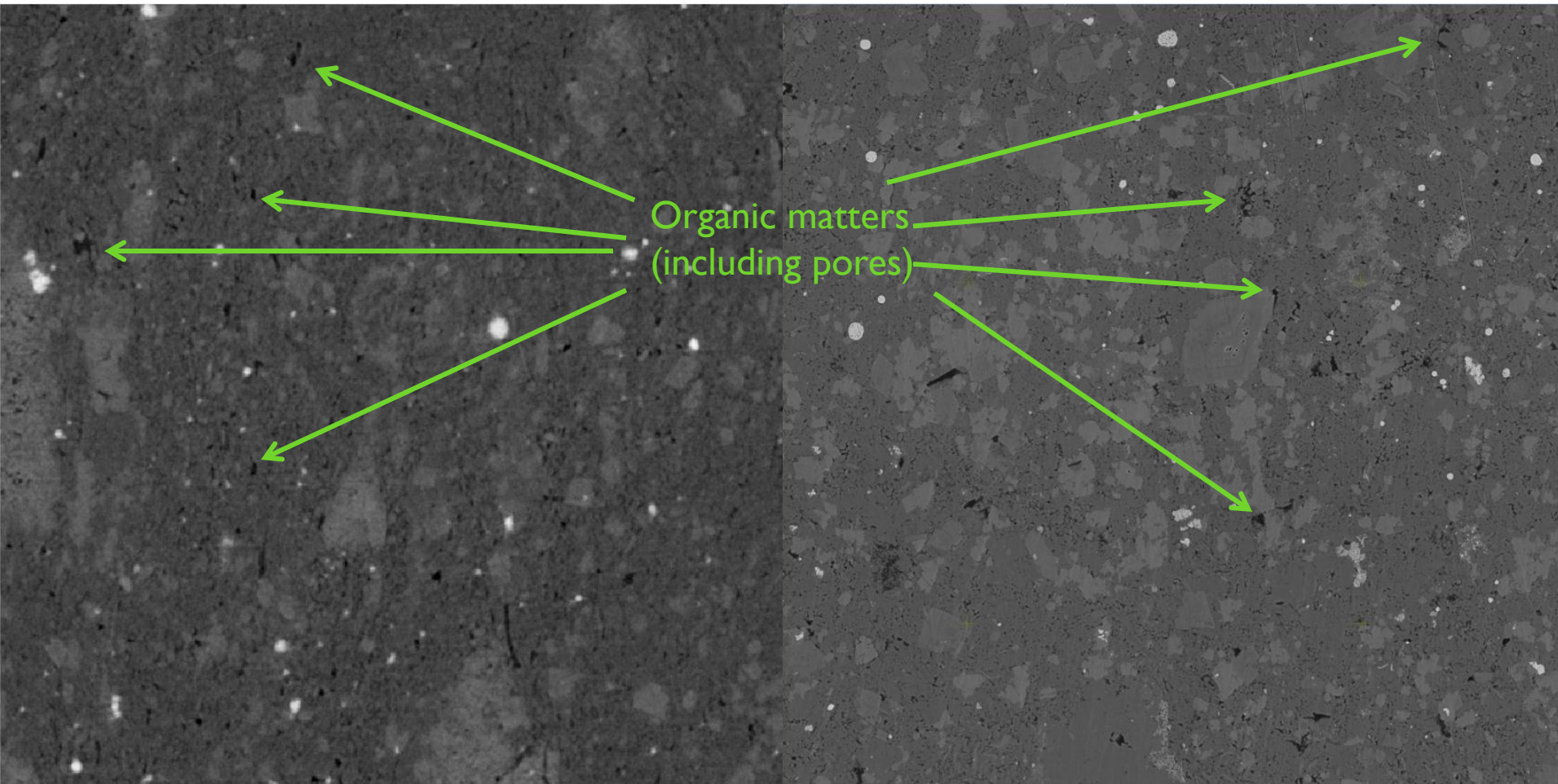
Pore throat diameter (nm): >150, 150, 100, 50, 20, 15, 10, <5

Volume Fraction: 0, 0,2, 0,4, 0,6

A bimodal distribution

Small pore connected network (including locally connected large pores)

Synchrotron μCT   vs.   SEM

Organic matters
(including pores)

0.5 mm

0.5 mm

- From μCT: large OM particles (> 0.74 μm)

3D Reconstruction with Synchrotron µCT Images
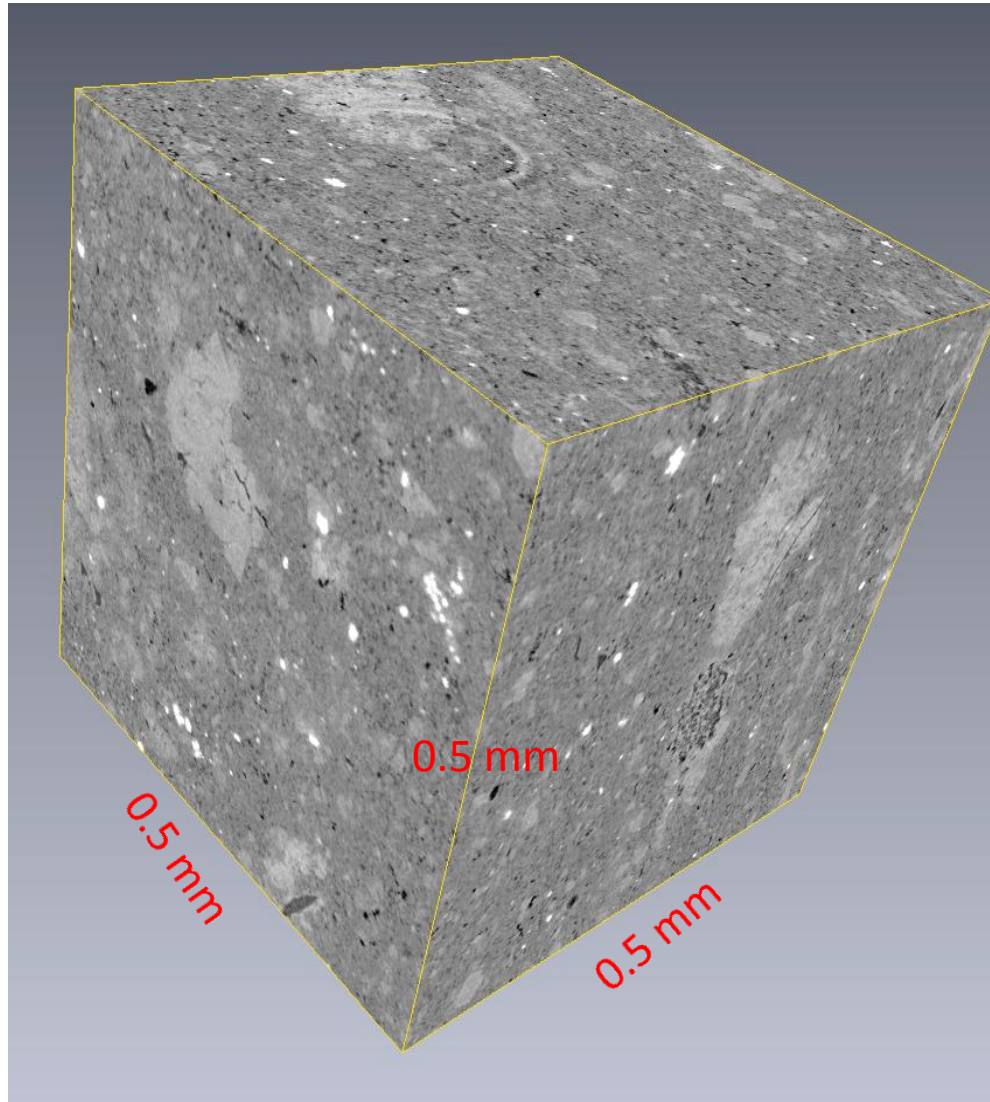
0.5 mm
0.5 mm
0.5 mm

