How Depositional Environment, Diagenesis, and Thermal Maturity Affect the Evolution and Significance of Organic and Mineral Pore Systems in Unconventional Oil and Gas Reservoirs: Current Understanding and Future Research*

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Abstract

Mudrocks act as sources and seals for conventional petroleum systems and are sources and reservoirs for unconventional petroleum resources. Complex lateral facies heterogeneity and stacking patterns are typical of mudrock systems. A spectrum of transport and depositional processes impacts original composition, grain assemblages, texture (grain size, shape, and sorting), and fabric (arrangement of particles). Diagenesis (compaction, dissolution, cementation, and replacement) and thermal decomposition of kerogen resulting in generation, expulsion, and migration of petroleum add complexity to the mudrock system by altering porosity, permeability, wettability, and rock strength. Over the past decade, our understanding of pore systems in unconventional reservoirs and the interplay of depositional, diagenetic, and petroleum generation processes in pore system evolution has been significantly improved. However, much is still unknown as we are unable even to predict pore systems in a given lithofacies and/or depositional setting. Establishing linkages between pore systems, permeability, and multiphase fluid flow is an active research effort. Imaging techniques provide a direct means to quantify the proportion and distribution of pore types and the controls on pore development and size distribution. On the other hand, bulk measurement techniques such as gas adsorption, nuclear magnetic resonance (NMR) spectroscopy, small-angle neutron scattering (SANS), and mercury injection for capillary pressure (MICP) measurement rely on assumptions and models to infer pore dimension and connectivity.

Our work utilizes both imaging and measuring techniques and aims to (1) provide a process-based understanding of the origin and evolution of different pore types and to (2) establish linkages between depositional processes, mineralogy, and variations in pore systems. Examination of Pliocene-Pleistocene eastern Mediterranean sapropels using field-emission SEM imaging allowed inspection of petrographic textures before alteration related to thermal maturation. Results indicate that sediments rich in marine kerogen are subject to substantial compactional porosity loss during early burial. The dominant organic matter (OM) behaved in a highly ductile manner, pervading into some of the interparticle

mineral pore spaces, whereas other mineral pores were not pervaded by ductile OM. These primary interparticle and intraparticle mineral pores determine the mineral pore network before petroleum generation and constrain petroleum migration and redistribution during thermal maturation of OM. Therefore, depositional and early diagenetic processes control the subsequent mineral-pore and OM-pore network. After OM maturation, pore evolution is closely tied to the thermal cracking and kinetics of different OM types.

Laboratory gold-tube pyrolysis experiments on the Devonian Woodford and Mississippian Barnett siliceous mudstones show that algal cysts (Tasmanites) have better generation and expulsion potential than does amorphous organic matter (AOM). After expulsion, larger pores were found in algal cysts than in AOM. Generated petroleum is chemically fractionated with the expelled products enriched in hydrocarbons and gases and retained bitumen enriched in heteroatomic polar compounds and asphaltenes. Fractionation processes in the mudrock lead to different morphologies of observed pores at each generation stage. With increasing thermal maturation, predominant pore types changed from primary mineral pores, to mineral pores containing relic OM, to coexisting mineral pores and various OM pore types, and finally to OM spongy pores. Residual kerogen and retained solid bitumen both host OM pores in the oil window. In the dry gas window, a solid bitumen/pyrobitumen network, which hosts abundant OM pores, becomes important. We have observed these differences in both gold-tube pyrolysis residues and by comparing OM pores in naturally matured samples of oil-window Eagle Ford Formation and dry-gas window Marcellus Shale.

Mineralogy is commonly used to assess rock strength and response to hydraulic fracturing; however, variations of texture and fabric within each lithofacies could be as important as bulk composition. Our work on the Triassic lacustrine Yanchang Formation of the Ordos Basin in China found that mineral pores were more abundant in well-sorted and coarser grained mudrocks than those in poorly-sorted and finer grained mudrocks. Lithofacies affects relative abundance of mineral pores versus OM pores in the oil-window. Lithofacies and TOC both affect permeability in dry gas-window mudrocks. Primary mineralogy, texture, and fabric determine the following diagenetic processes such as compaction and cementation, and our work suggests that these factors could indirectly affect OM pore sizes, resulting in differences of up to at least two orders of magnitude.

There remains a lack of clear understanding on the large-scale depositional and diagenetic control of pore systems. This is in part as a result of limited and biased sampling in many studies. For example, the OM-lean facies in unconventional reservoirs are always less studied than the oil or gas-producing OM- rich facies. Also, representative elemental area (REA) of SEM images that can be used to construct 2D or 3D simulations has not been fully established. Cleary, more research is needed to upscale the nm- to µm- size pore systems to responses in wireline logs and basin-scale stratigraphic variation. Finally, phase diagrams and fluid dynamics are found to be pore-size dependence. A multidisciplinary approach integrating geology, geochemistry, petrophysics, and fluid flow is desperately needed to eventually lead to predictability.

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Session 1: Source Rock Analysis
AAPG Hedberg Conference, Houston, Texas, March 4th, 2019

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History of Finding or Measuring OM Pores

- The nature of gas storage and transport in shales has been a topic of discussion since 1930s and has been debated for decades (Walters, 2018).
- Emmett (1948) "Adsorption and Pore-size Measurements on Charcoals and Whetlerites" – B.E.T equation to low-temperature adsorption isotherms of N_2.
- Zwietering and Van Krevelen (1954) "Chemical structure and properties of coal IV – Pore structure"
- Gan et al. (1972) "Nature of the porosity in American coals"
  - Gas (N_2 and CO_2) adsorption
  - He and Hg displacement and Hg porosimetry
  - Total porosity ranges from 4.1% to 23.2%, pore diameters from 1.2 nm to 2.96 µm
  - Low-rank coals: macropores (>30 nm) dominate
- Harris and Yust (1976) used TEM to observe mesopores in exinite and intertinite in a high-volatile bituminous coal
  - Finest pores observed in vitrinite ranged in size from < 2 nm to 20 nm in diameter, with the majority in the smaller end of the size range.
  - Inertinite appears to be the most porous maceral and typically contains a broad range of pores from 5 through 30 nm.
  - The least porous maceral is exinite: featureless material except for the presence of irregular and tubular pores
- Thomas and Damberger (1976) "Internal surface area, moisture content, and porosity of Illinois Coals: Variations with Coal Rank"

History of Evolving Concepts of OM Pores

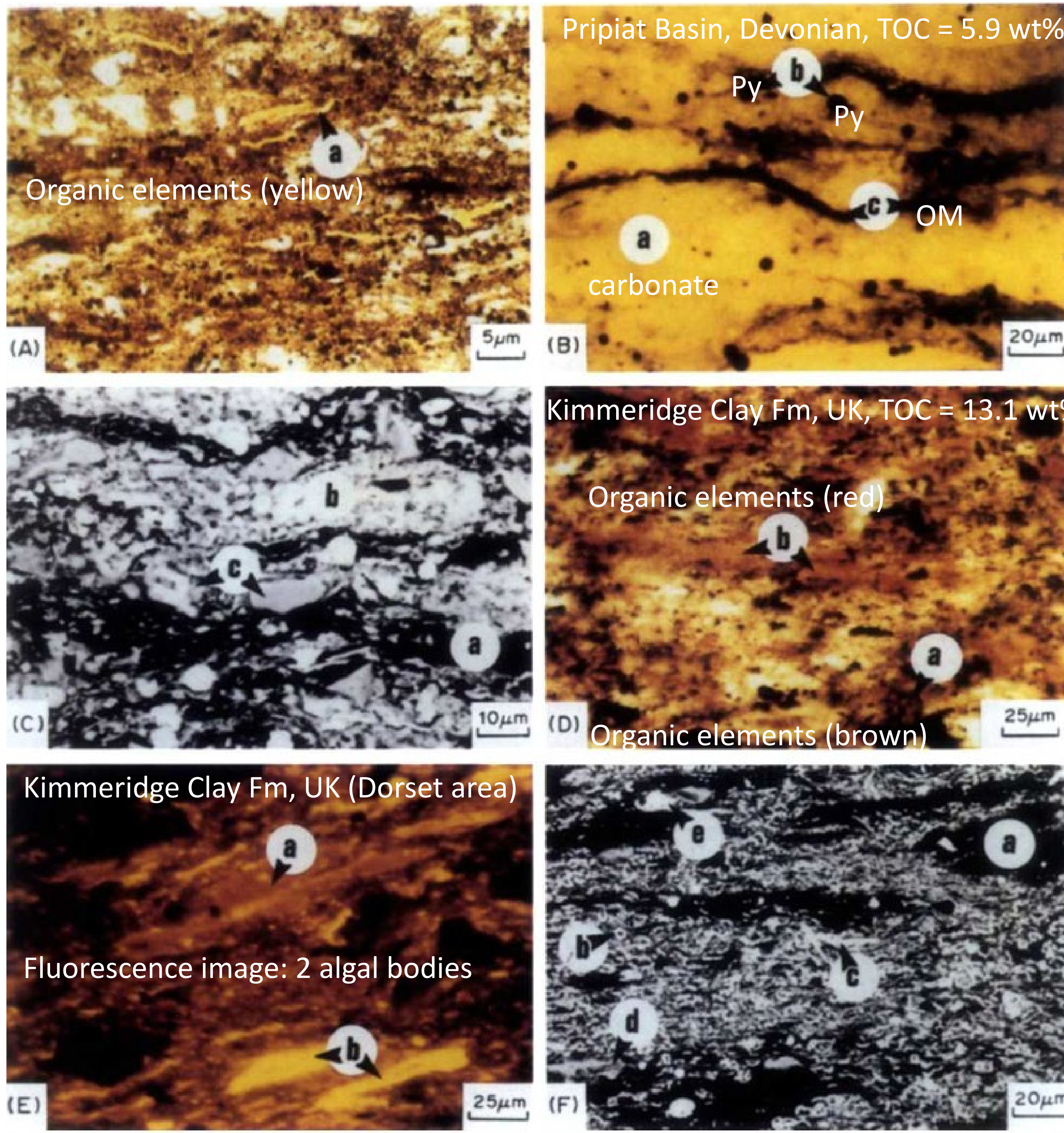
- IUPAC classification of pore size

Pore sizes	Coal rank (ASTM Designation D388-98a)
Micropores *d*<2 nm	high volatile bituminous coal A and higher
Mesopores 2 nm<*d*<50 nm	high volatile bituminous coal (C+B)
Macropores *d*>50 nm	lignites+sub-bituminous

- Between 1976 and 1981, U.S. government cut and retrieved nearly 17,000 ft of Devonian shale drill core under the Eastern Gas Shale Project (EGSP) -> petrophysical measurements were developed. Productivity is related to a variety of geological factors such as TOC, maturity, spacing of natural fracture, & stratigraphy (Soeder, 1988).
- Concept of shale gas storage continues to evolve: gas adsorption into the OM is similar to that of coals. Required porosity not found in the mineral matrix (Charpentier et al., 1993).
- Clarkson and Bustin (1996): Coal composition is important in determining the micropore capacity and size distribution.
- New techniques (ion-milled) and imaging (FE-SEM) emerged (Reed and Loucks, 2007; Loucks et al, 2009):1st time OM pores were finally observed in the Barnett Shale.

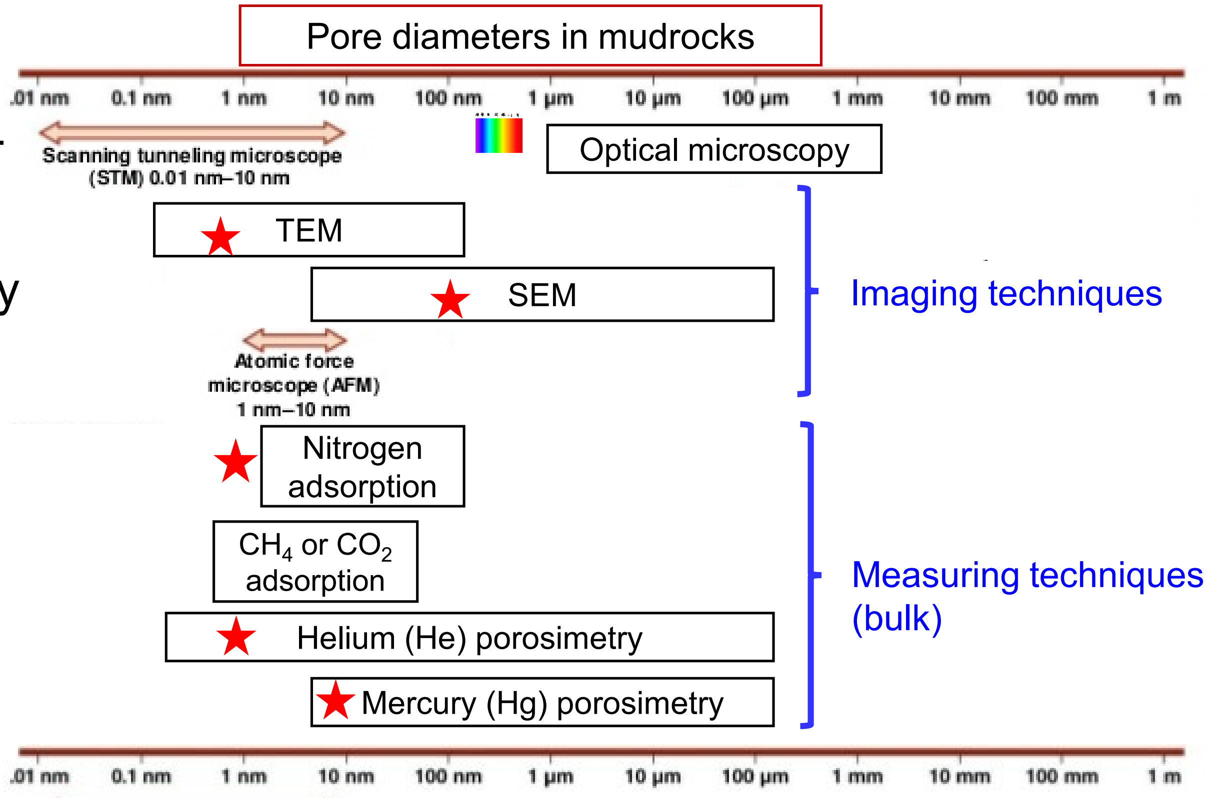
Importance of Petrography

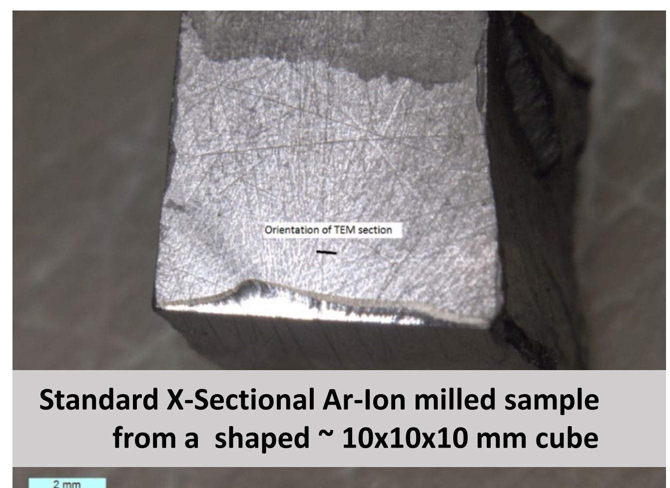
- Lewan (1987): Petrographic study of primary migration in Woodford
- Belin (1992): Applied BSE to study source rock microtexture



Common Techniques Applied Today

- Optical microscopy
- X-ray micro- and nano-computed tomography (μ-CT and nano-CT)
- HPMI (high-pressure mercury intrusion)/MICP
- NMR (nuclear magnetic resonance)
- FE-SEM
- FIB-SEM
- HIM (helium-ion microscopy)
- Gas adsorption
- He porosimetry
- AFM (atomic force microscopy)
- TEM
- Small-angle/ultrasmall-angle neutron scattering techniques (SANS/USANS)



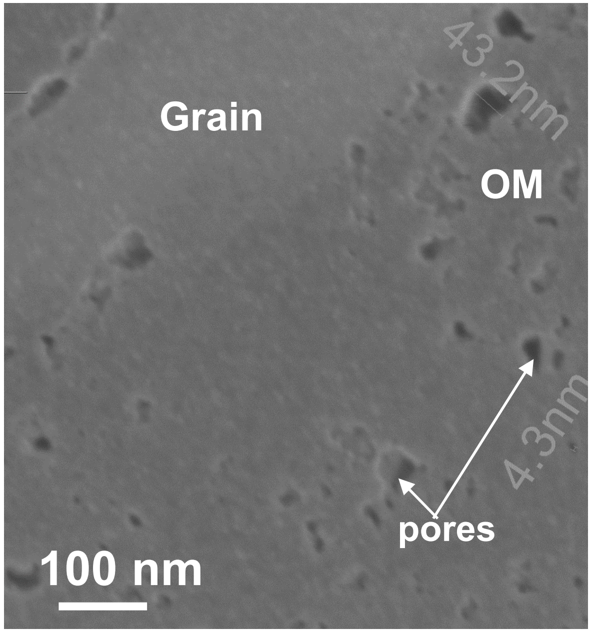


Standard X-Sectional Ar-Ion milled sample from a shaped ~ 10x10x10 mm cube

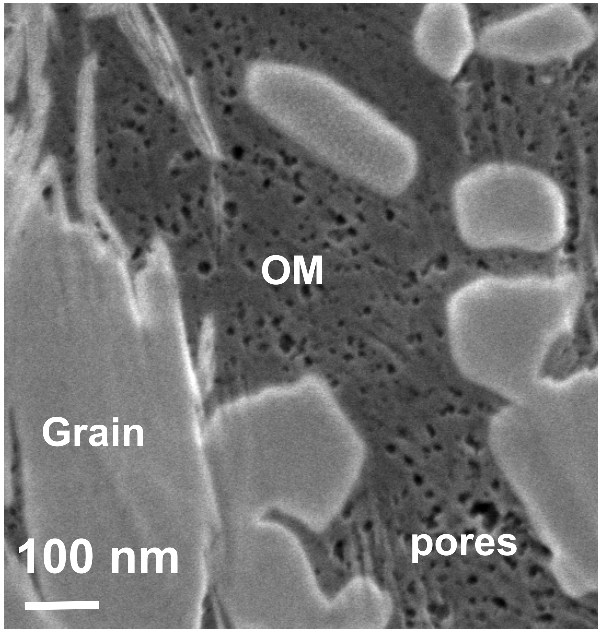
(Bustin et al., 2008; Clarkson et al., 2012; Reynolds et al., 2018)