- Pixel resolution: 0.74 µm

Organic Matter Particle (including pores) Segmentation
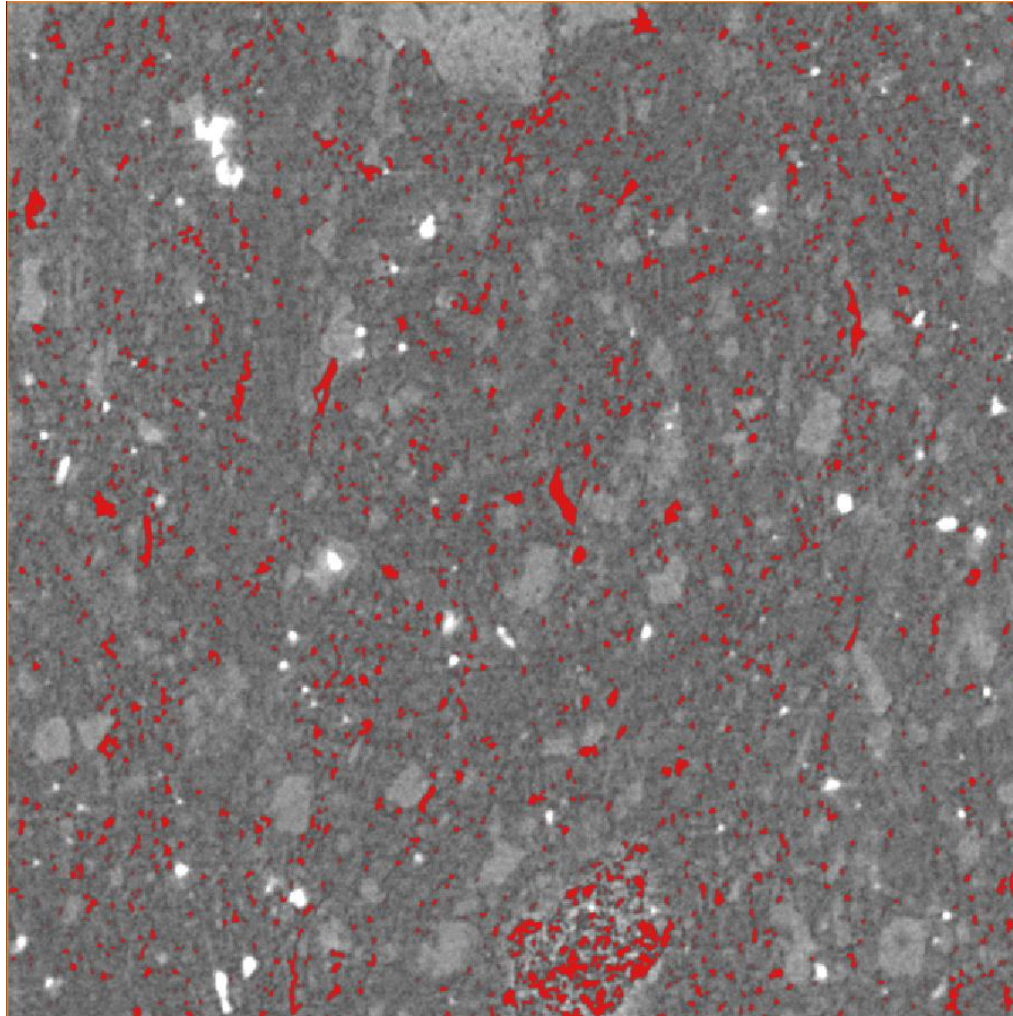
0.5 mm

OM Particle Network

$$TOC = \frac{f_{om+pore} - n}{1 - n} \frac{\rho_{om}}{\rho_r}$$
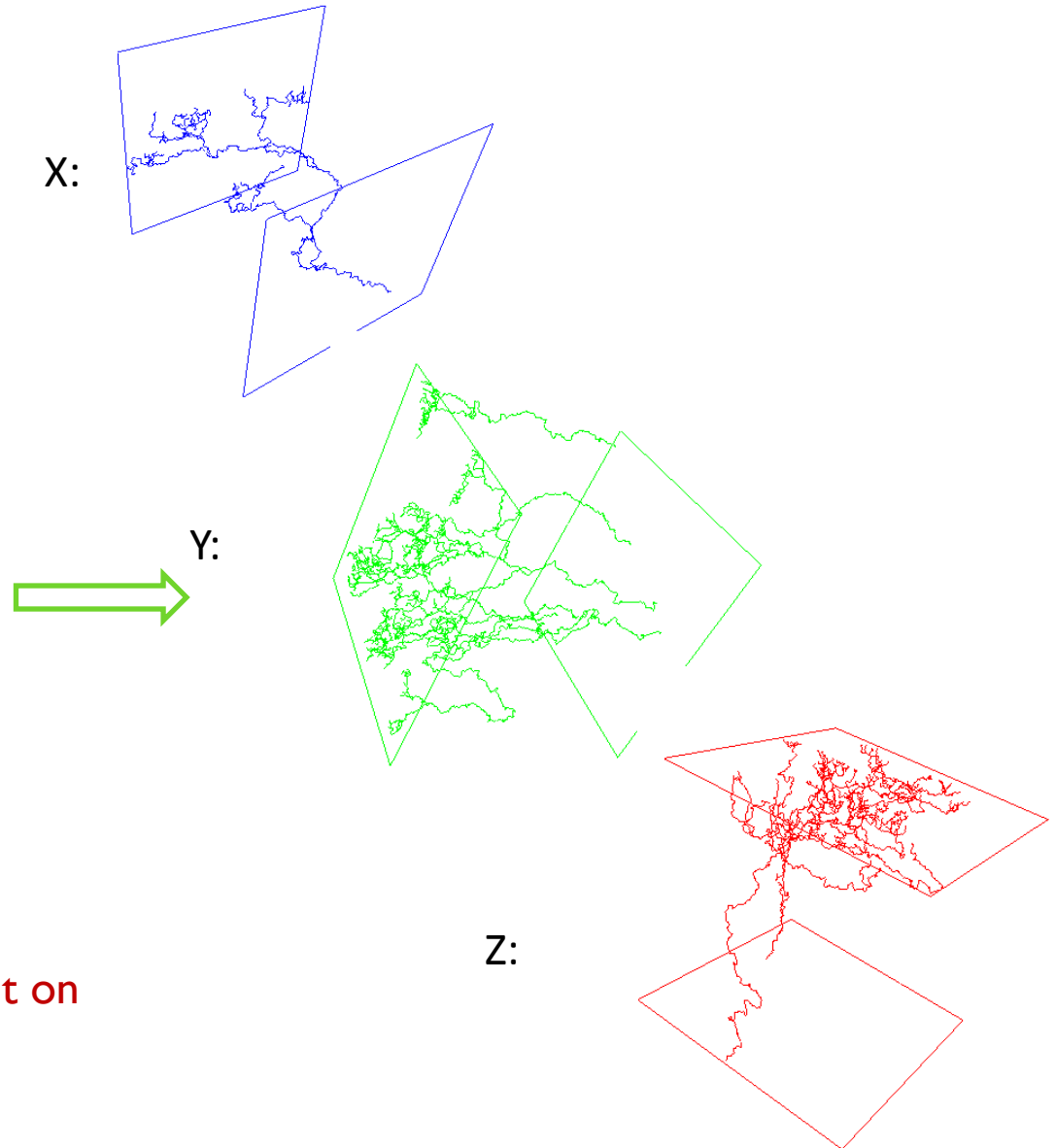
Volume fraction

Density

0.5 mm

• OM particle
• Connected OM particle
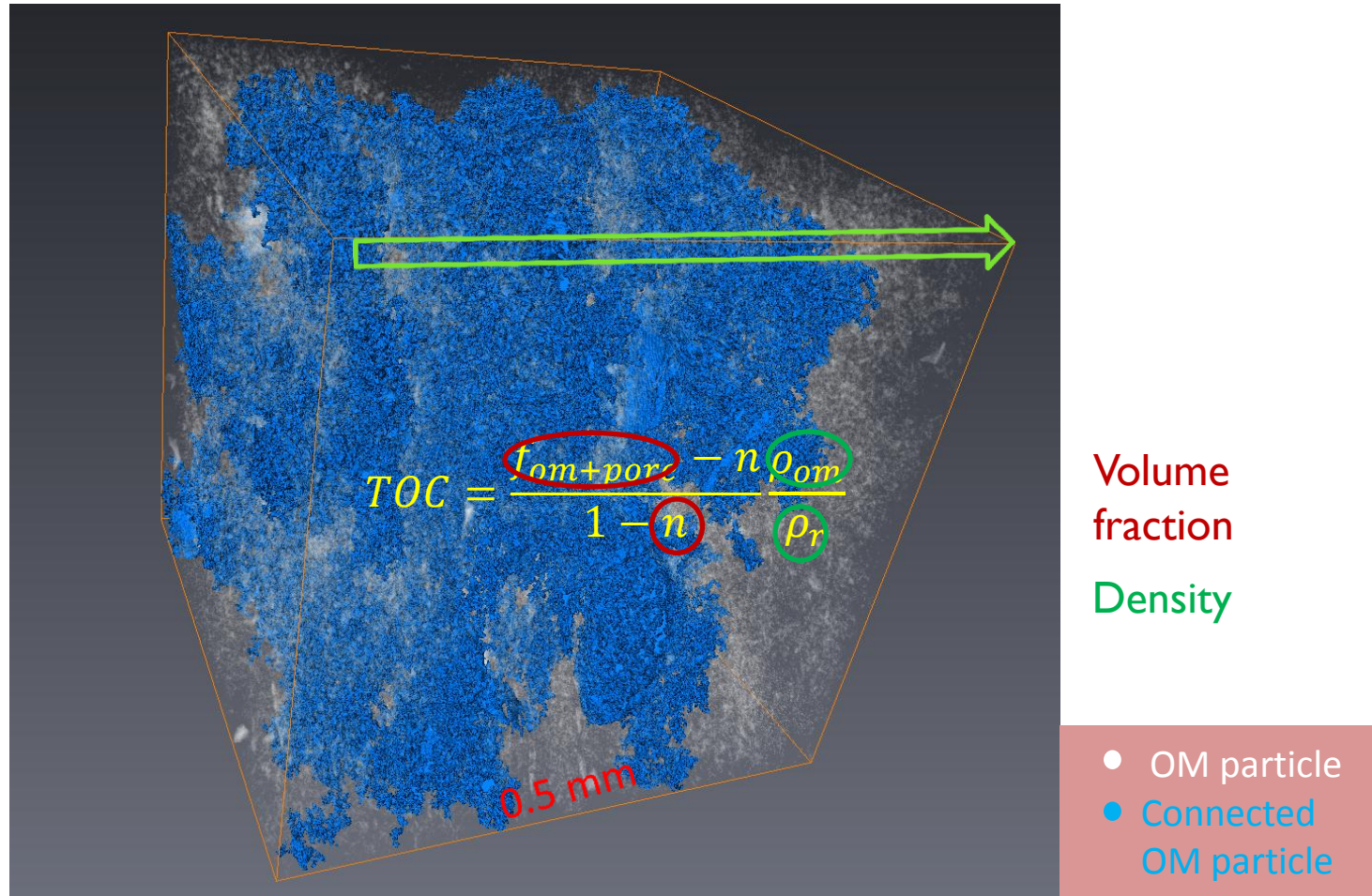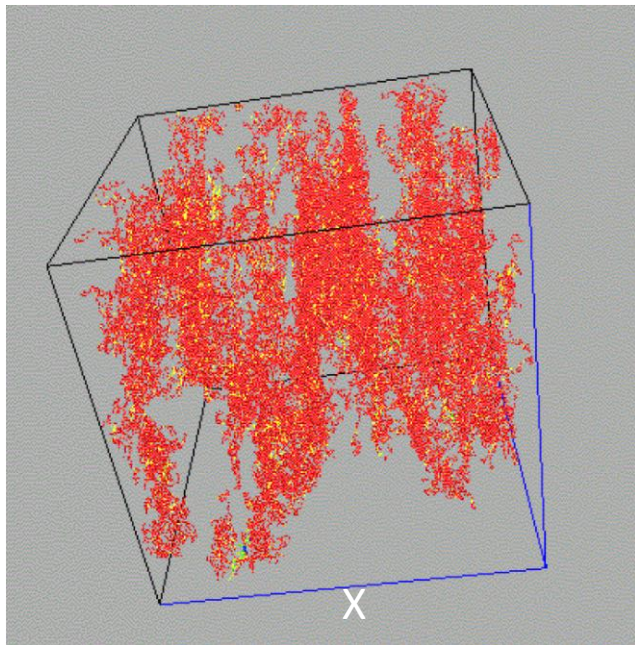
- All (OM+pore) volume fraction (OM volume/Total volume): 7.8%