Images Illustrate the Signal Capture from Different Microscopic Techniques

10 KeV
SEM secondary electron signal (~5 nm Ir coating)
~5 nm resolution

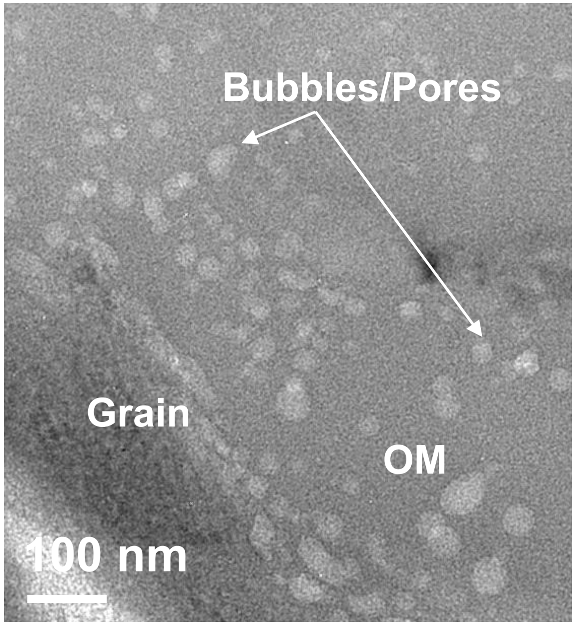


600 KeV
SEM secondary electron signal (uncoated)
~2 nm resolution



300 Kev
TEM bright field image

~ 0.1 nm resolution

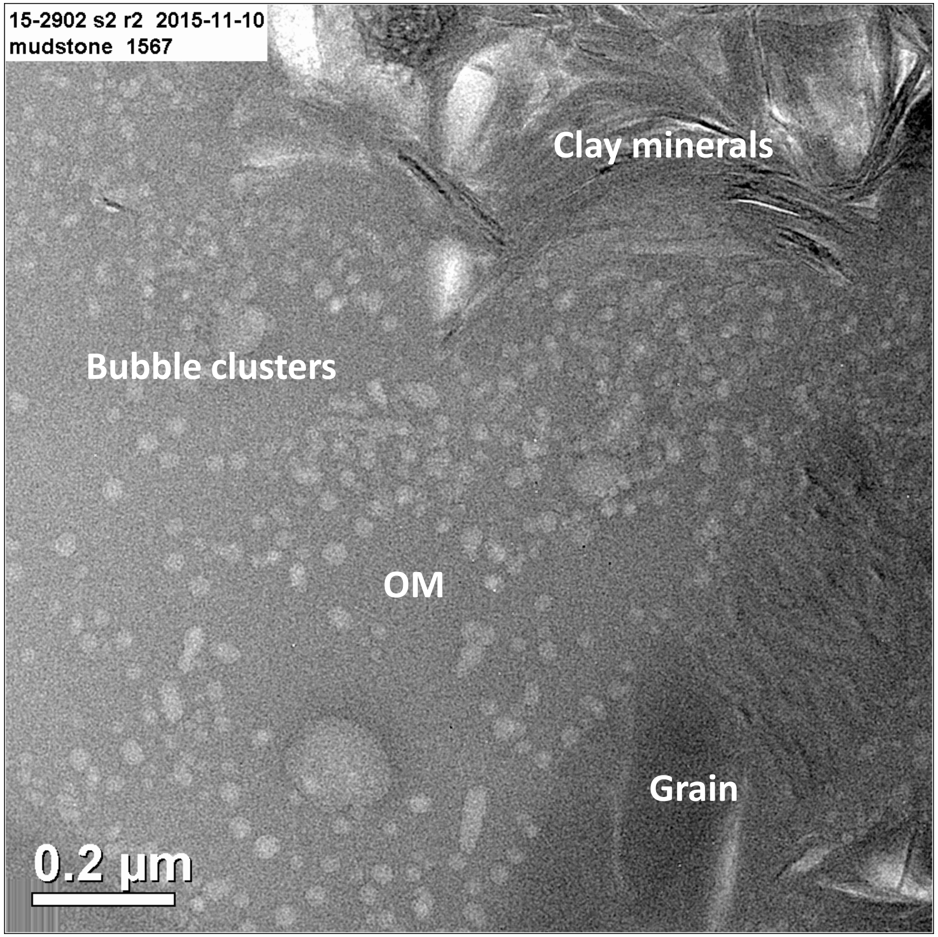


(Photo credit: Patrick Smith)

TEM vs. LV HRSEM Image of OM and Pores

Yanchang Fm, Ordos Basin, China: 1567 m

300 Kev TEM micrograph of FIB-prepared sample



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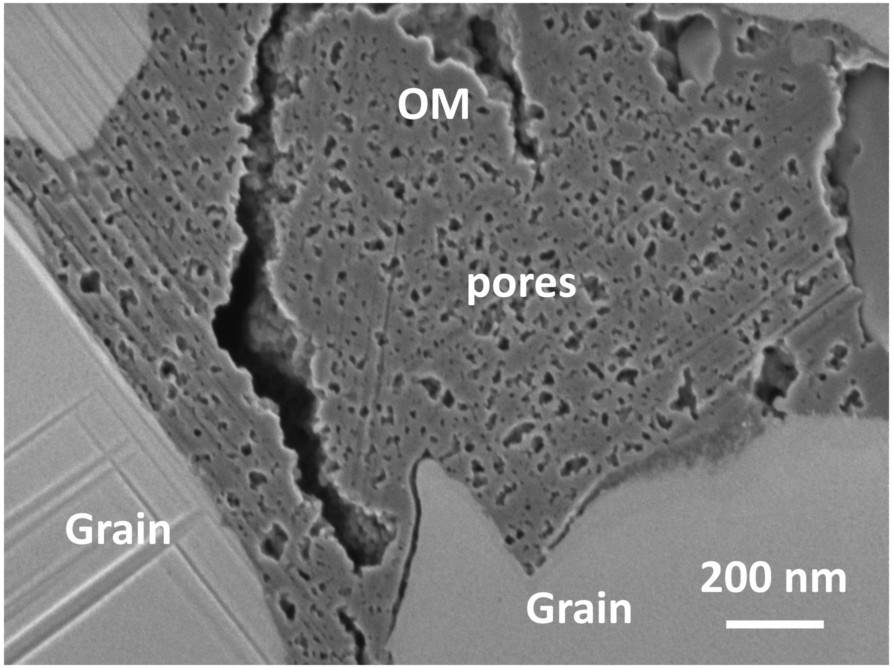
Low Voltage 700V HRSEM image of uncoated sample



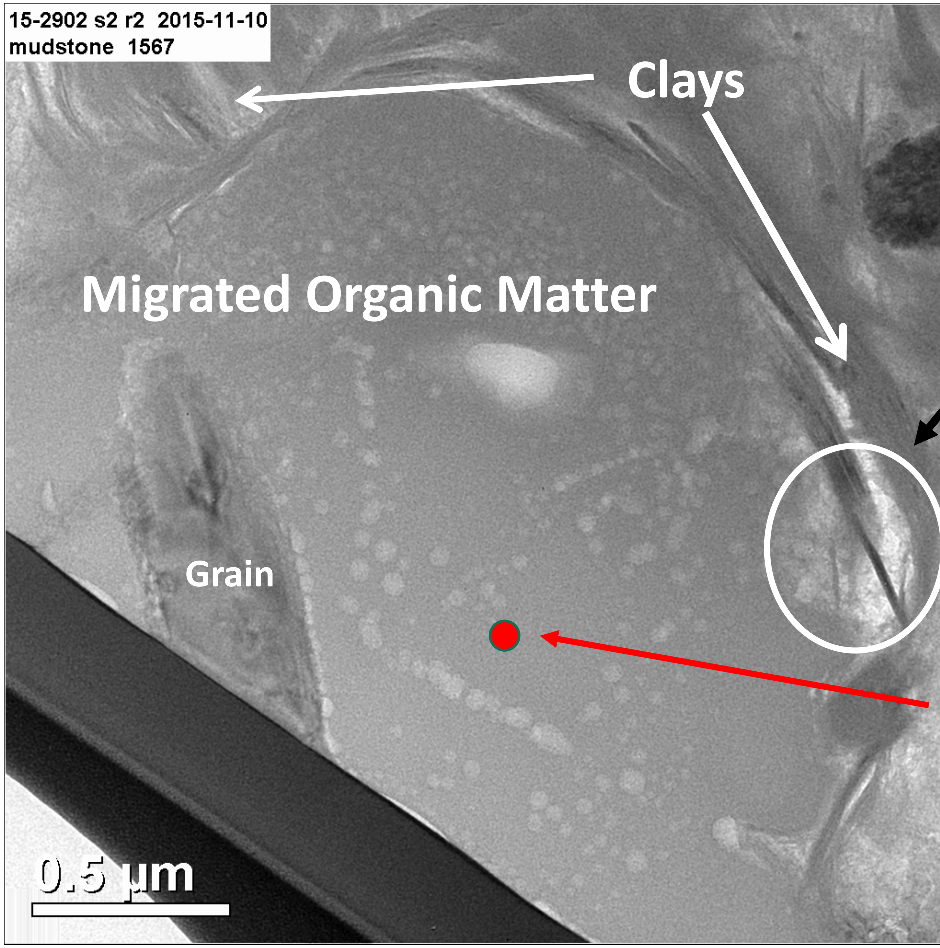
Zeiss Inc.

40.4% Clays
10.9% Calcite
23% Quartz

(Photo credit: Patrick Smith)

TEM Image of Mineral, OM, and Pores

Yanchang Fm, Ordos Basin, China: 1567 m



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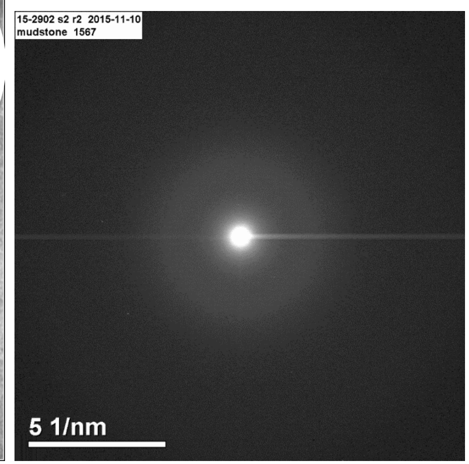
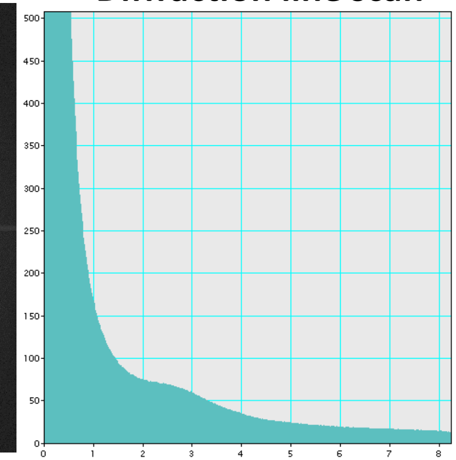
Note:

Bubble strings and clustering at OM mineral interfaces. Interparticle Clay Voiding.

Carbon amorphous Electron Diffraction pattern



Diffraction line scan



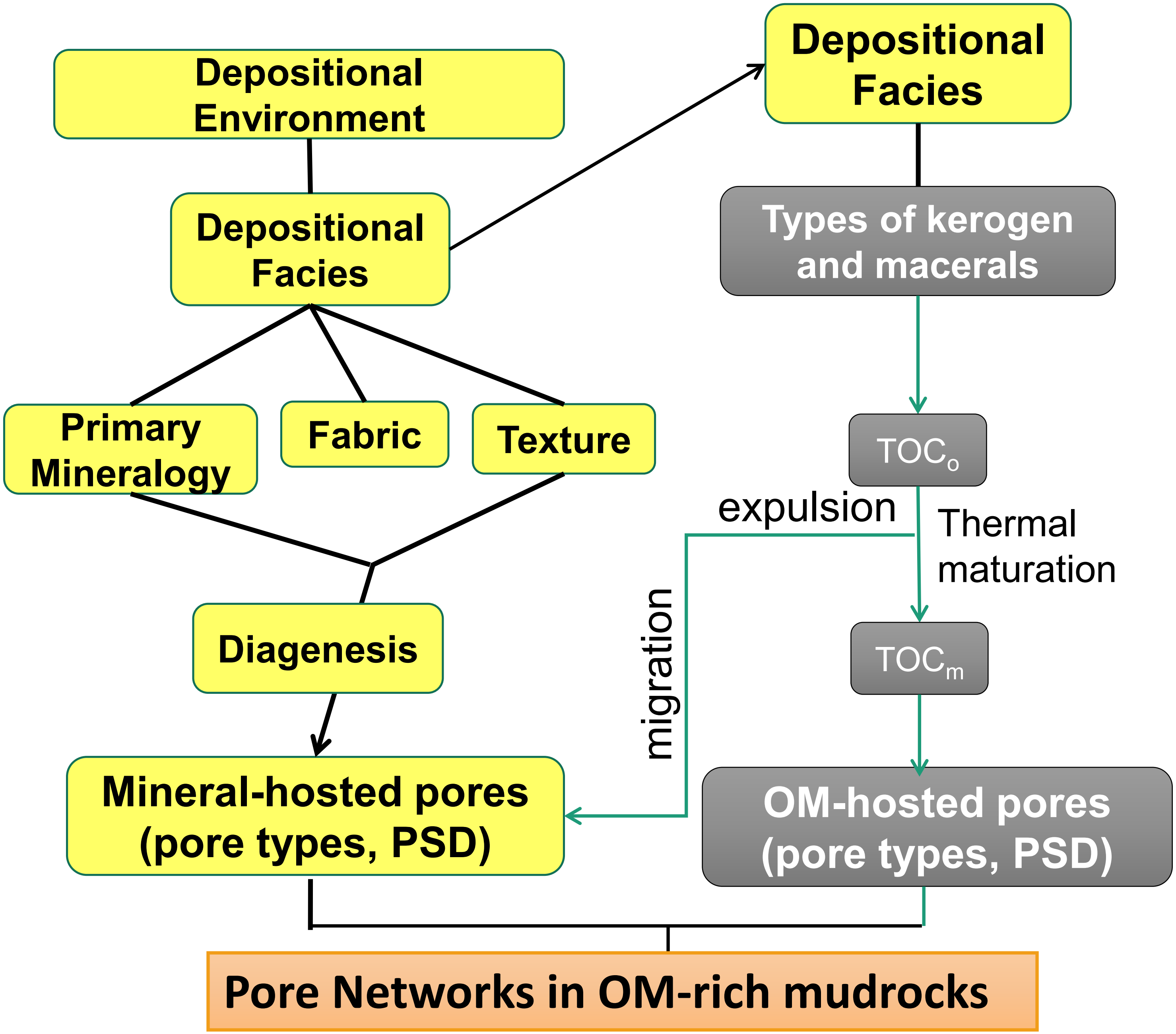
(Photo credit: Patrick Smith)

Pore Systems in Mudrocks

1. Review past findings and present ongoing research effort.

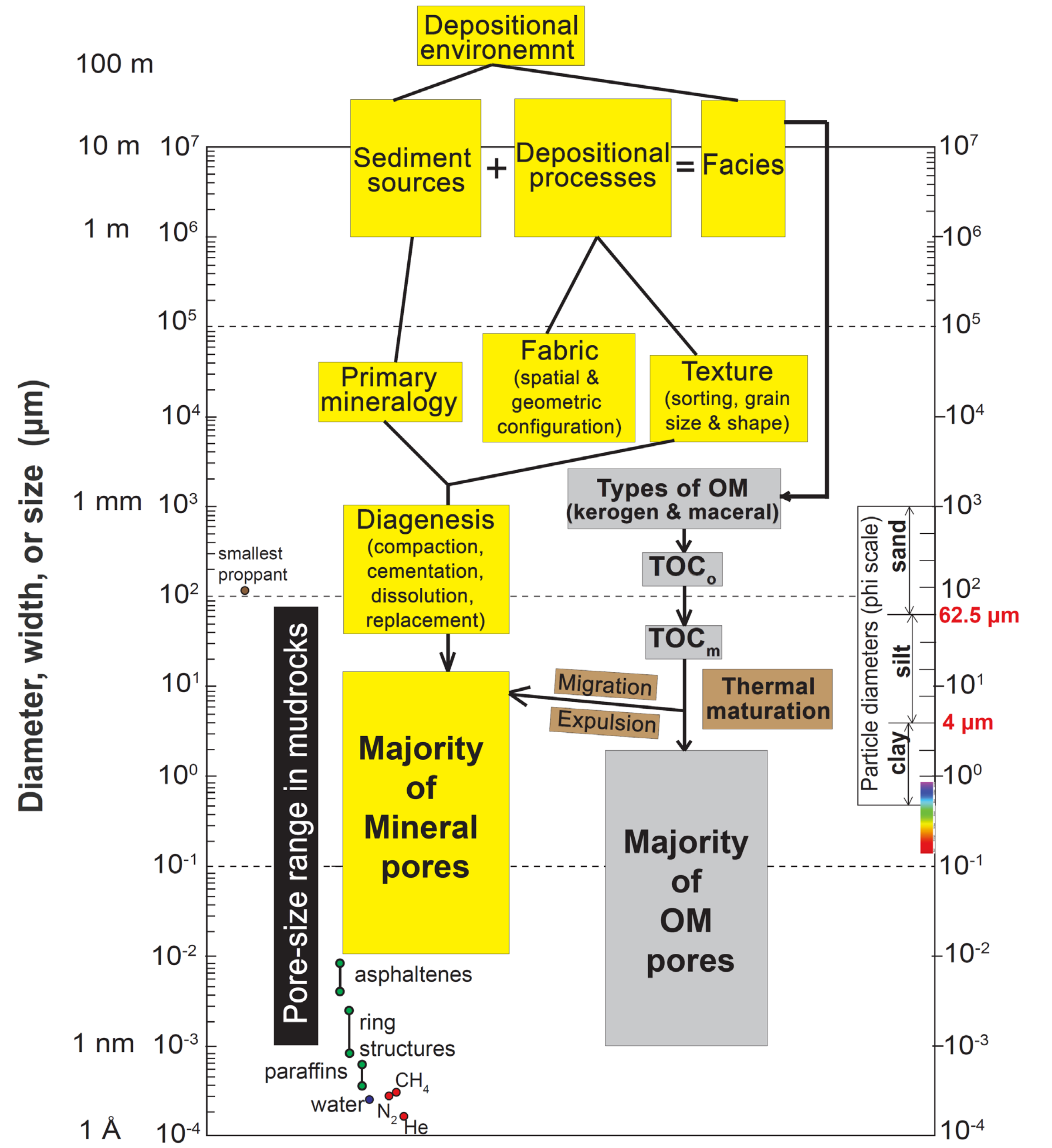
2. Present updated concepts relative to controls on pore systems and their connectivities.