- Equivalent to TOC of 3.3%

- Connected OM volume fraction: 3.7%; Connected OM/All OM: 47.3% (volume)

Shortest Path Analysis

Media axis of the connected OM
particles (the skeleton)

Layered connection: bedding effect on
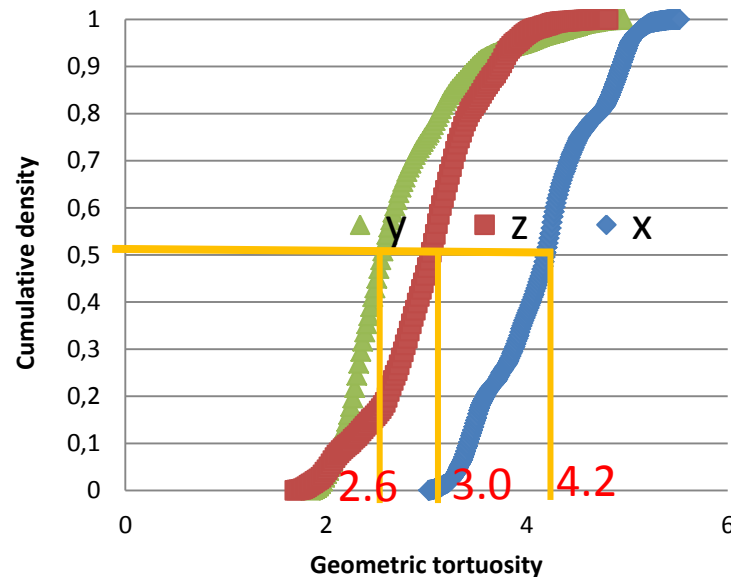organic matter connection

X:

Y:

Z:

Geometric Tortuosity

Geometric tortuosity:

$$\tau_g = \frac{L_{ij}}{d_{ij}} \qquad\qquad K \propto \frac{1}{\tau^2}$$
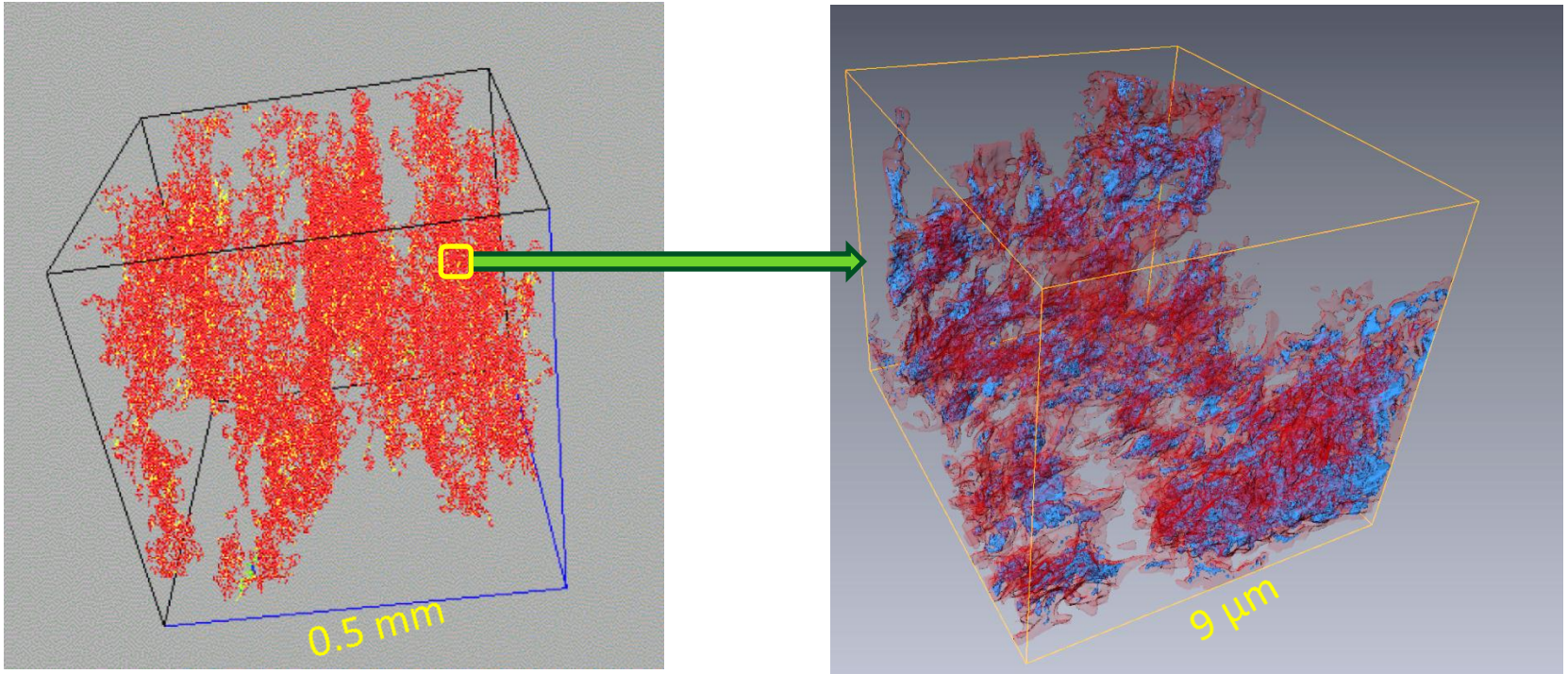
L_{ij}: shortest path between point i (entrance face) and point j (exit face)

D_{ij}: Euclidean distance (straight line distance) between i and j.

X direction (vertical to bedding) has the largest tortuosity

Integration of Two Scales

Organic matter particle network in **mm-scale** ⟷ Organic matter pore network in **μm-scale**

Permeability Analysis: µm-scale

A   B
C   D

2 µm
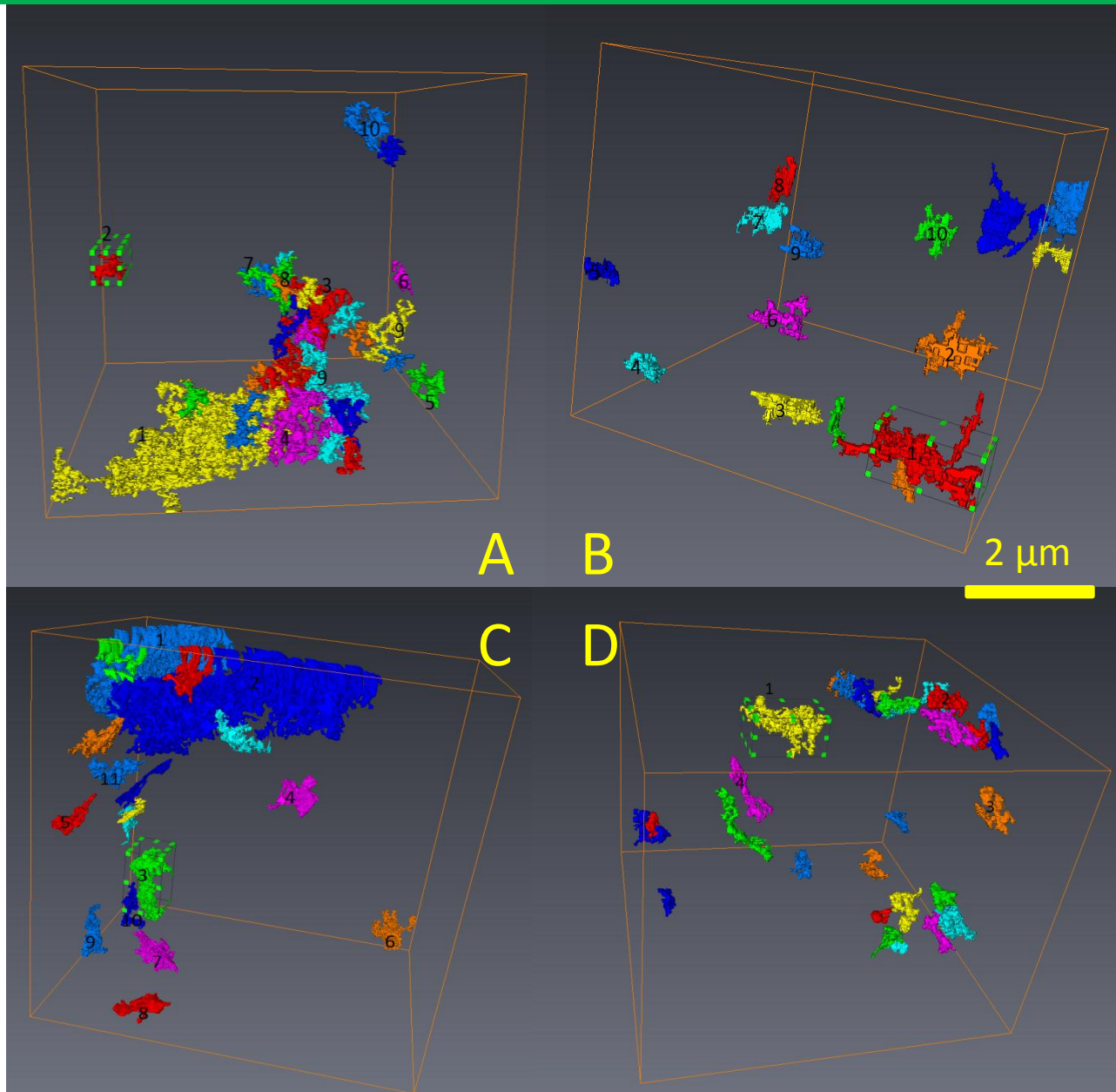
Pore
Networks in
OM from
FIB

50
permeability
and pore
size analysis

Stokes
equation:
Finite volume
method

Avizo Xlab
Hydro (VSG)
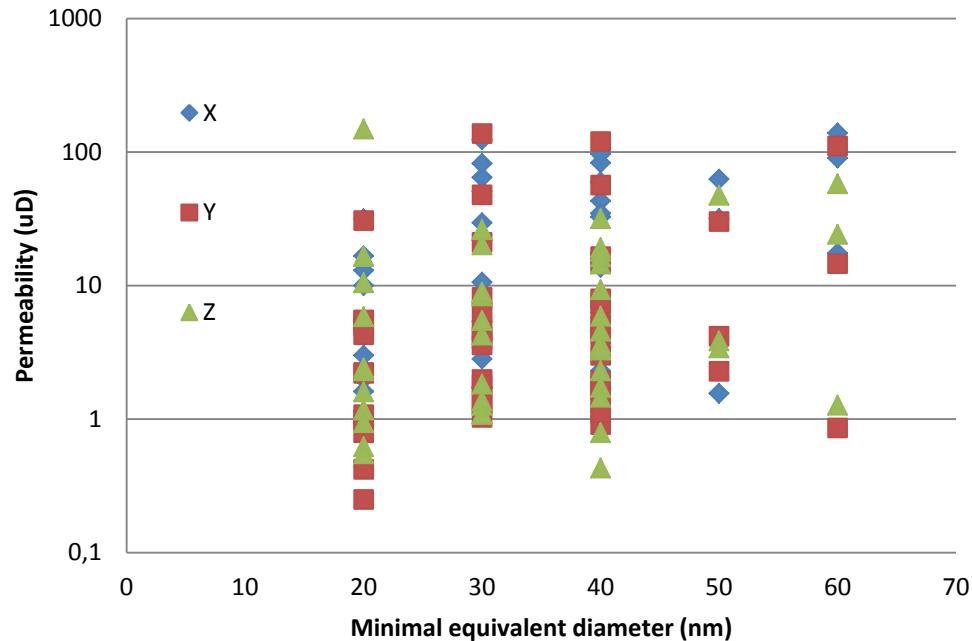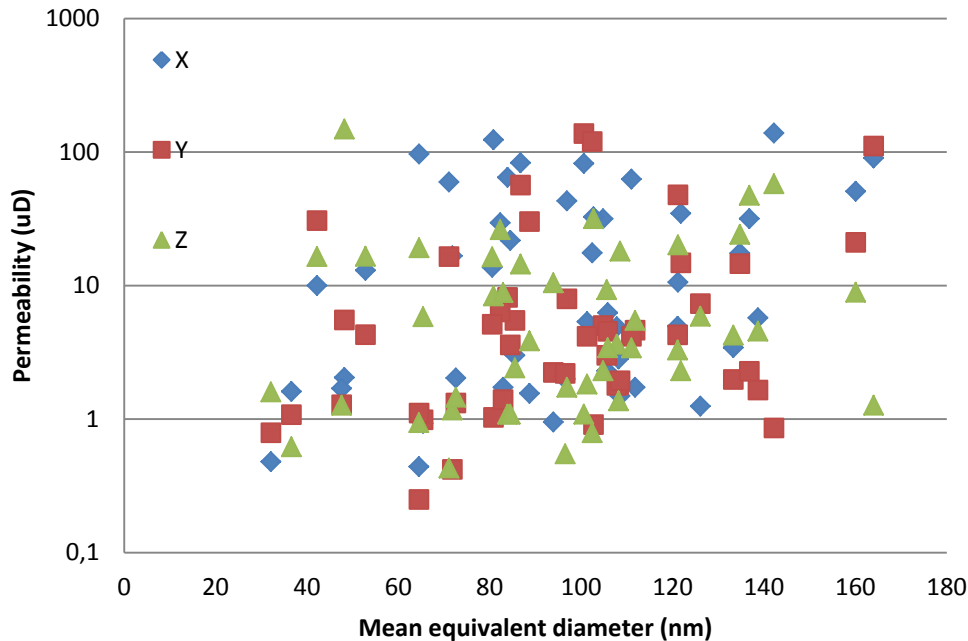
- Permeability for local connected large-pore network (μm- or sub-μm scale):