3. Discuss unresolved challenges such as upscaling and REA/REV.



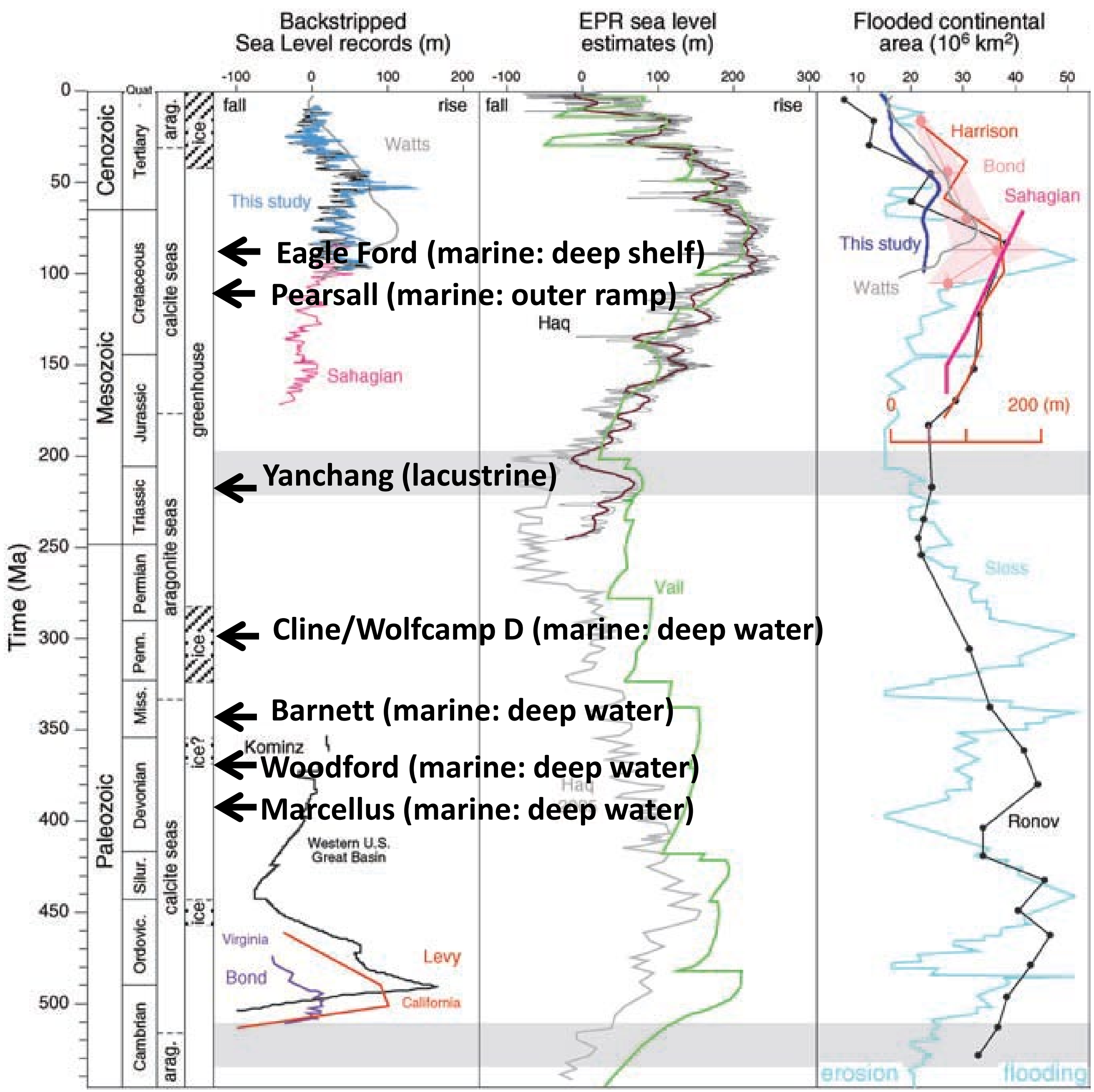
Sizes of Gas, Oil, and Pores

- Nanometer-sized OM pores in solid bitumen are more likely to host gas molecules; μm-sized pores are more likely to host oil.
- 3 nm pore size associated with illite-smectite group of clays (Kulia and Prasad, 2013)
- Adsorption also plays a significant role in OM pores (Zhang et al., 2012)



(modified after Ko, 2017)

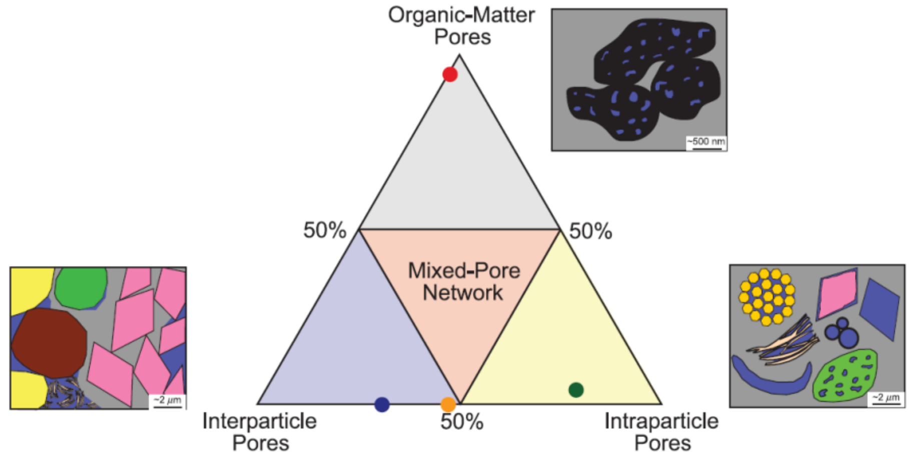
Studied Mudrock Formations

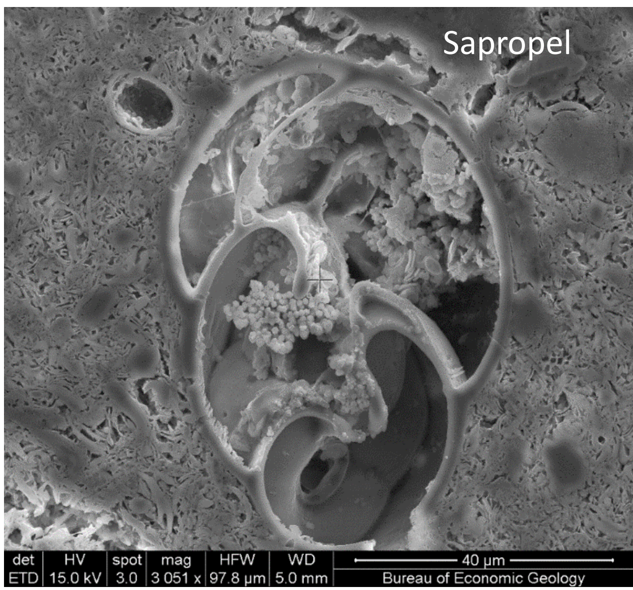


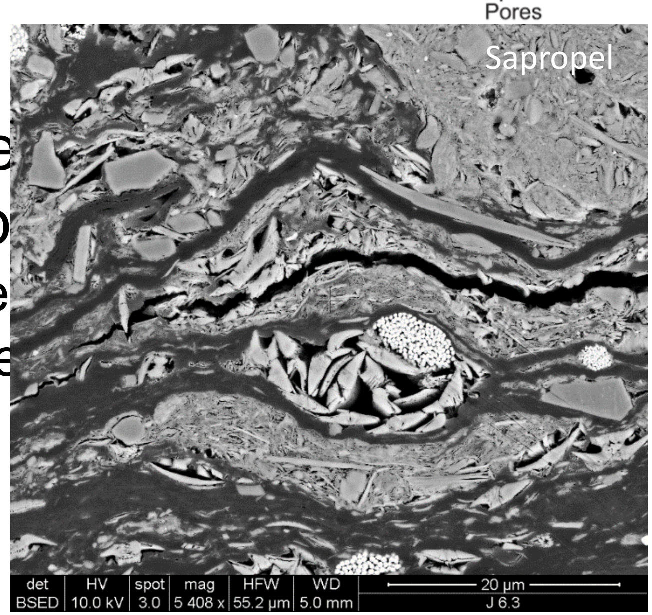
- Pliocene-Pleistocene ODP samples: eastern Mediterranean marine sapropels (32 to 193 m below sea floor)
- USGS drilled shallow outcrop core (Buda to Austin Chalk), McLennan County, TX: shallow burial (103.6 m)
- Supercollider project cores, Ellis County, TX: shallow burial (450 ft)

Introduction of Pore Types: Primary Mineral Pores

- **Mineral pores**
  - Primary mineral pore
    - Interparticle pore
    - Intraparticle pore
  - Modified mineral pore



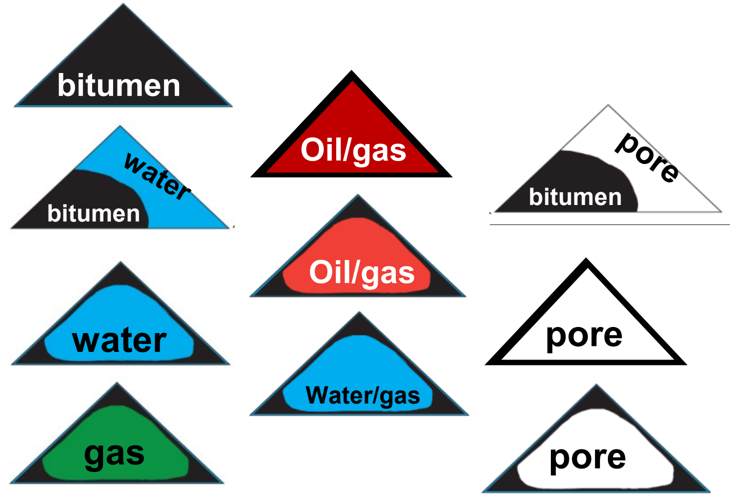


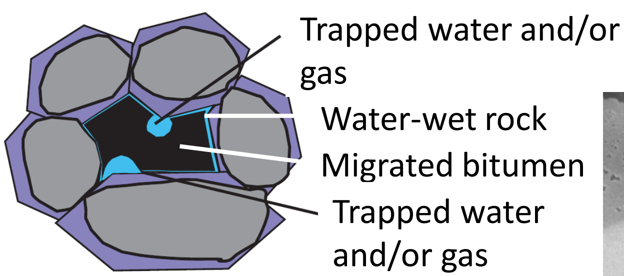


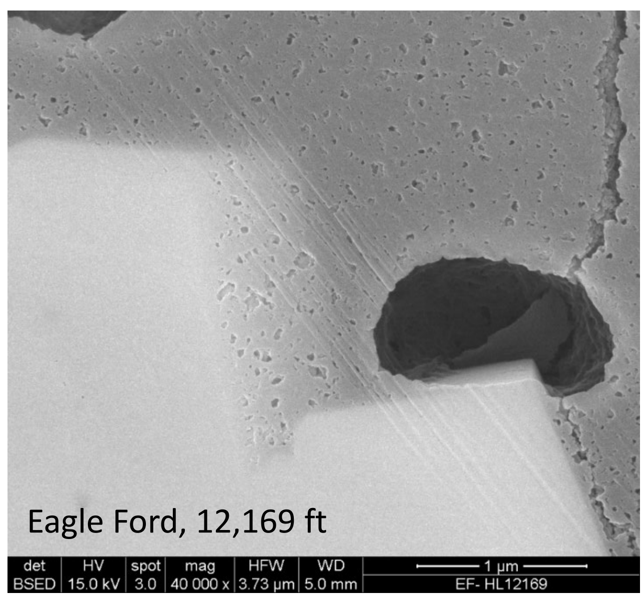
(Loucks et al., 2012; Milliken et al., 2014; Ko et al., 2016)

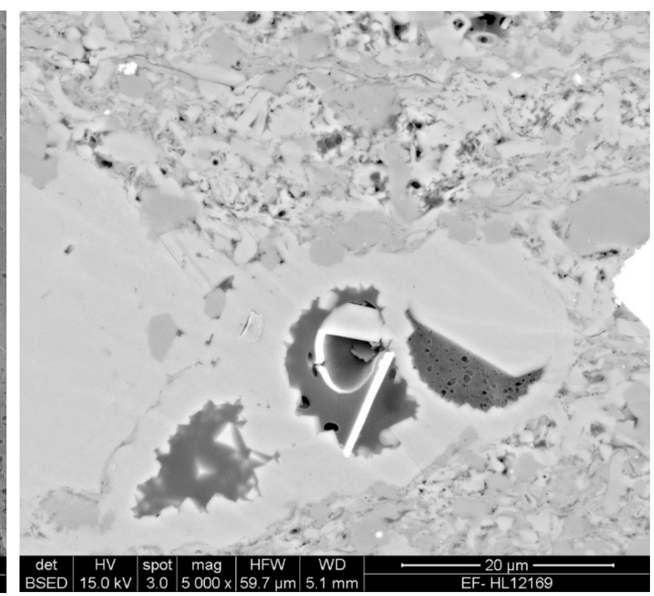
Introduction of Pore Types: Modified Mineral Pores

- **Mineral pores**
  - Primary mineral pore
    - Interparticle pore
    - Intraparticle pore
  - Modified mineral pore









(Loucks et al., 2012; Ko et al., 2016)

Introduction of Pore Types: Modified Mineral Pores

- **Mineral pores**
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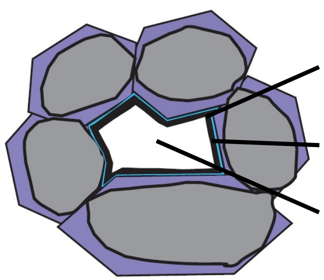
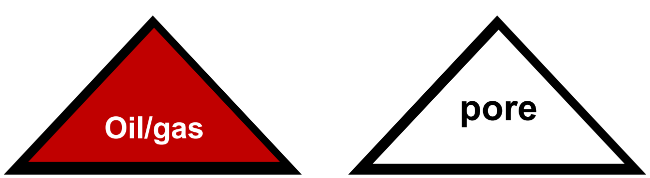
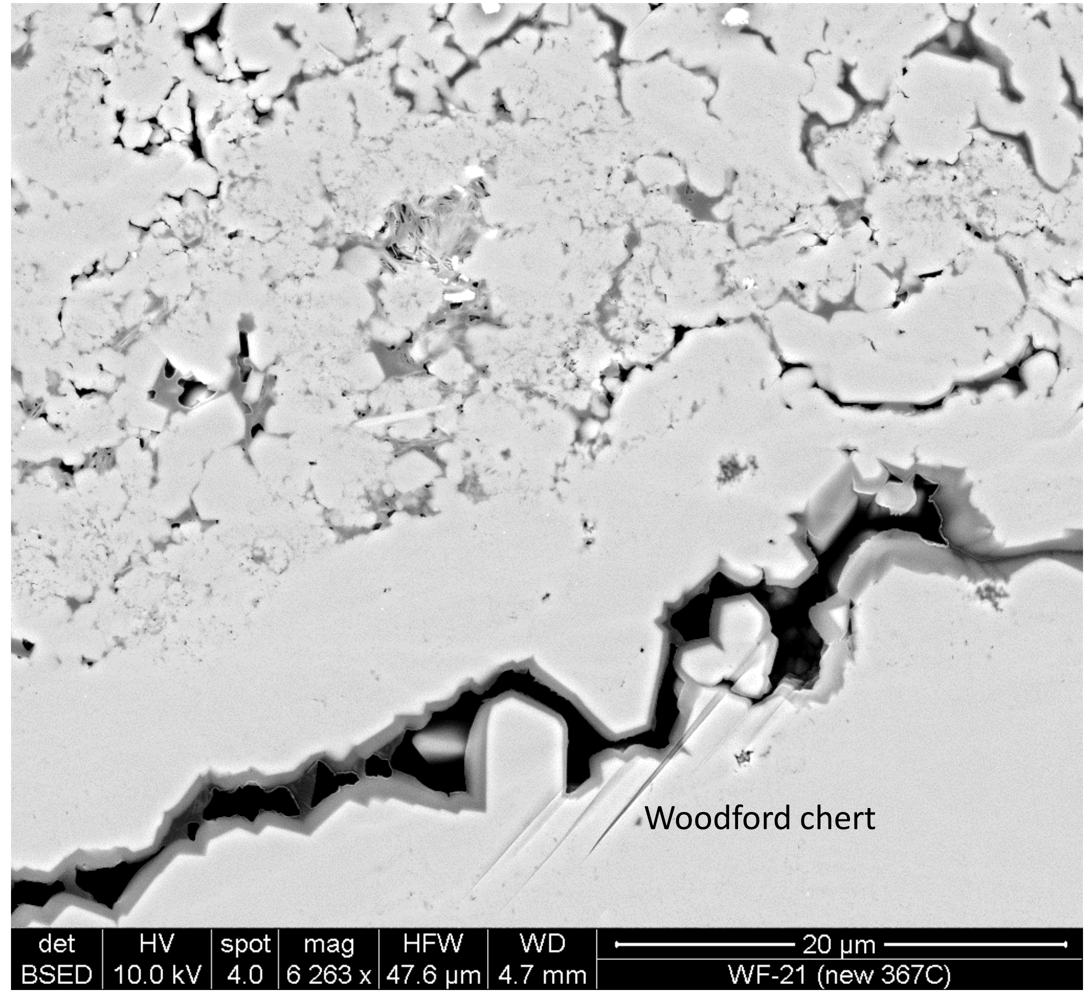
    - Intraparticle pore
  - Modified mineral pore

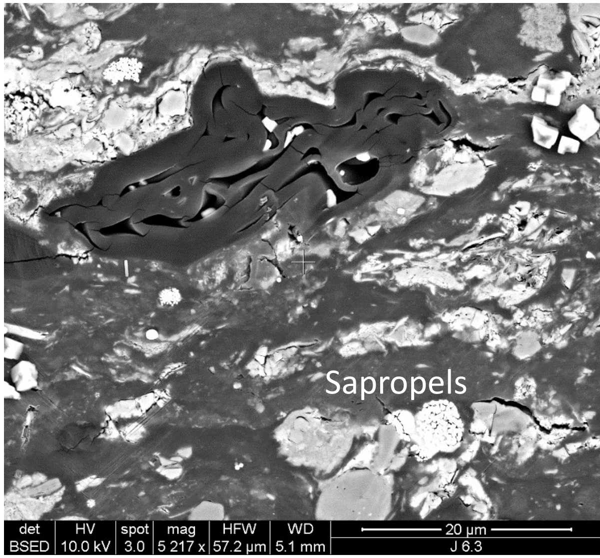


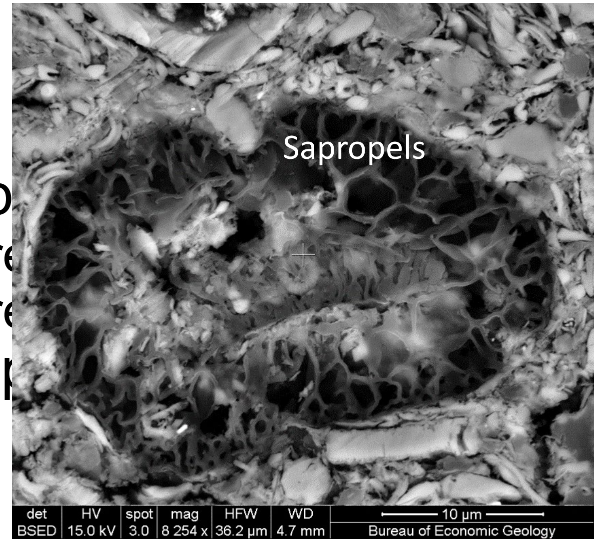


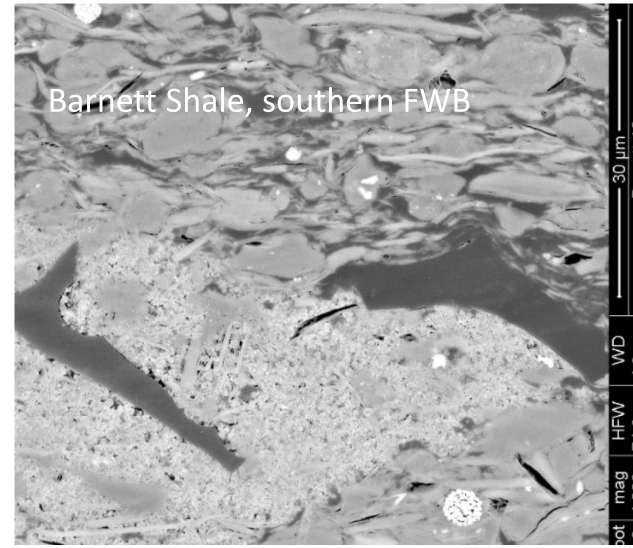


(Loucks et al., 2012; Ko et al., 2016)