  ~1-100 μDarcy

- No clear correlation between permeability and pore diameter:
  - Many other local influencing factors

- No preferential flow direction at such scale (μm- or sub-μm scale)
  - Larger permeability in X, Y, or Z

An Empirical Equation for Up-scaled Permeability Estimation

Large pore (> 20 nm) network: K_{l_eff}

Small pore connected network (w/ locally connected large pores): K_{s_eff}
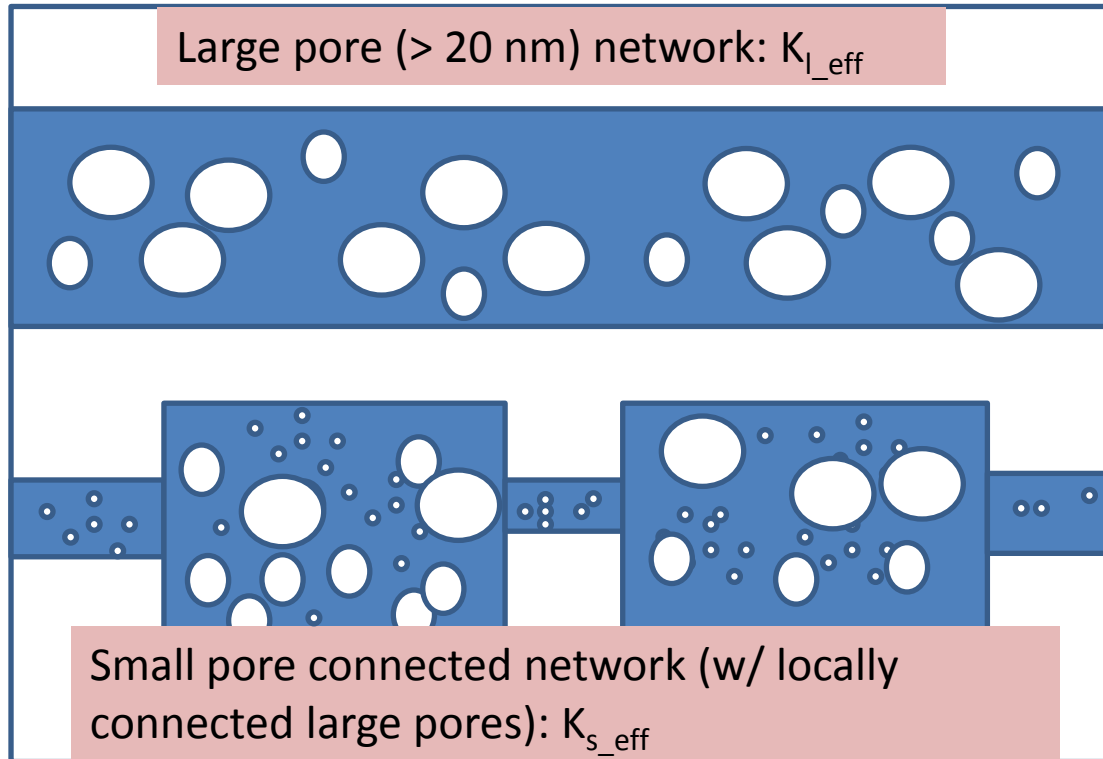
$$K_{l\_eff} = (harmatic\ mean,\ arithmatic\ mean\ of\ the\ large - pore\ permeability)$$

$$K_{s\_eff} = \frac{n_{sc}}{(\frac{n_s}{K_s} + \frac{n_l}{K_{l\_eff}})}$$

$K_s = (10\ nD,\ 100\ nD)$

Up-scaled effective permeability:

$$K_{eff} = \frac{1}{\tau_g^2} n_{omc} (f_l K_{l\_eff} + f_s K_{s\_eff})$$

Tortuosity of organic matter

Organic matter (connected) volume fraction

Large or small pore network fraction

Up-scaled Permeability Estimation Results

	Ks = 10 nD		Ks = 100 nD	
	Kl_eff = 3.9 uD	Kl_eff = 21.8 uD	Kl_eff = 3.9 uD	Kl_eff = 21.8 uD
permeability in each direction (nD)				
Parallel to bedding Y	1.4 **2 nD**	8.7	2.0 **22 nD**	9.3
Z	3.4	21.5	4.3	22.4
Perpendicular to bedding X	0.6 **0.6 nD**	2.6	1.0 **3 nD**	2.9
contribution from small connected (<10 nm) and large (>10 nm) pore networks				
small	4.9%	1.0%	32.6%	8.7%
large	95.1%	99.0%	67.4%	91.3%

Comparable to measured value of : ~35-100 nD in bedding direction
~2 nD in perpendicular to bedding

Slight underestimation may be caused by the lower TOC.

Conclusions

- Integrated method must be used to understand the full picture of the shale pore network: FIB, μCT, HIM, and MICP;

- Such an integrated method generated reasonable permeability (2-22 nD in bedding direction; 0.6-3 nD in vertical to bedding direction);

- This method provides a basis for further up-scaling to core and formation scale.

Acknowledgement

- Dan Gostovic, Robert Flores, etc. FEI Visual

- Rob Reed, BEG, UT Austin

- Patrick Smith, BEG, UT Austin

- Damon Smith, Nanoscience Center, UT Austin

OM particle analysis from SEM
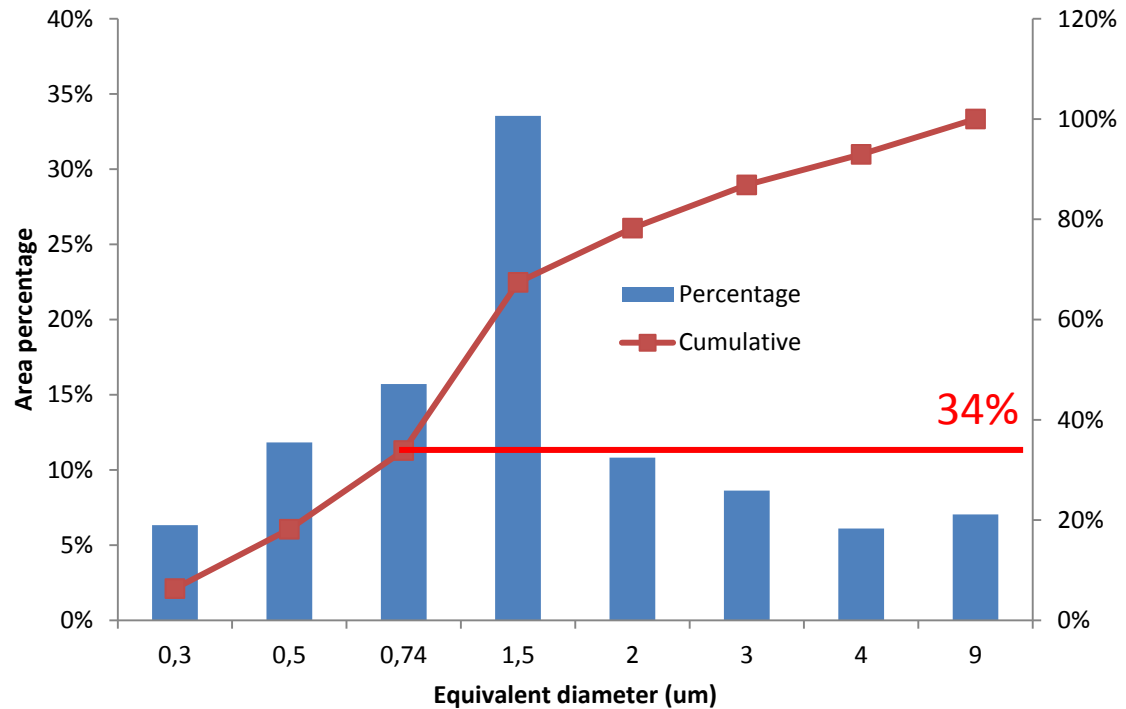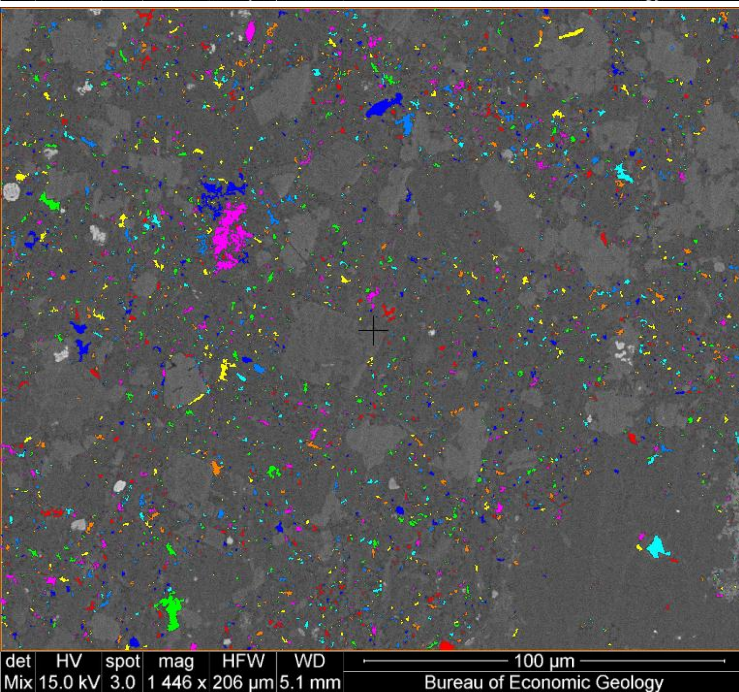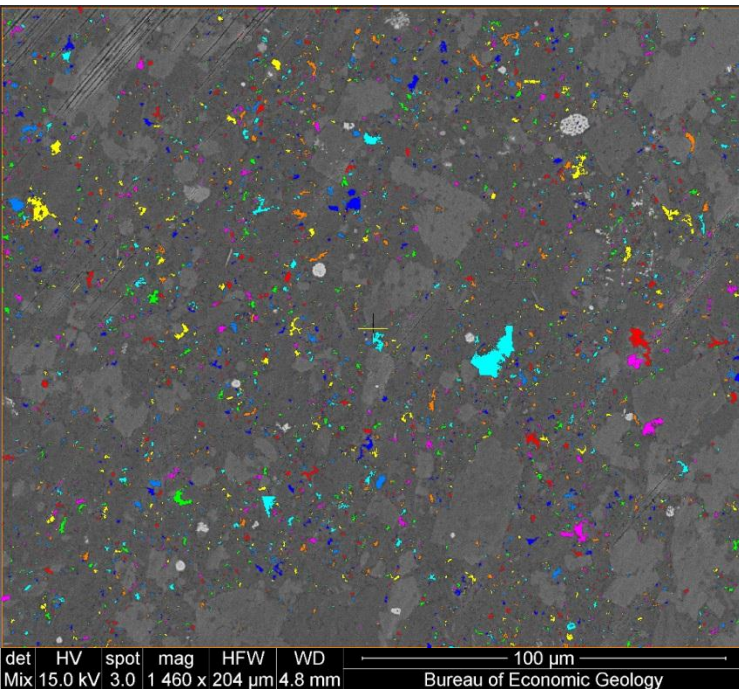
- 20 SEM images;
- OM particle size distribution from SEM images indicates ~34% OM particles < 0.74 um
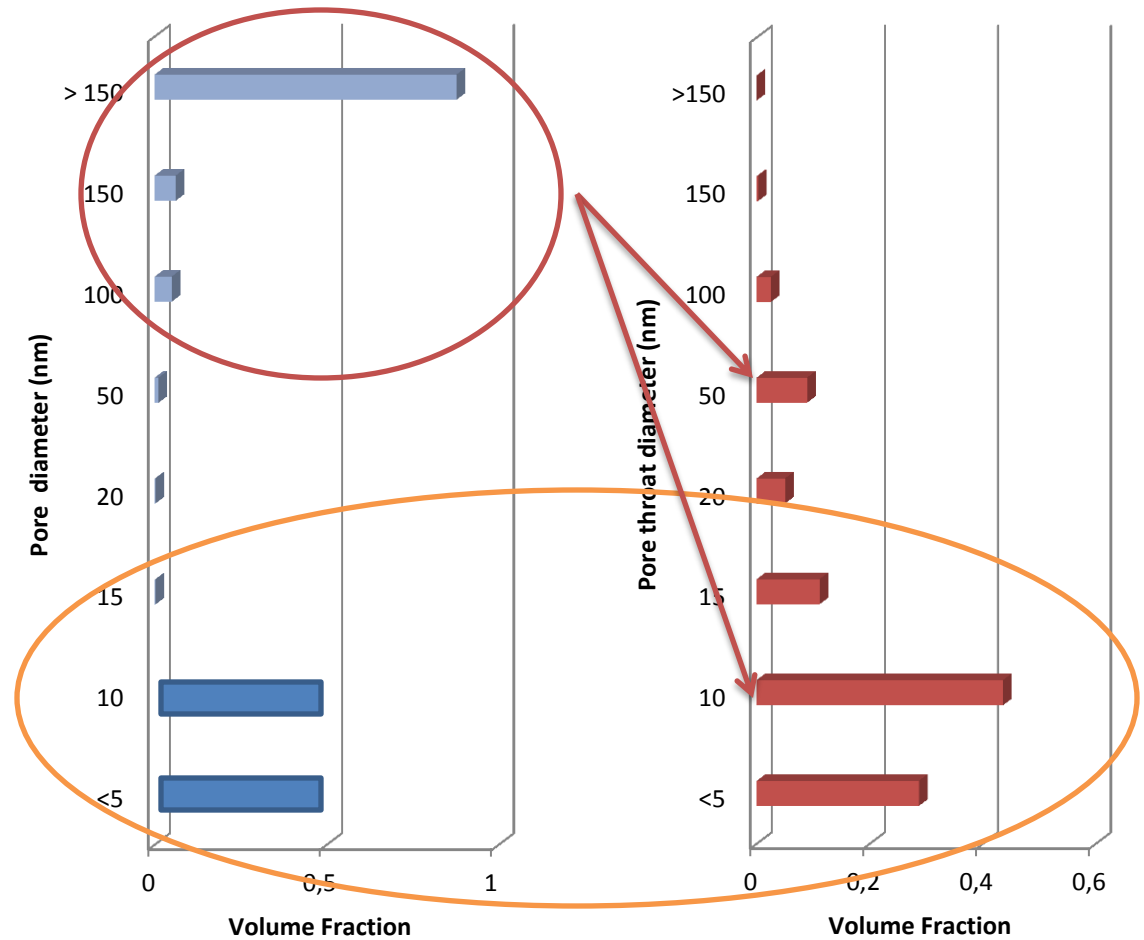
- The small (<0.74 µm) OM particles may contribute to overall OM particle connectivity;