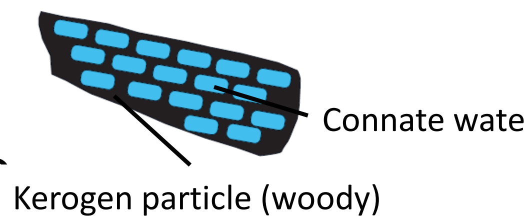
Introduction of Pore Types: Primary OM Pores

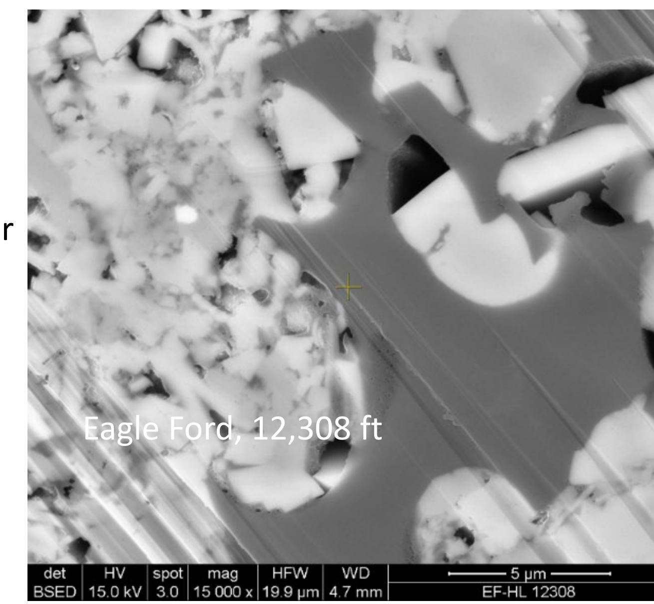






- **OM pores**
  - Primary OM pore
  - Secondary OM pore
    - OM bubble pore
    - OM spongy pore

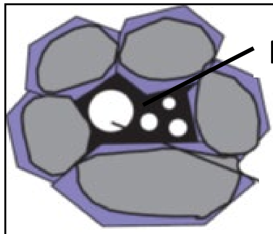


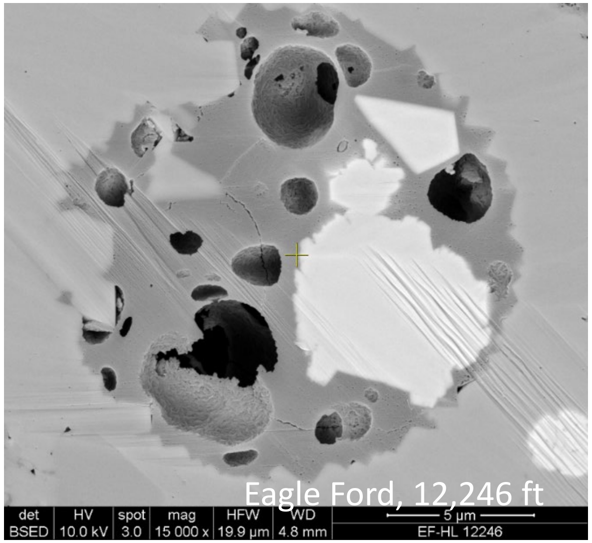


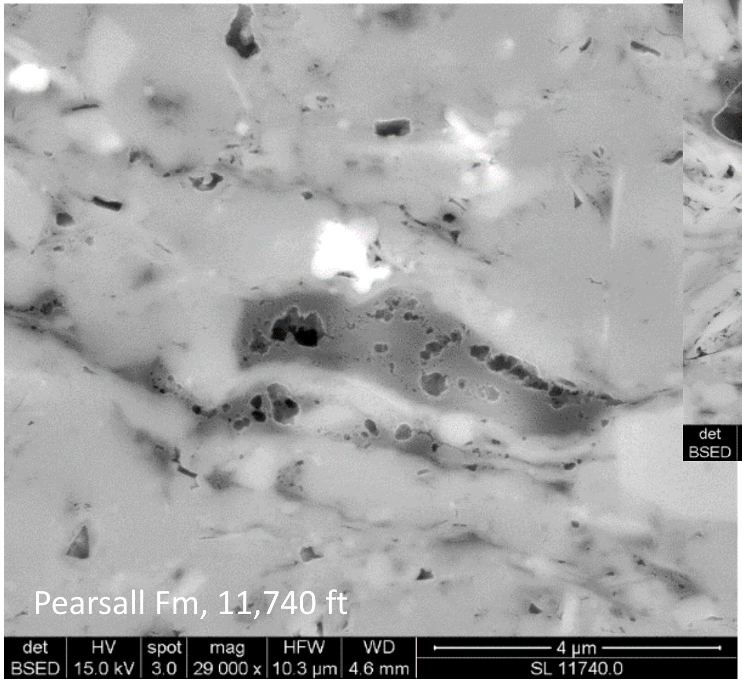
(Milliken et al., 2014; Ko et al., 2016)

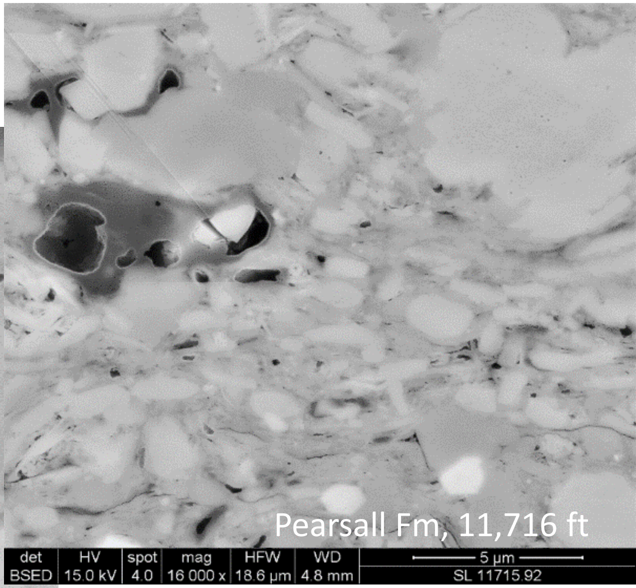
Introduction of Pore Types: OM Bubble Pores

- **OM pores**
  - Primary OM pore
  - Secondary OM pore
    - OM bubble pore
    - OM spongy pore





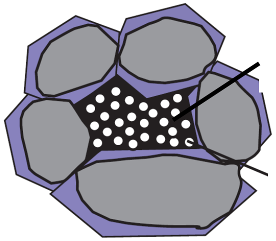




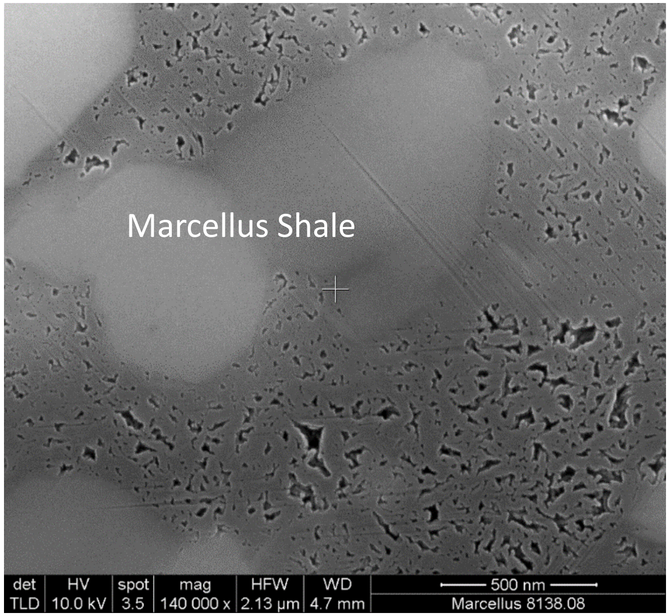
(Loucks et al., 2012; Ko et al., 2016)

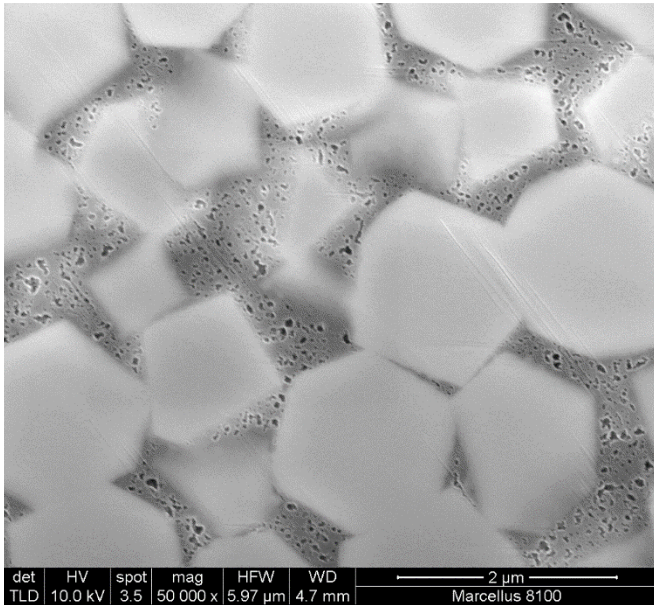
Introduction of Pore Types: OM Spongy Pores

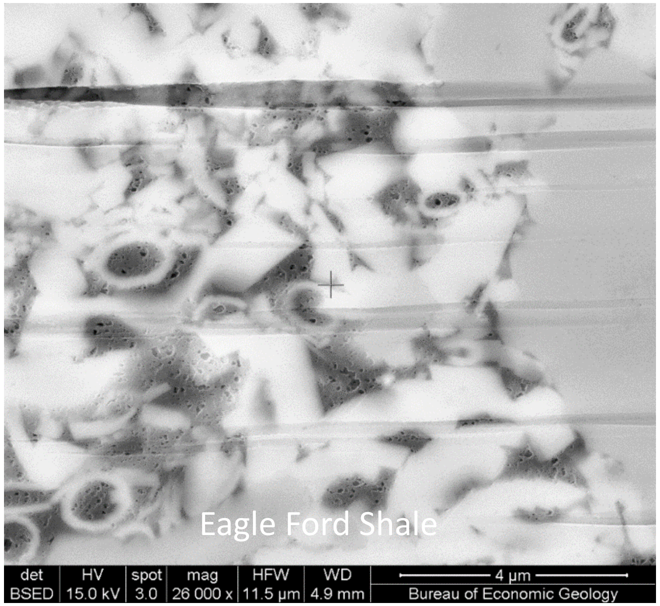
- **OM pores**
  - Primary OM pore
  - Secondary OM pore
    - OM bubble pore
    - OM spongy pore

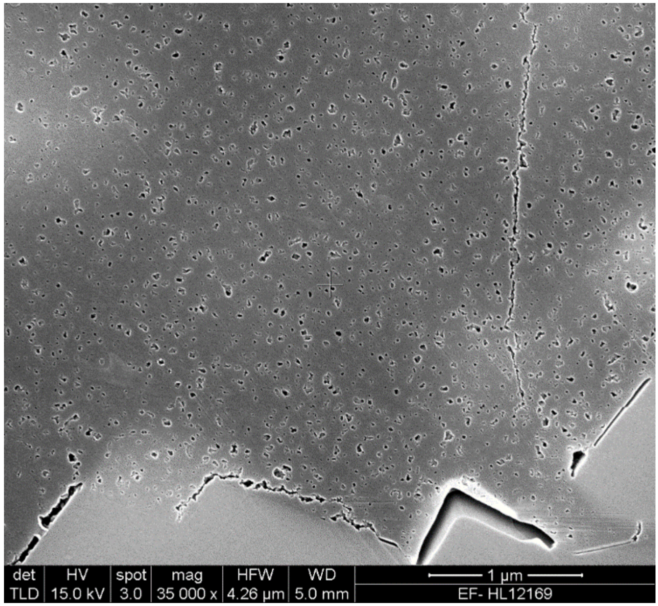


(Ko et al., 2017)



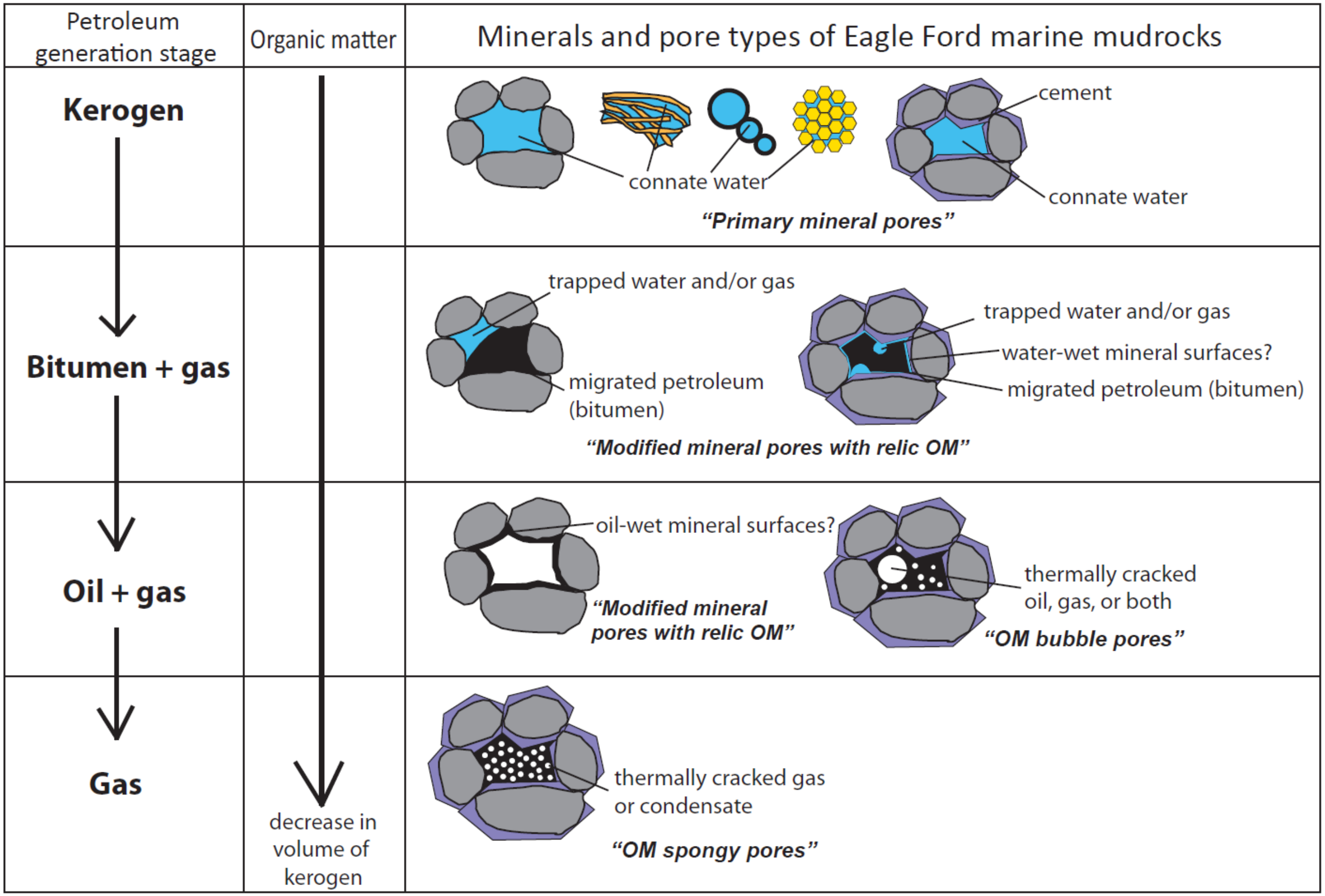






Eagle Ford Pore Evolution Model - Thermal Maturation

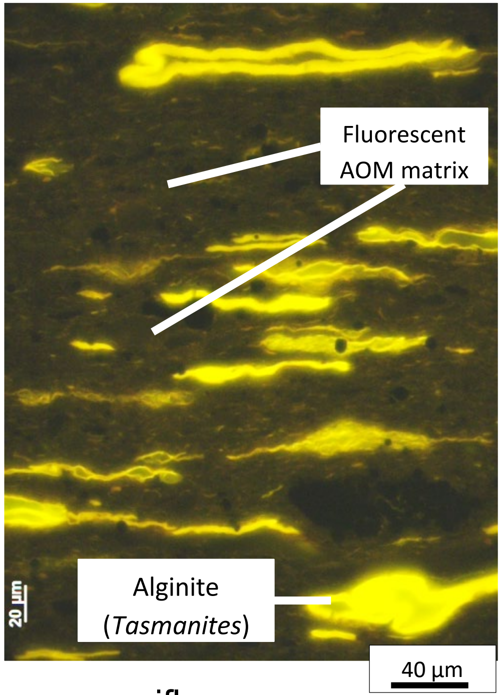
- Combine lab artificial maturation and subsurface Eagle Ford core samples



(Ko et al., 2017)

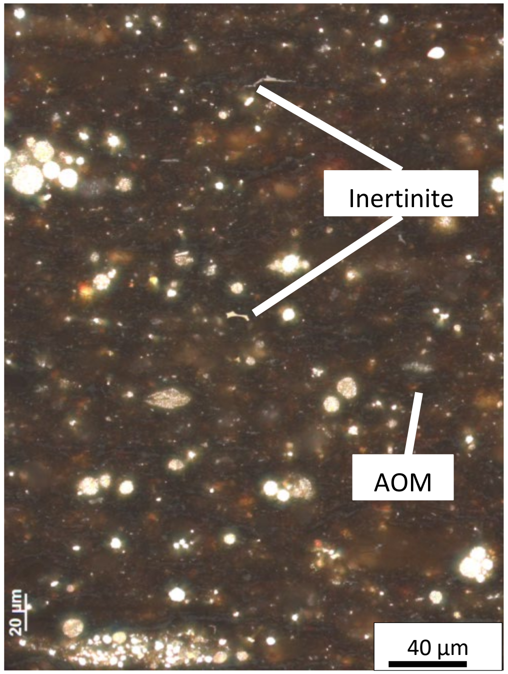
Establish Relationships Between Kerogen Type and Pore Systems

Woodford Siliceous Mudstone



epifluorescence

- Abundant thick-walled prasinophytes (*Tasmanites)* and *Leiosphaeridia* (unknown spherical alga) are present flattened along the bedding plane.



incident white light

- Groundmass contains abundant dispersed, fluorescent amorphous organic matter (AOM). AOM is also called bituminite.
- Inertinite and rare vitrinite

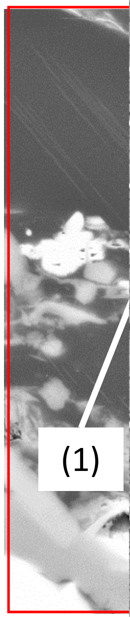
Unicellular *Tasmanites* and *Leiosphaeridia* marine alginite and variably degraded alginite remnants ("ghosts") within the amorphous kerogen are products of cell lysis, photo-oxidation, and microbial alteration; these processes are characteristic of algal bloom periods. Algal bloom episodes and consequential bacterial activity played a significant role in the accumulation of oil-prone kerogen/maceral.

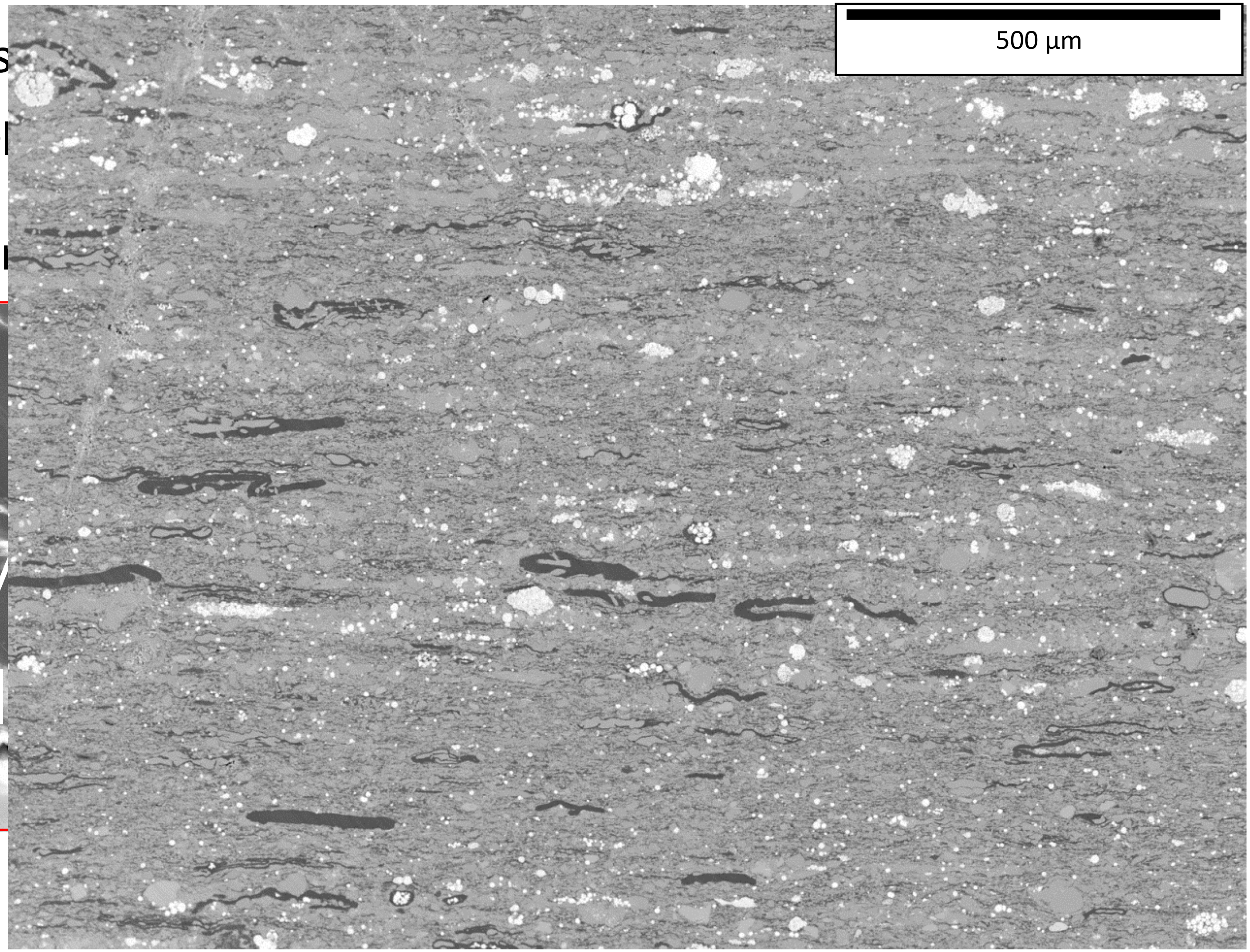
(Ko et al., 2018)

Macerals in SEM: Woodford Mudrock

Based on observation and description of size, abundance, mineral-mixing, and morphological characteristics:

1. *Tasmanites*
2. Stringy, dis
3. Particulate
(vitrinite/inerti

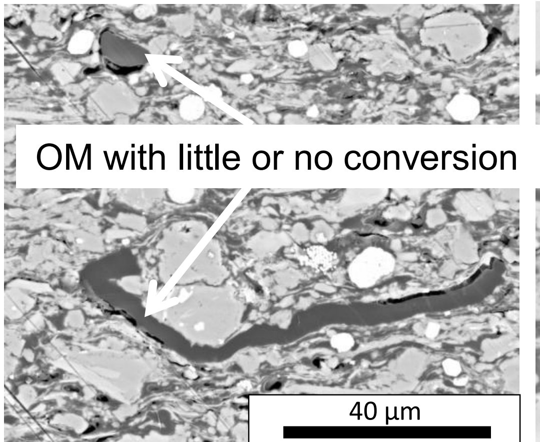


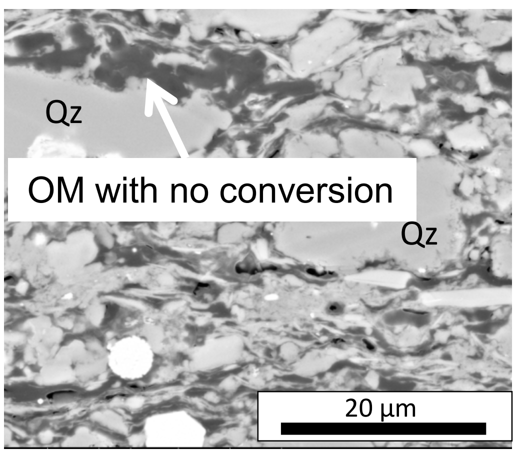


(Ko et al., 2018)

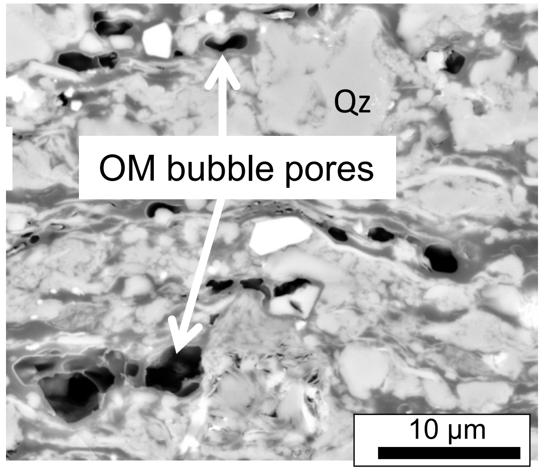
Woodford OM & Pore Evolution: Bitumen Generation

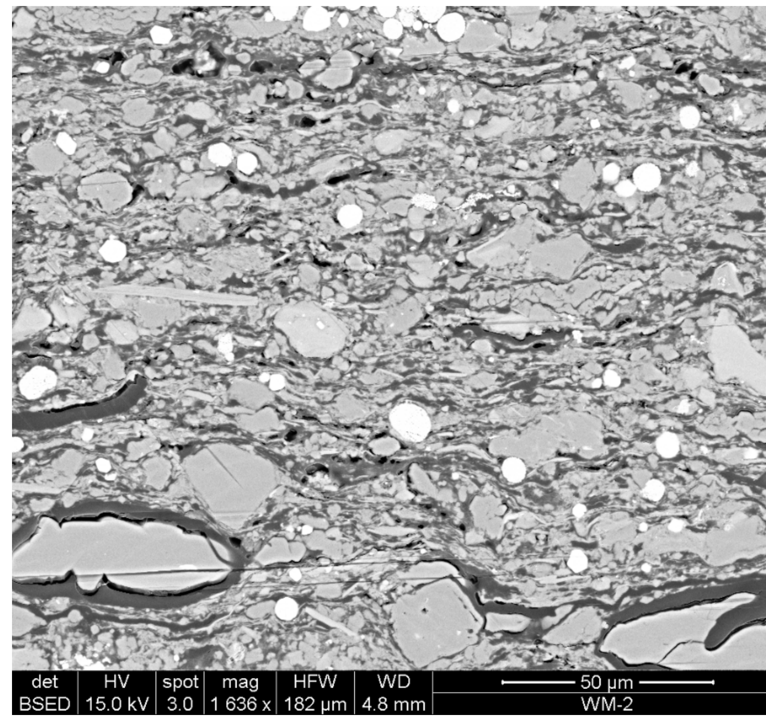
1. Alginite (*Tasmanites*)





2. AOM

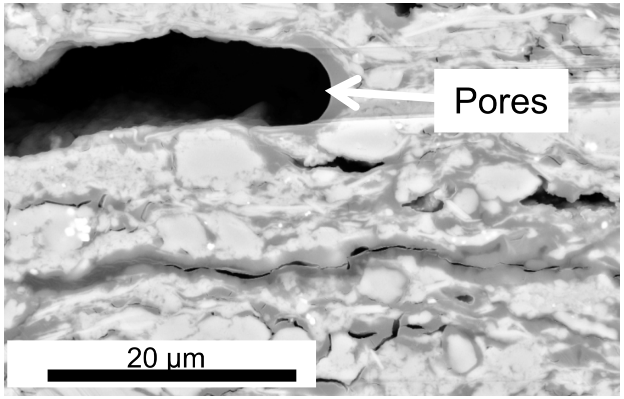




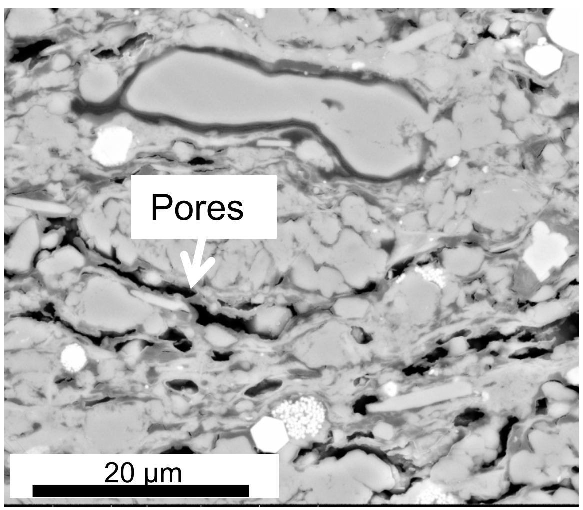
(Ko et al., 2018)

Woodford OM & Pore Evolution: Oil Generation

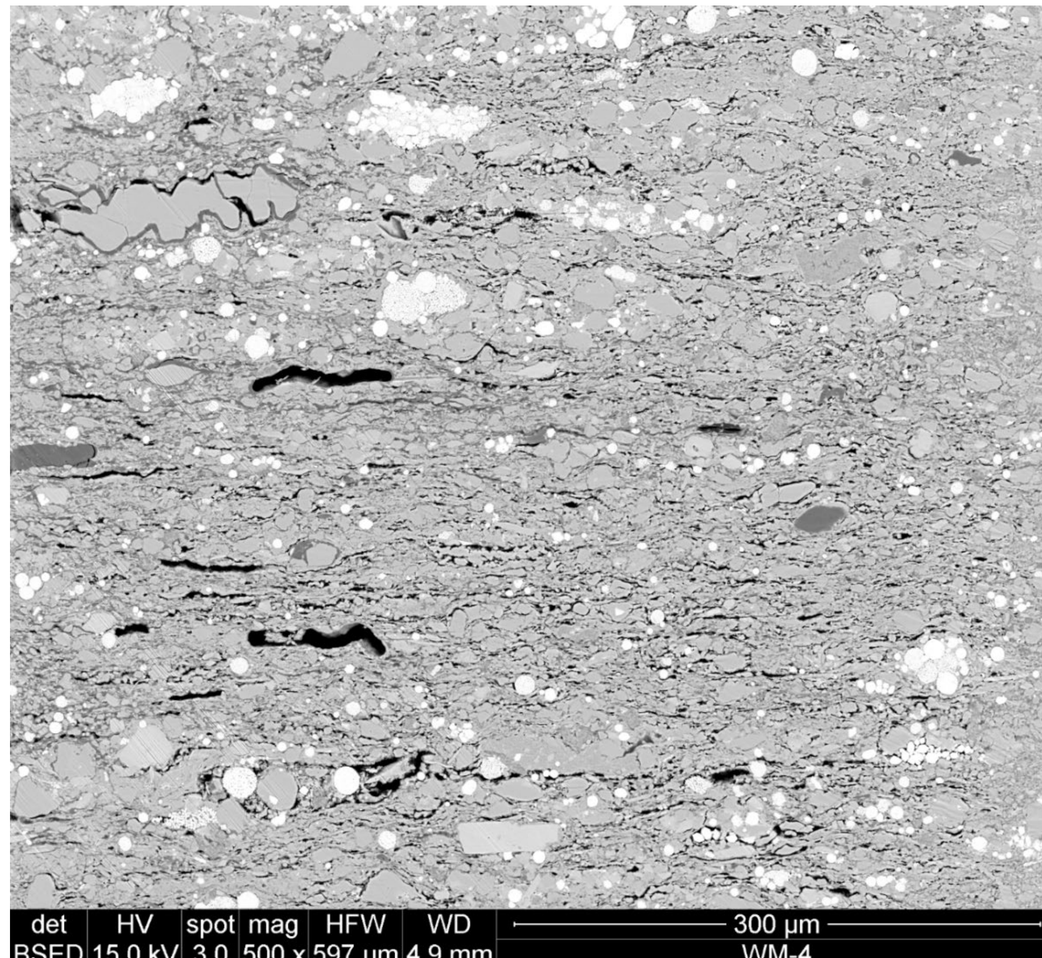
1. Alginite (*Tasmanites*)



2. AOM



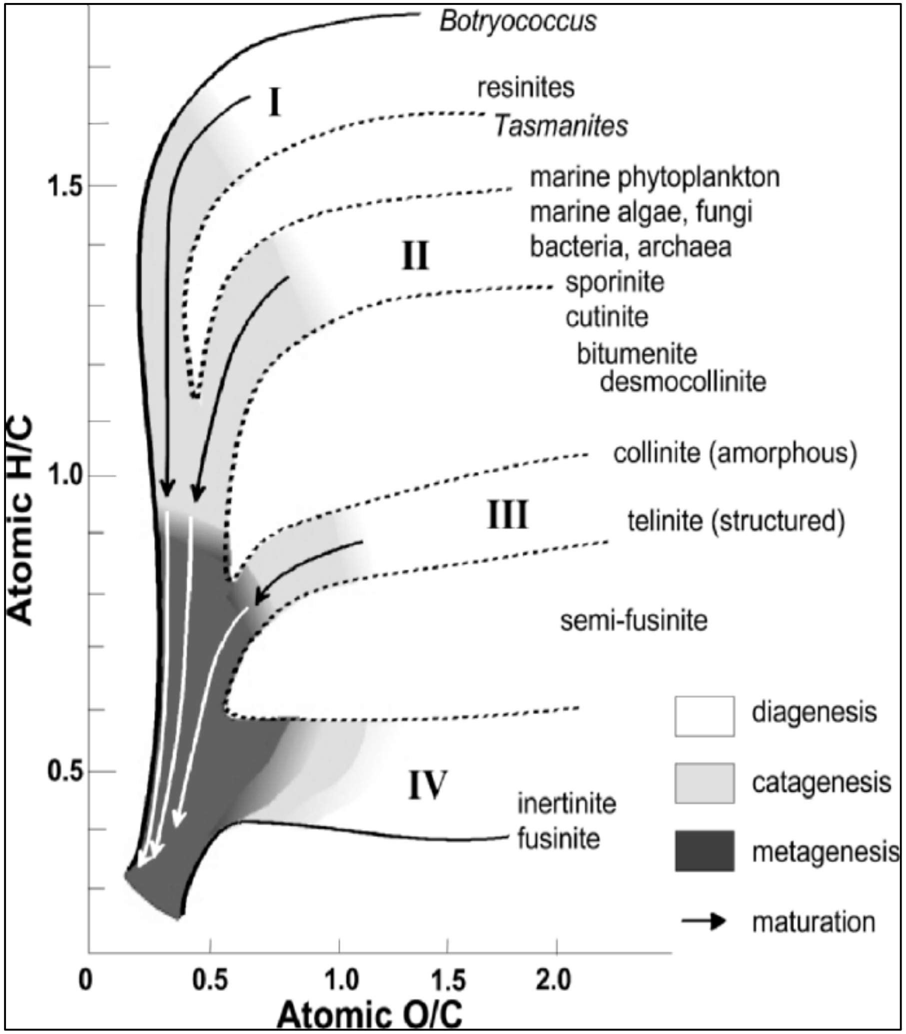
- Transformation ratio is high
- Expulsion efficiency is high



(Ko et al., 2018)

Kerogen Classification under Correlative Optical and SEM

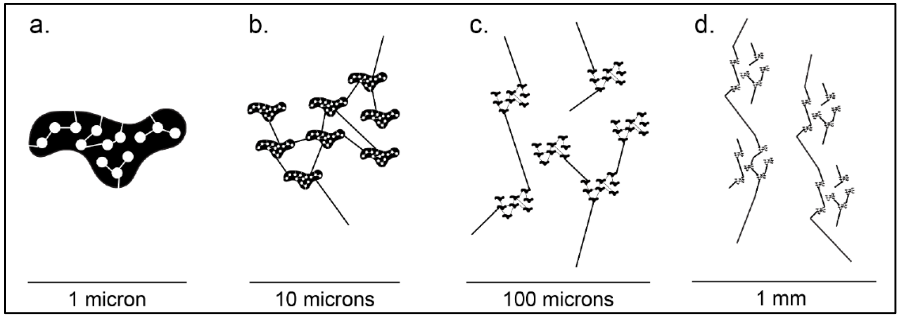
- Inert OM (Type IV)
  - Opaque or dark, non-fluorescent
  - Discrete shape, non-ductile
- Gas-prone OM (Type III)
  - Translucent, very weak or no fluorescence
  - Discrete shape, non-ductile
- Oil-prone OM (Type II)
  - Translucent, weakly to strongly fluorescent
  - OM with different extent of mineral mixing
- Very oil-prone OM (Type I)
  - Translucent, very strong fluorescence
  - OM with little to no mineral mixing



(Walters, 2014)

Major Learning on OM Pores in Recent Years

- Increased variation in OM pore types, sizes, and distribution from immature to oil window.
- Micron-sized OM bubble pores formed in middle to late oil and wet gas/ condensate window.
- Nm-sized OM spongy pores are dominant in dry gas window.
- Significant porosity found in < 5 nm pores in mature mudrocks.
- Correlative optical and electron microscopy studies are useful to link organic petrographer-defined maceral to OM and pores under SEM.
- Petrographic recognition of OM as secondary migrated solid bitumen rather than primary kerogen is important for reservoir properties like wettability.
- In order to form a connected solid bitumen network, the connected interparticle pore network system needs to be existed before petroleum generation.