- But may not contribute to pore connectivity, especially large pore connectivity since large pores are mostly present in large OM particles.

Large pores in large OM
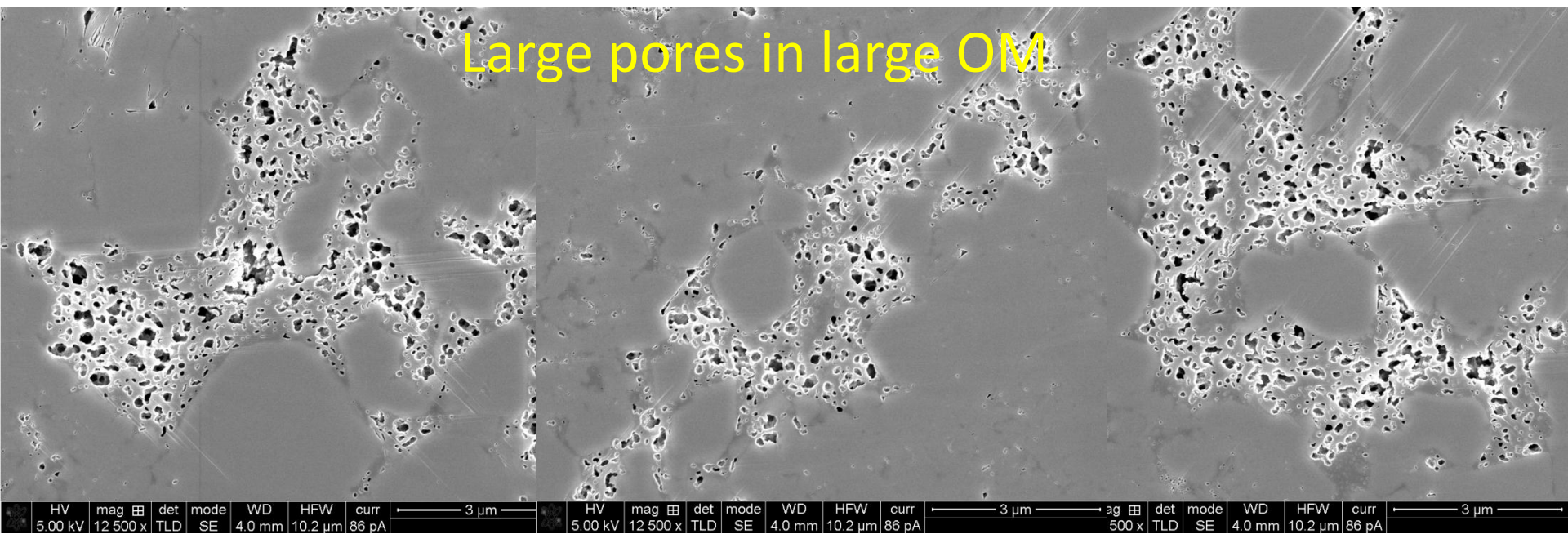
Pore size distribution: Image vs. MICP
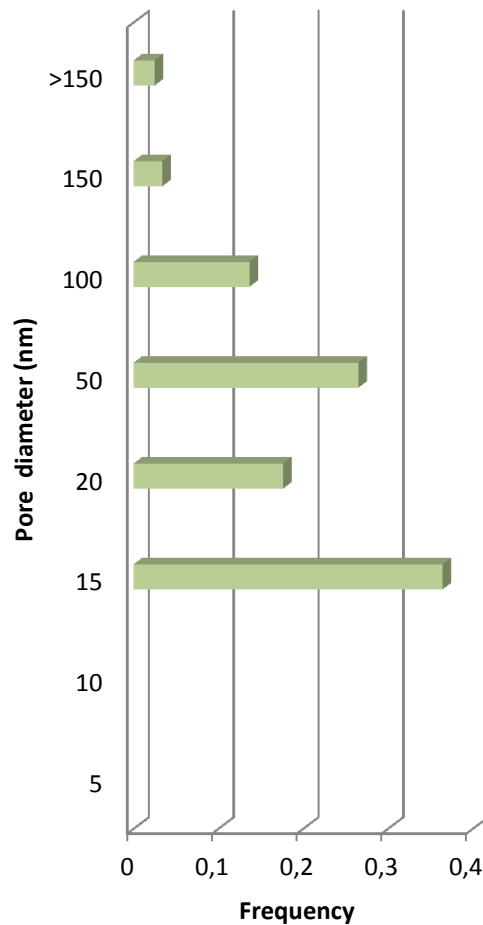
FIB/SEM Image analysis   MICP

Permeability estimation

	Ks = 10 nD		Ks = 100 nD	
	Kl_eff = 3.9 uD	Kl_eff = 21.8 uD	Kl_eff = 3.9 uD	Kl_eff = 21.8 uD
permeability in each direction (nD)				
y — Parallel to bedding — 1.4 — **2 nD**	8.7	2.0 — **22 nD**	9.3	
z — 3.4	21.5	4.3	22.4	
x — Vertical to bedding — 0.6 — **0.6 nD**	2.6	1.0 — **3 nD**	2.9	
contribution from small connected (<10 nm) and large (>10 nm) pore networks				
small	4.9%	1.0%	32.6%	8.7%
large	95.1%	99.0%	67.4%	91.3%

Factors under or overestimate permeability
- Under:
  - Larger pore permeability can be larger;
  - Larger pore connection tortuosity can be smaller;
  - Small pore connection tortuosity can be smaller;
  - Organic matter connected fraction can be larger and organic matter tortuosity can be smaller.
- Over:
  - Larger pore connection tortuosity can be larger;
  - Smaller pore connected pore network tortuosity can be larger too;
  - Ambient condition vs. in-situ condition with stress;

From Loucks et al., (2009)