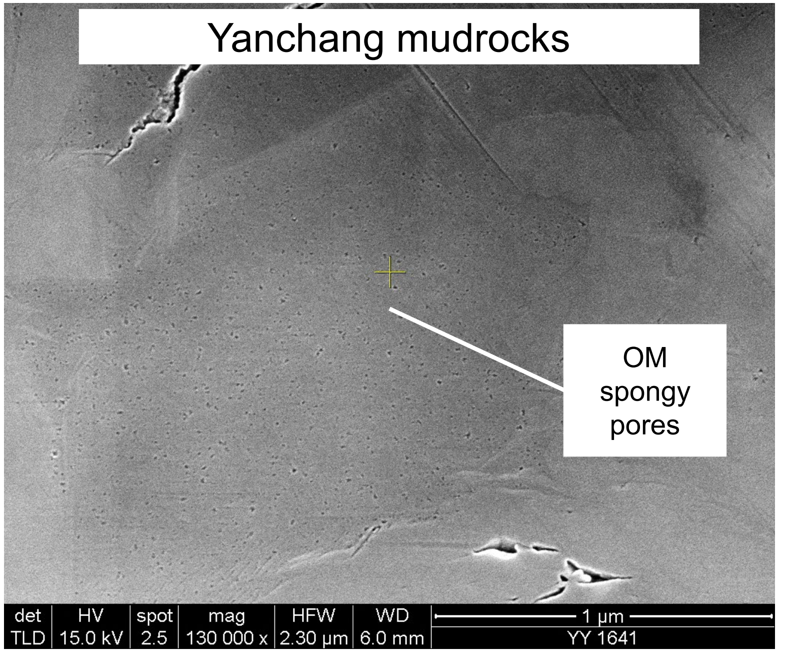


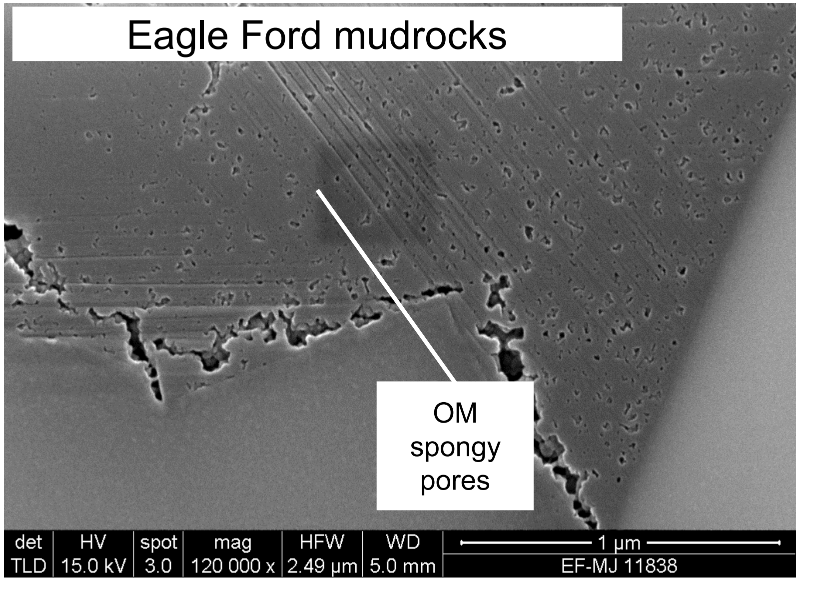
(Bernard et al., 2012; Loucks and Reed, 2014; Ko et al., 2016; Ko et al., 2017; Daigle et al., 2018; Wood et al., 2018)

Major Learning on OM Pores in Recent Years

- Types of OM and macerals affect the origin and evolution of OM pores owing to differences in chemical compositions and generation kinetics.

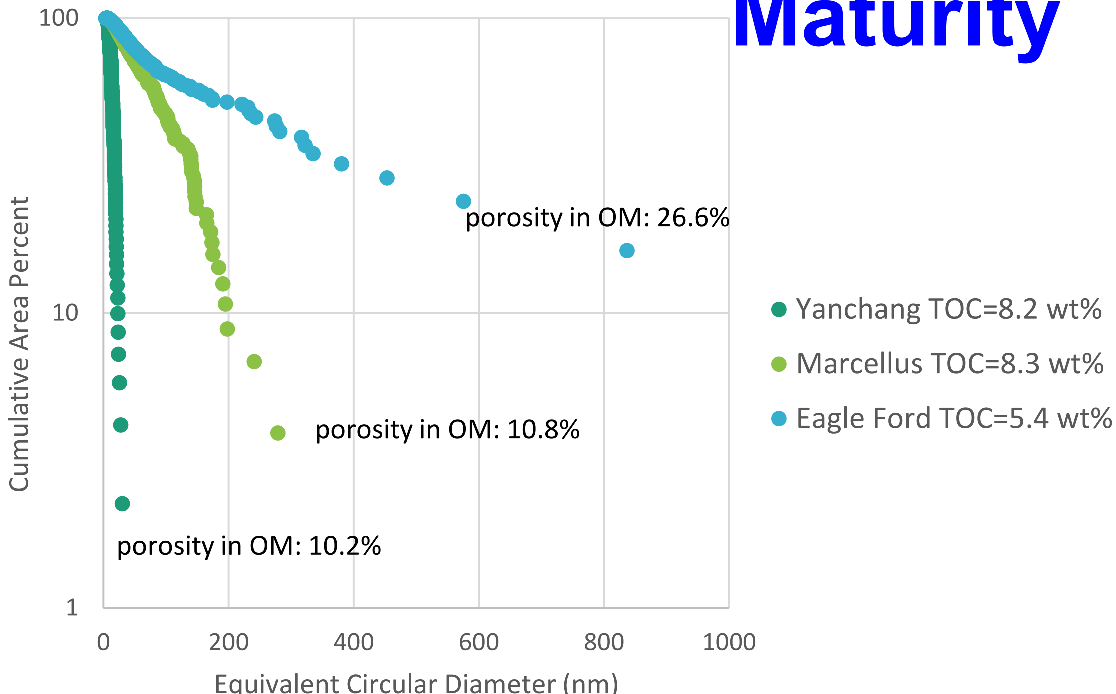
- The sizes of OM pores in the compaction-dominated clay-rich mudstones can be one to two orders of magnitude smaller than those in the mudstones that display abundant early cementation.





(Ko et al., 2017)

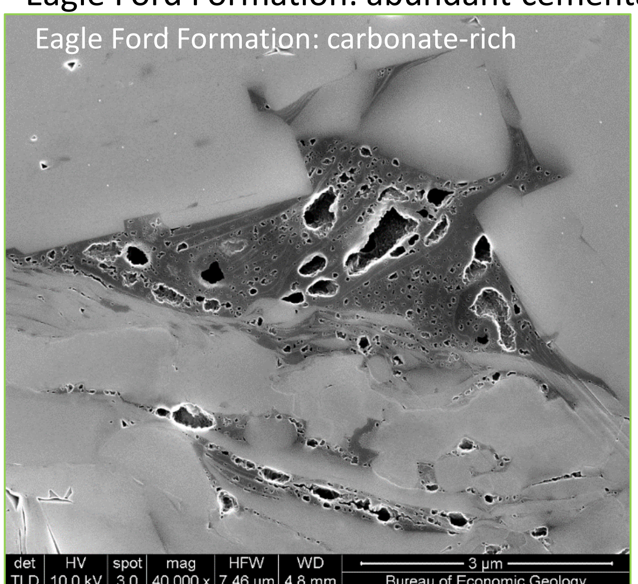
Comparative Sizes of OM Pores at Similar Maturity



Porosity in OM is a function of:

- **Thermal maturity**
- **TOC**
- **Organic matter type**
- **Compactional state**

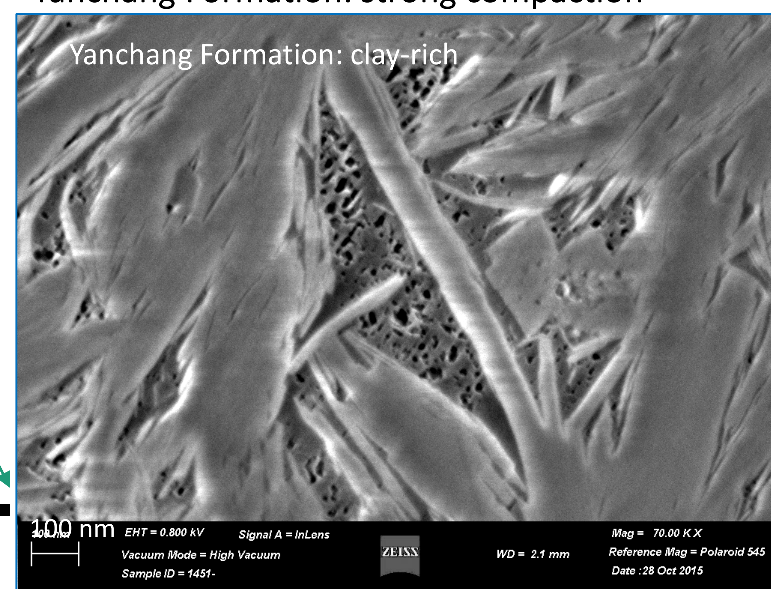
Eagle Ford Formation: abundant cementation



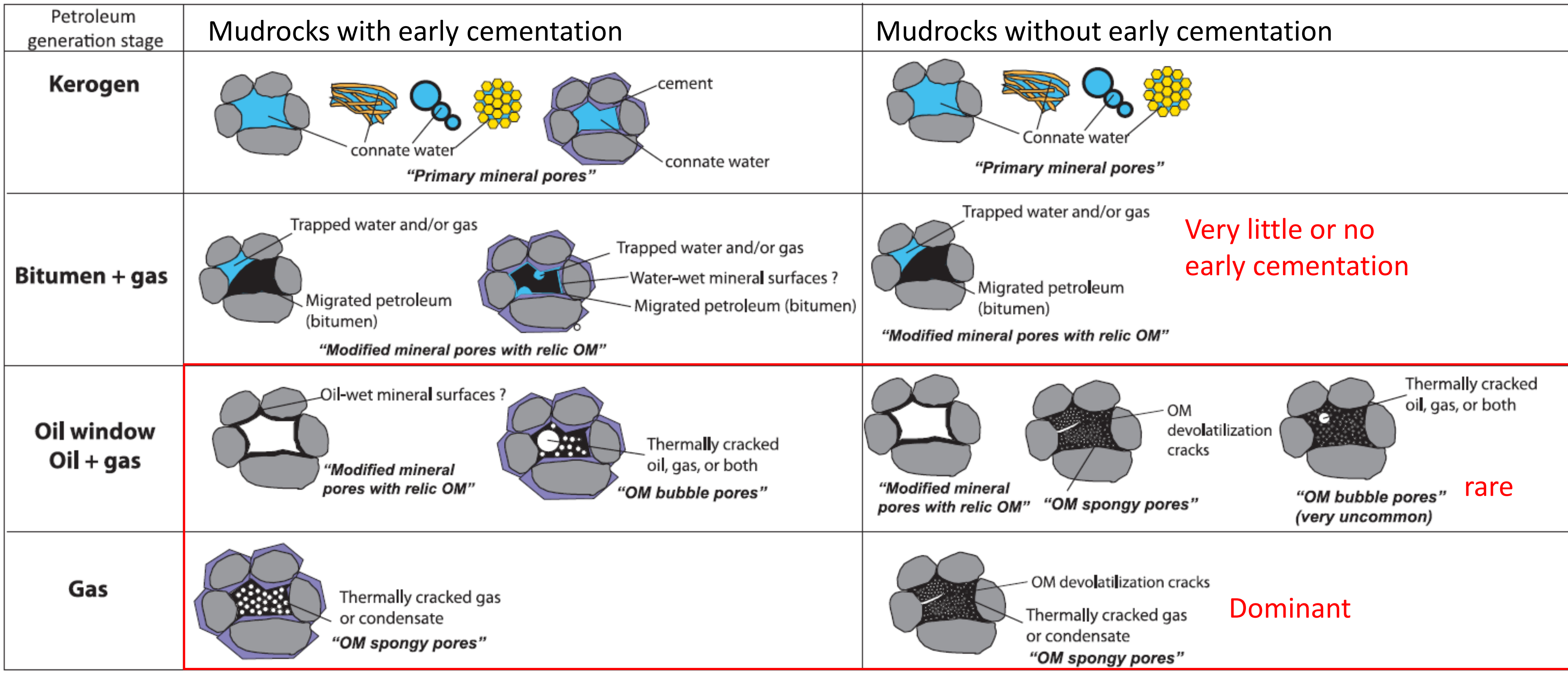
Note contrasting scale:

3 microns vs 100 nm

Yanchang Formation: strong compaction

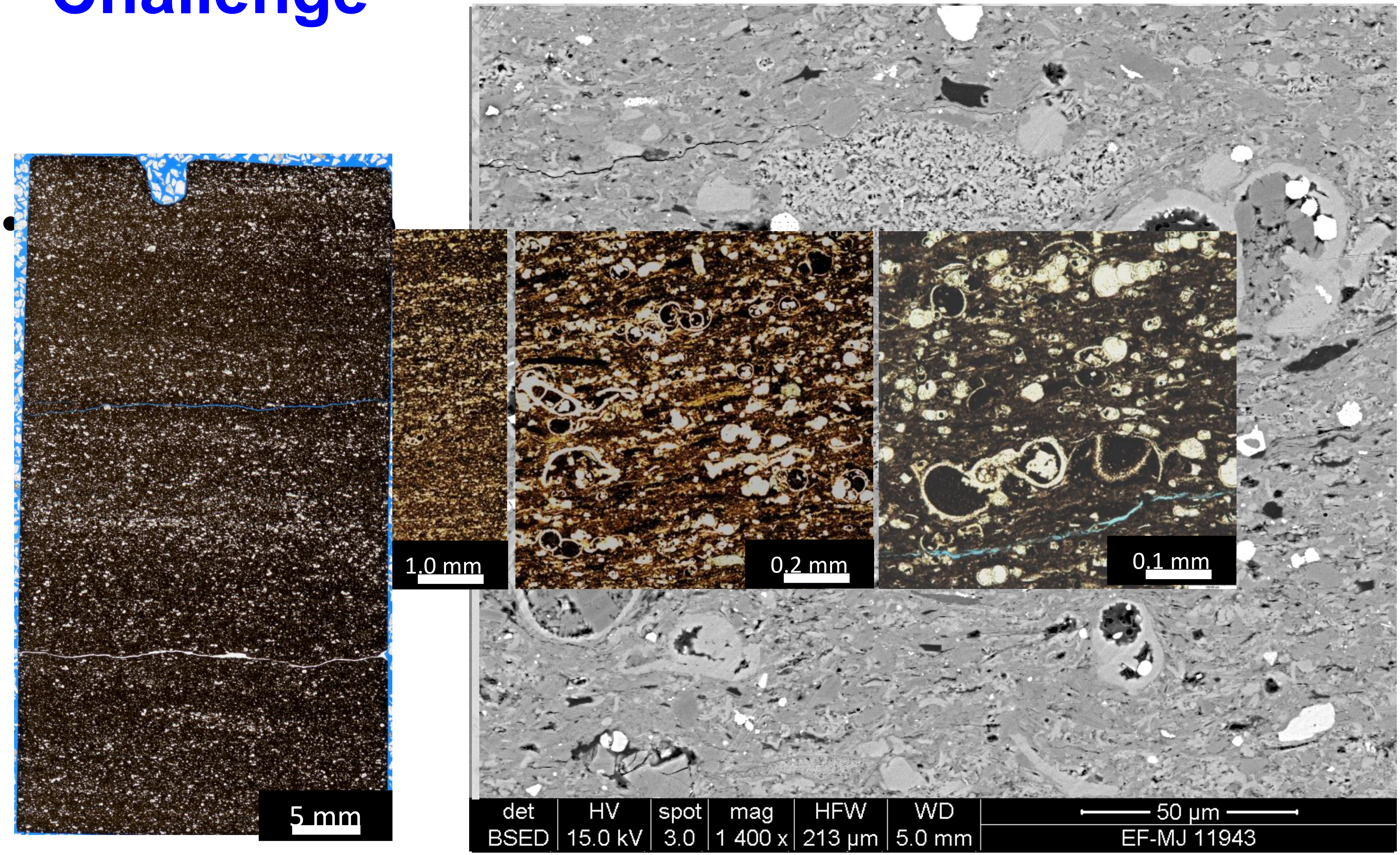


Pore Evolution & Expulsion Differ in Detrital vs. Biogenic Sediment Dominated Mudrocks

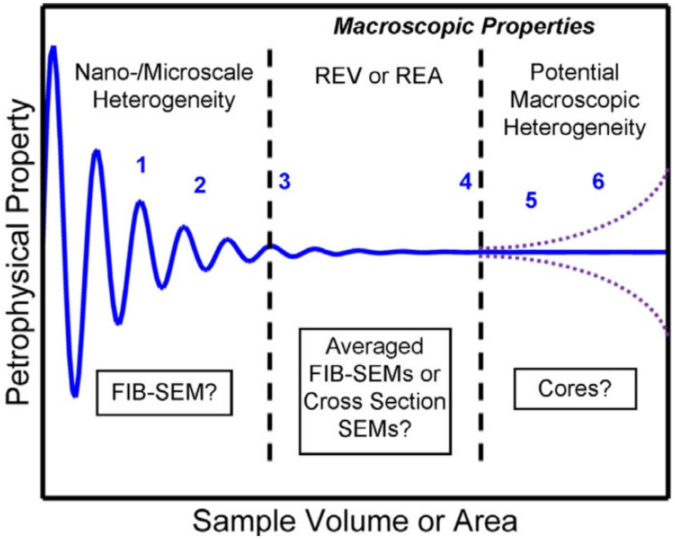


- A mudrock with abundant early calcite and quartz cementation retards compaction and adds brittleness to the rock (e.g. Eagle Ford, Pearsall).
- A mudrock without early cementation contains smaller OM pores than mudrocks with abundant cements (e.g., Yanchang, upper Marcellus).

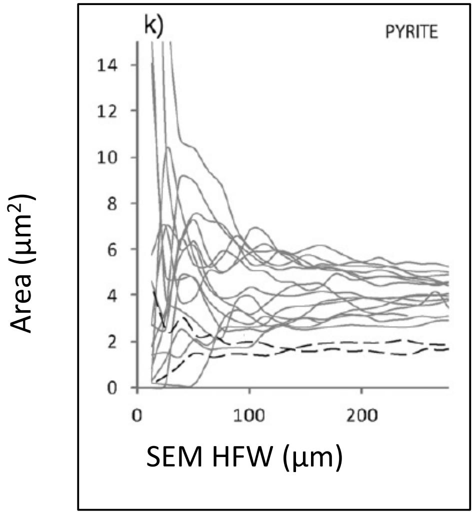
Upscaling Challenge



Challenge: Representative Elementary Area/Volume (REA/REV)



(Kelly et al., 2016)



(Houben et al., 2016)

- REA/REV definition: smallest sample volume or area over which a measured attribute of a media renders a value representative of the bulk media itself

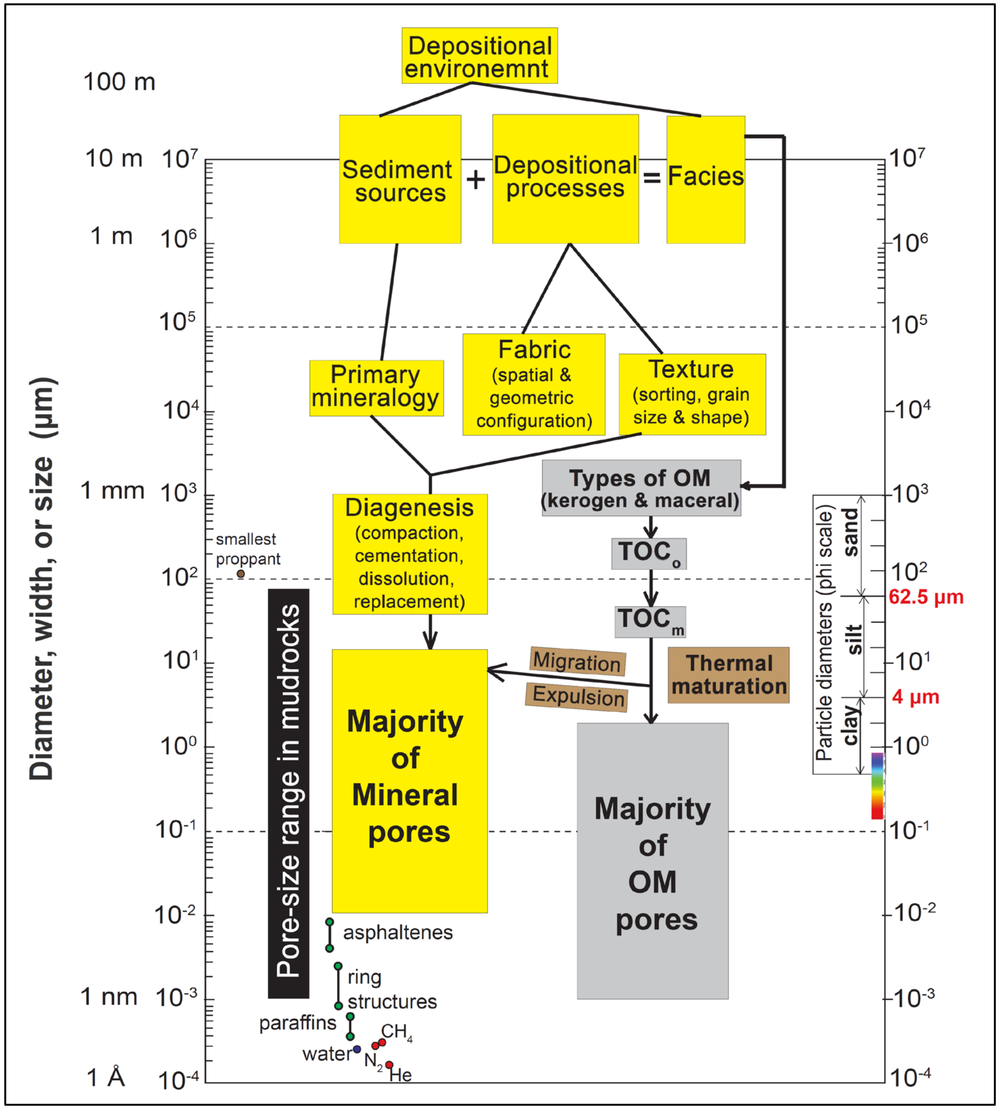
- REA for mineralogy was investigated using box counting method by other researchers:
  - 140 μm* 140 μm (Posidonia, Klaver et al., 2012)
  - 155 μm* 155 μm (Boom Clay, Hemes et al., 2013)
  - 250 μm* 250 μm (Opalinus Clay, Houben et al., 2014)
  - 200 μm* 200 μm (Haynesville and Bossier, Klaver et al., 2015)
  - 200 μm* 200 μm (Posidonia, Houben et al., 2016)

- REA for mineralogy and texture might be different by facies in each formation.

Challenges

- More studies are needed for correlating organic petrographer's maceral to OM in SEM and its pore evolution.

- More studies are needed to link pore systems to depositional facies and stratigraphy.



Conclusion / Applicability

- The thermal maturity-related pore evolution model helps predict pore types and heterogeneity.

- Mineralogy is important but how minerals were arranged in mudrocks (texture and fabric) and origins of minerals are as important as mineralogy.

- Diagenesis, especially early cementation, affects pore sizes and possibly petroleum expulsion.

- Pore evolution is affected by types of kerogen and maceral and the kinetics of kerogen and maceral. Predicting algal bloom events in exploring and producing areas can be important.

- Texture (sorting and grain sizes) of silt fractions affects PSD. The effect of sorting is more significant than grain sizes.

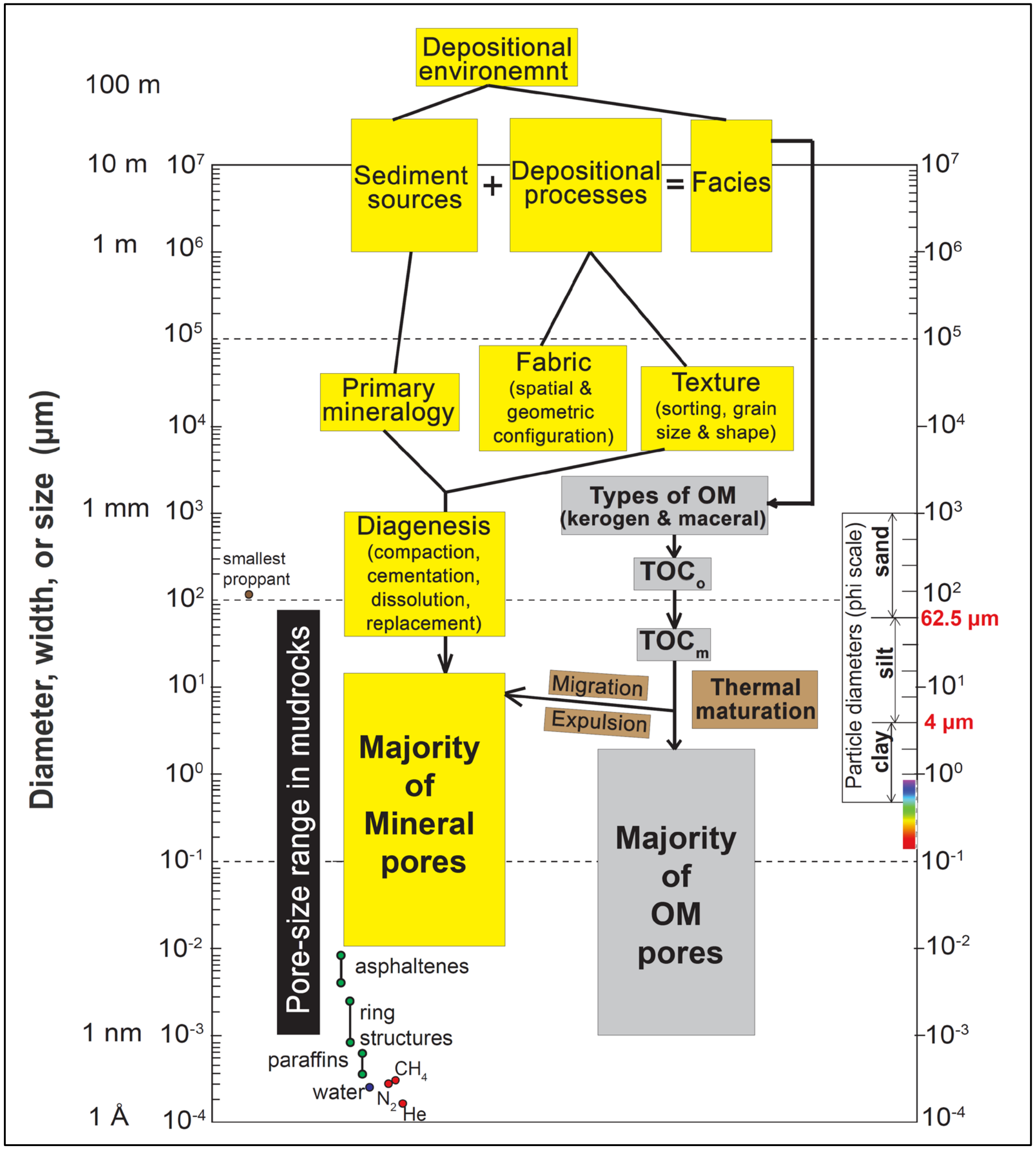
Ongoing Research Areas

- Mudrock diagenesis
  - Investigate the impact of quartz cementation on mudrock reservoir mechanical properties.
  - Investigate the diagenesis of OM-lean facies: are they flow barriers or pathways/reservoirs?
  - The relationship between cementation and compaction and at which depth mudrocks reach compaction equilibrium is still unknown

- Link pore systems to depositional facies and stratigraphy

Back up slides

Establish Relationships Between Lithology and Pore Systems



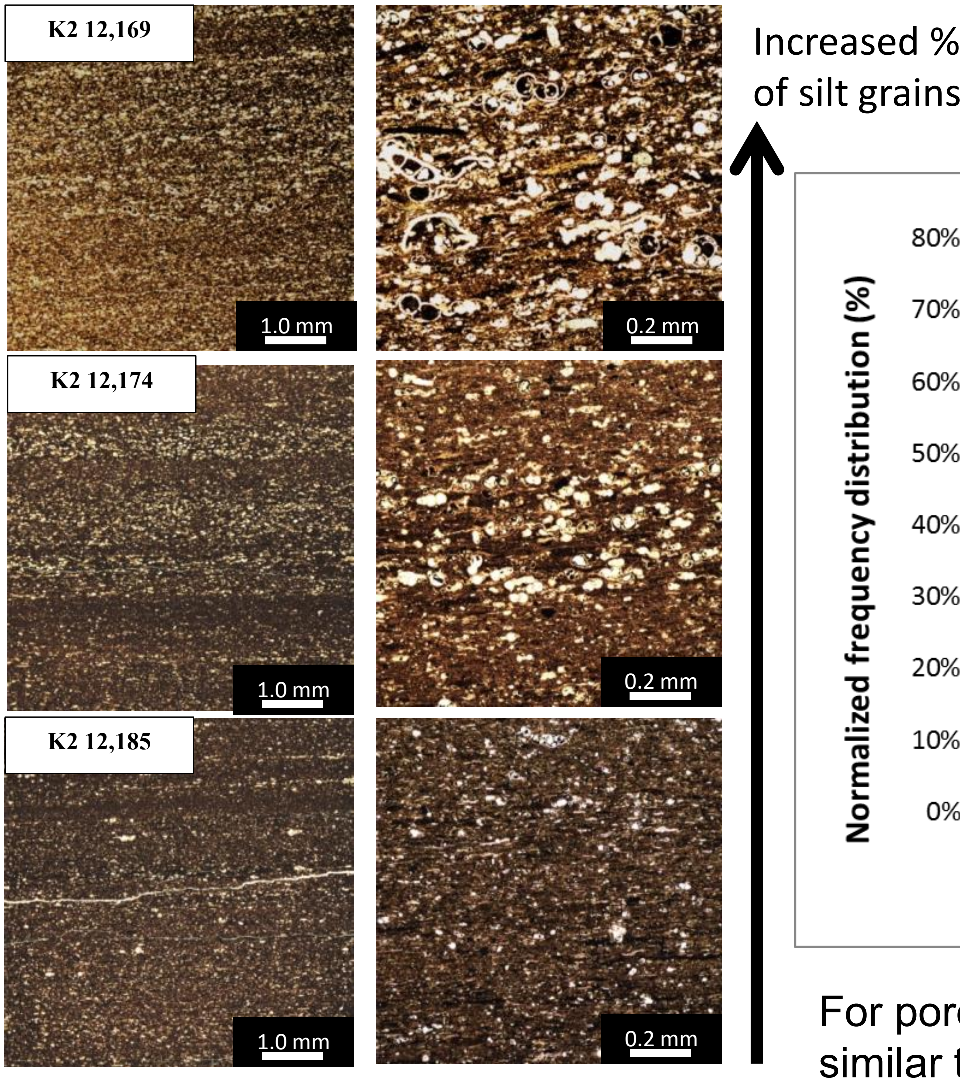
(modified after Ko, 2017)

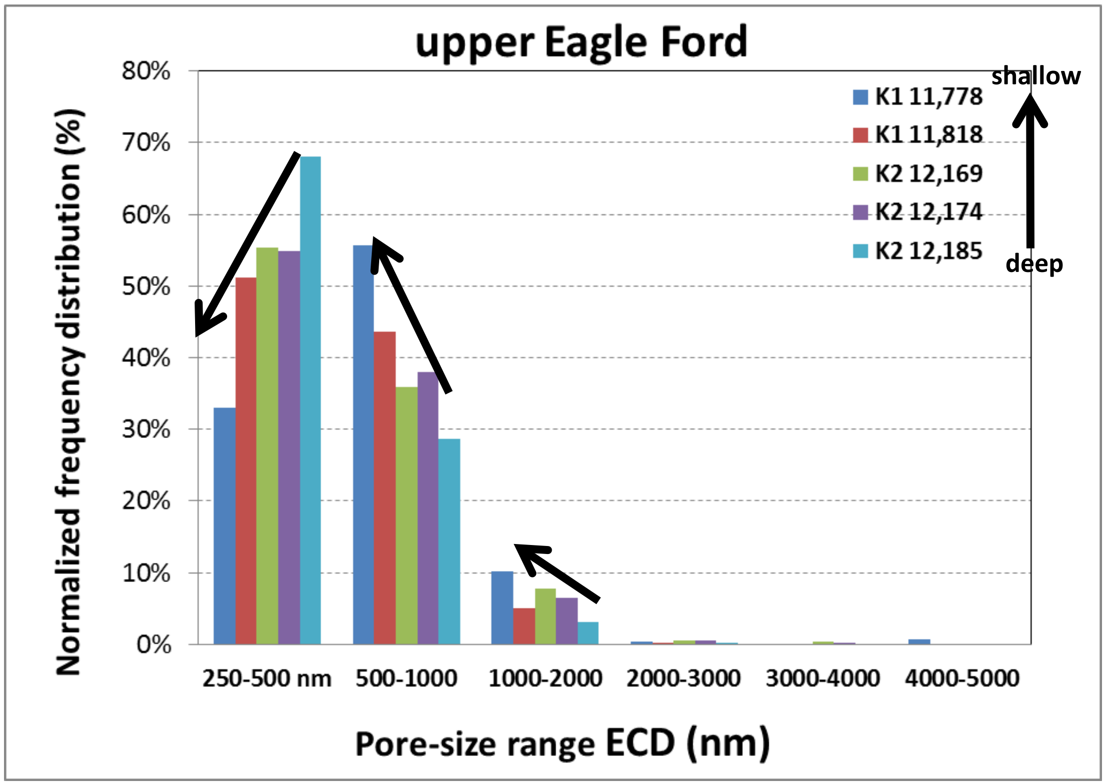
Two Mudrock Systems: Case Studies

- **Mudrocks with similar lithofacies/mineralogy**:
  - Eagle Ford (UEF marls), TX
  - Yangchang Fm (Chang 7), Ordos Basin, China

- **Mudrocks with similar lithofacies/ mineralogy**:
  - With minimum diagenetic alteration, **texture** (grain size, shape, and sorting) **and fabric** variation (arrangement of particles) affects size distribution of mineral pores and OM pores
    - Sorting and grain sizes of silts affect PSD of mineral pores. The effect of sorting is greater than grain sizes.

Pore-Size Distribution Affected by Texture: EF Marls





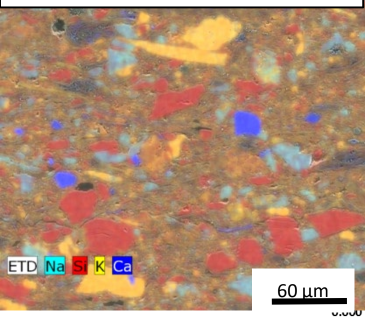
For pores < 200 nm, N_2 adsorption data also shows similar trend.
Deep sample has more nanopores than shallow samples do.

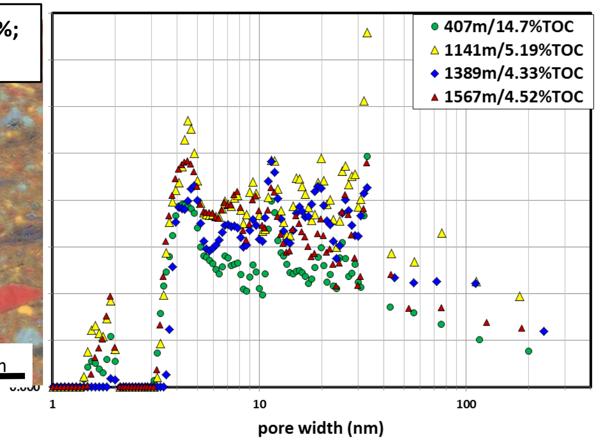
(Ko et al., 2017)

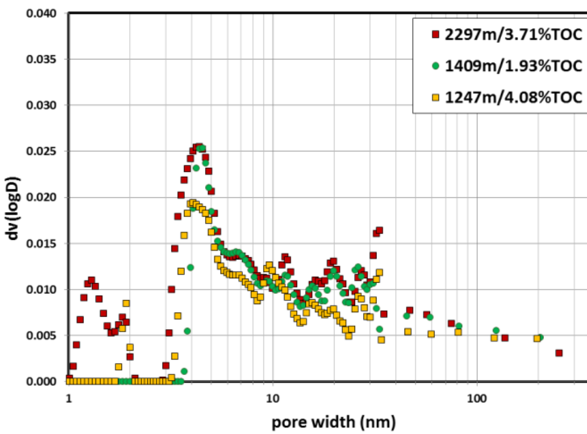
N_2 Pore-Size Distribution of Upper Triassic Yanchang Formation Samples

- Pores > 200 nm were not included

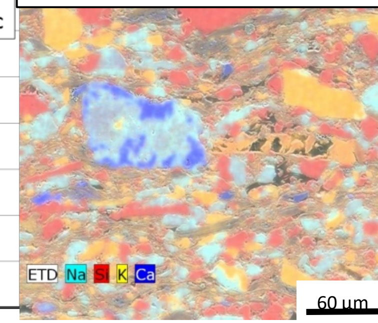
Depth = 1,567 m; He ϕ = 5.68 %; N2 ϕ = 5.6 %



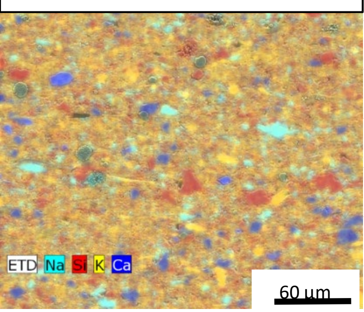


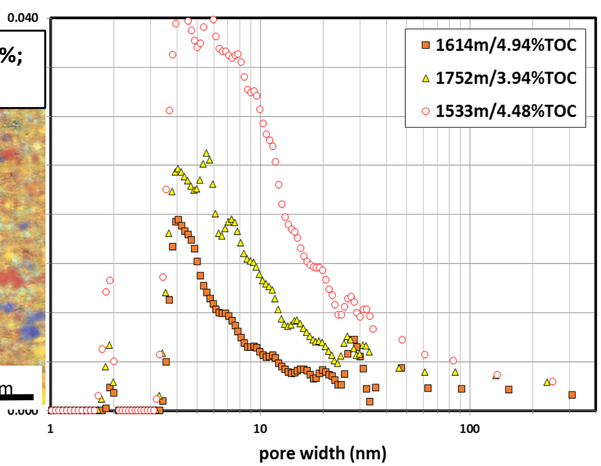


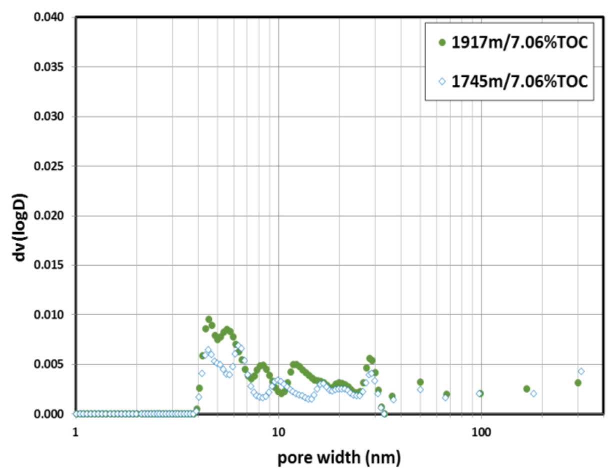
Depth = 1,247m; He ϕ = 2.66 %; N2 ϕ = 3.5 %



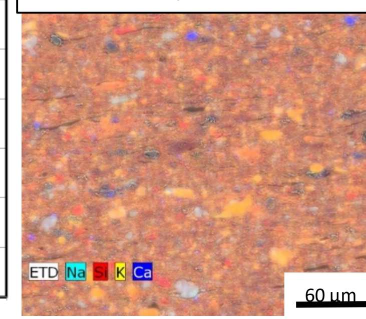
Depth = 1,533 m; He ϕ = 1.69 %; N2 ϕ = 7.5 %





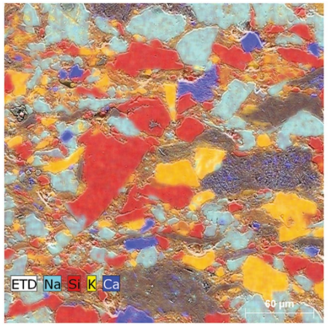


Depth = 1,745 m; He ϕ = 4.23 %; N2 ϕ = 1.1 %

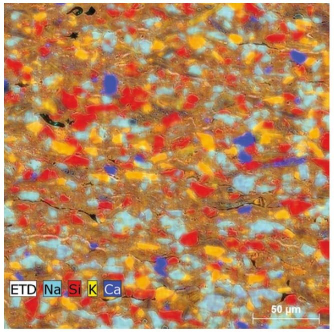


Texture Variation & Grain-Size Distribution, Chang 7

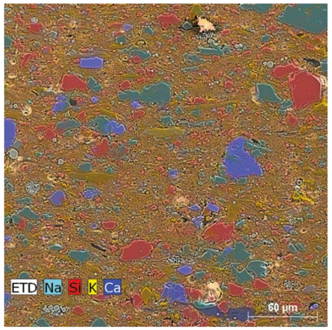
Texture variations in Chang 7 OM-rich mudstones



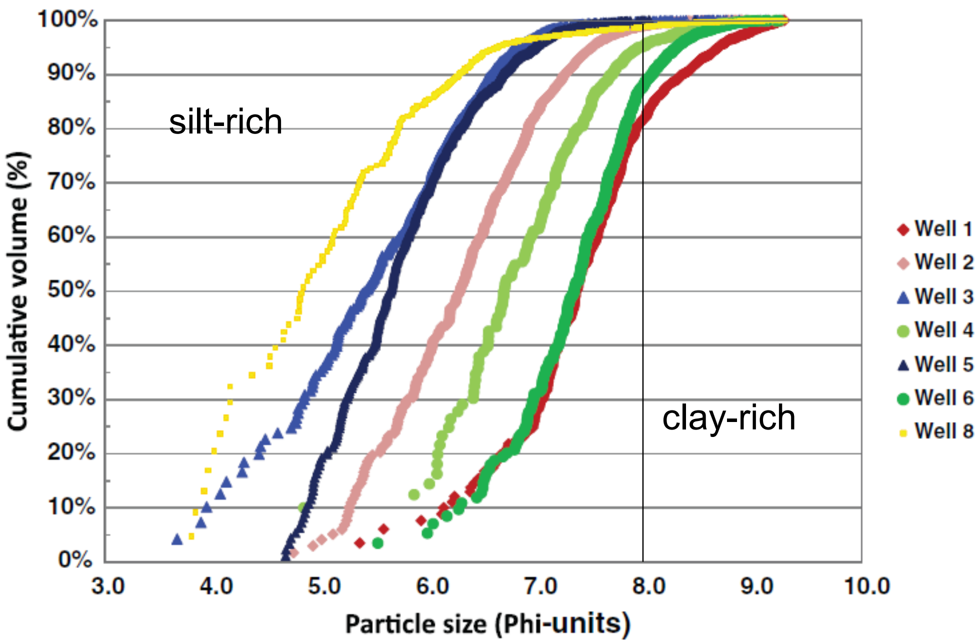
73.5% silt



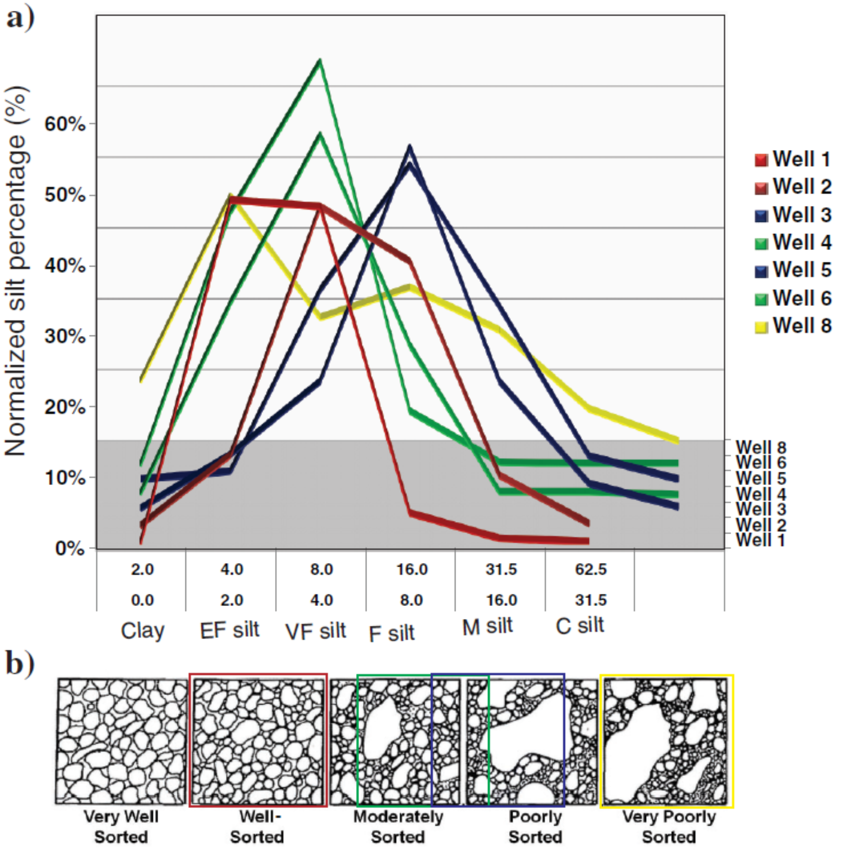
54.5% silt



38.4% silt

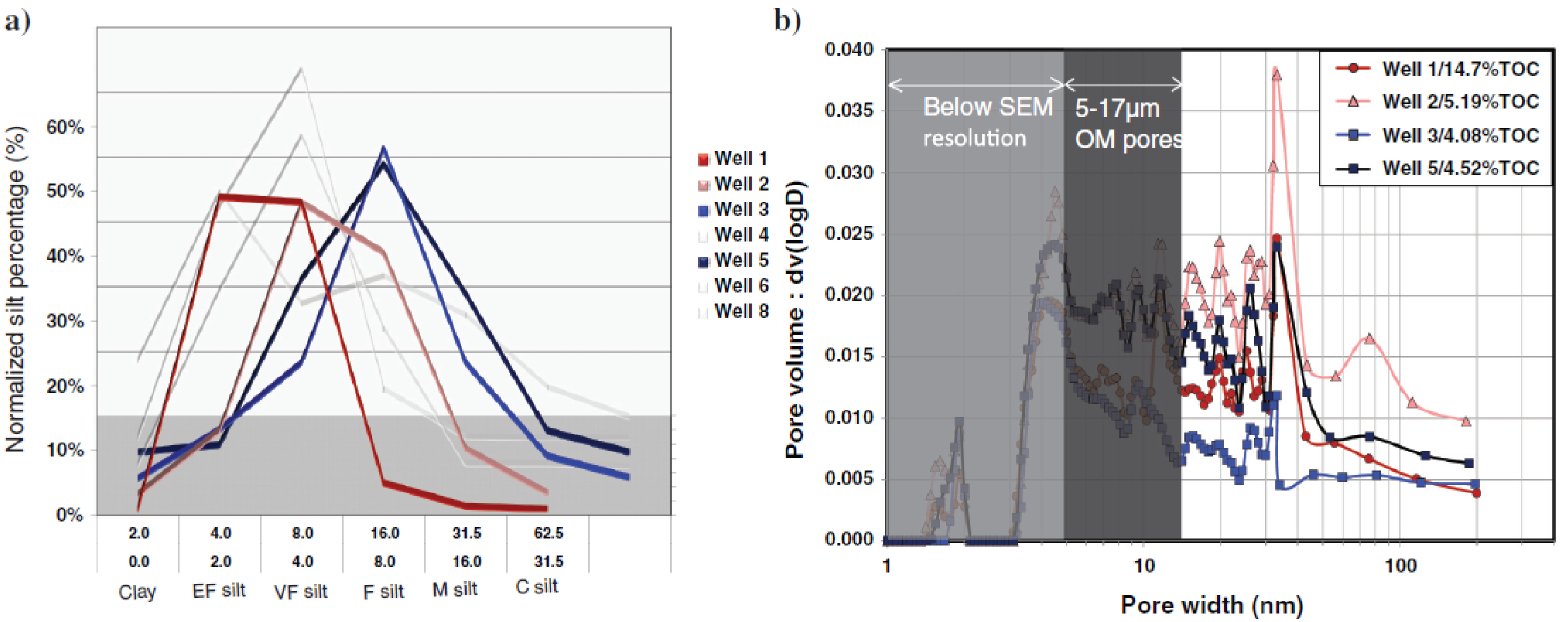


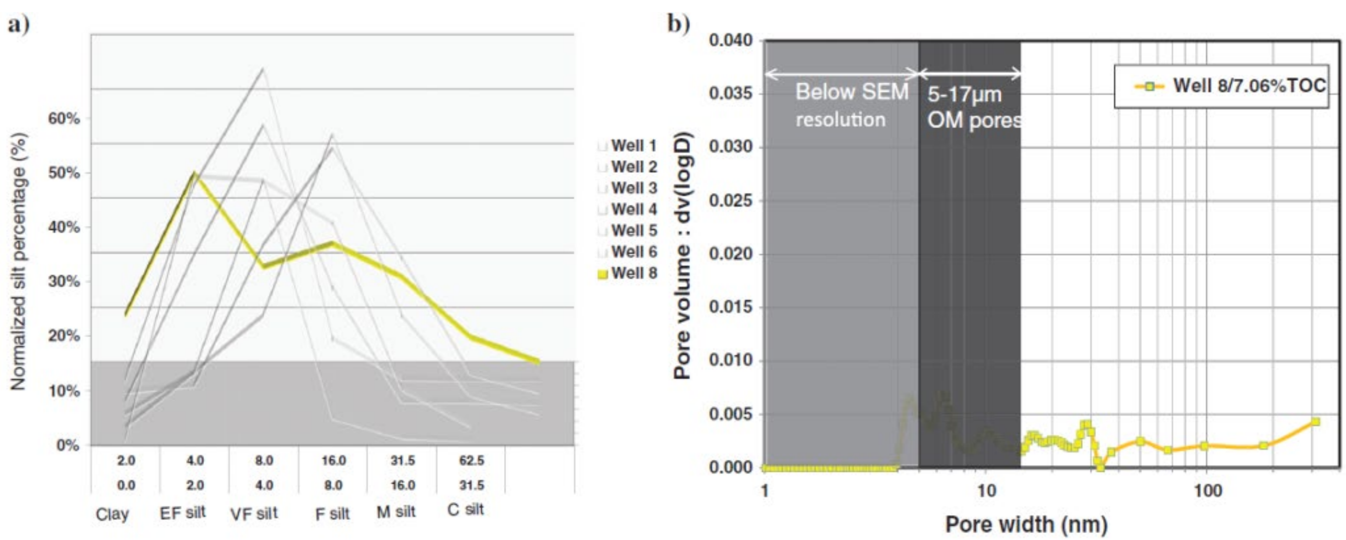
- Standard deviation implies sorting



(Milliken et al., 2017; Ko et al., 2017)

PSD Affected by Sorting and Grain Size



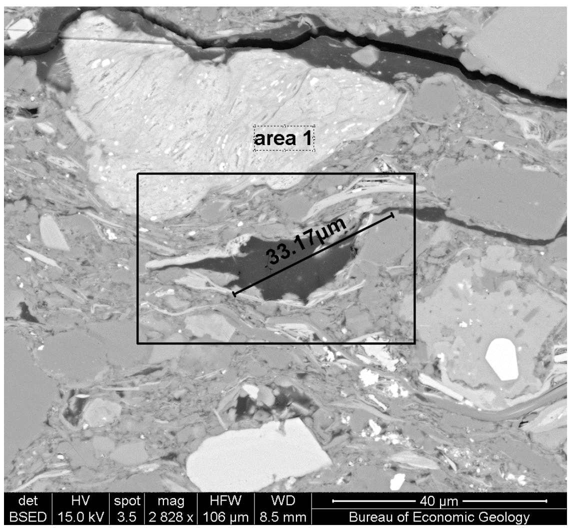


- A mudrock with well-sorted and coarser grains has higher mineral porosity.

(Ko et al., 2017)

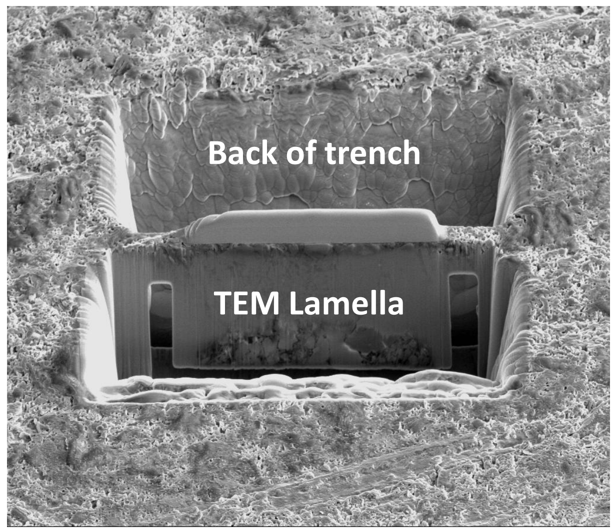
Focus Ion Beam (FIB) Area Preparation Process

SEM image of area of interest from Ar-ion polished sample



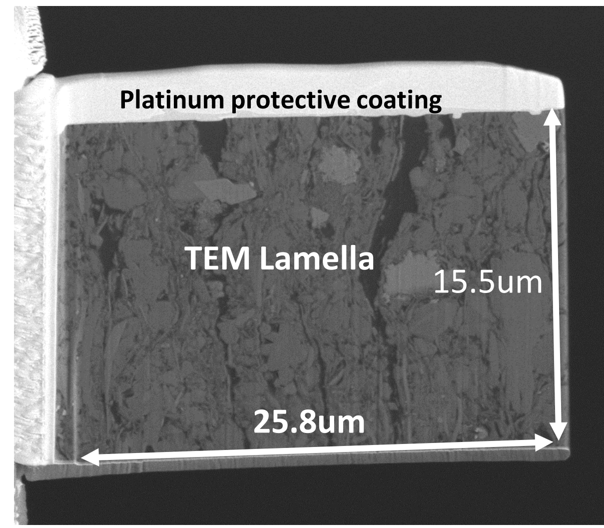
50 um

Gallium ion beam sputtered Trenched area with lamella



20 um

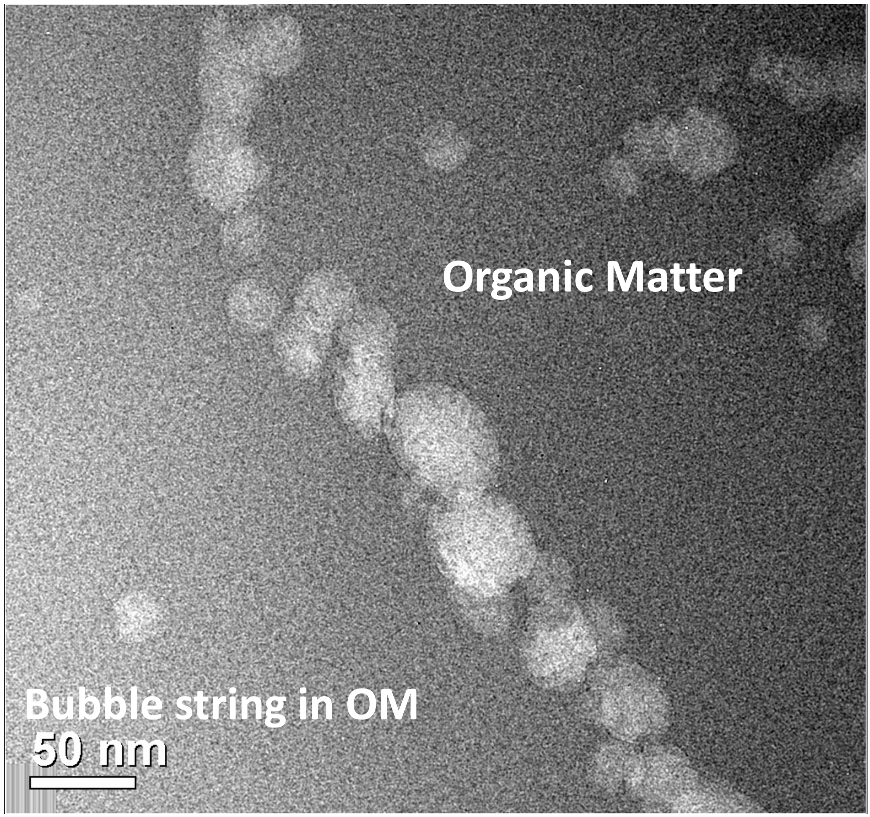
Final 15X25 um ˜ 100 nm thick lamella attached to TEM grid



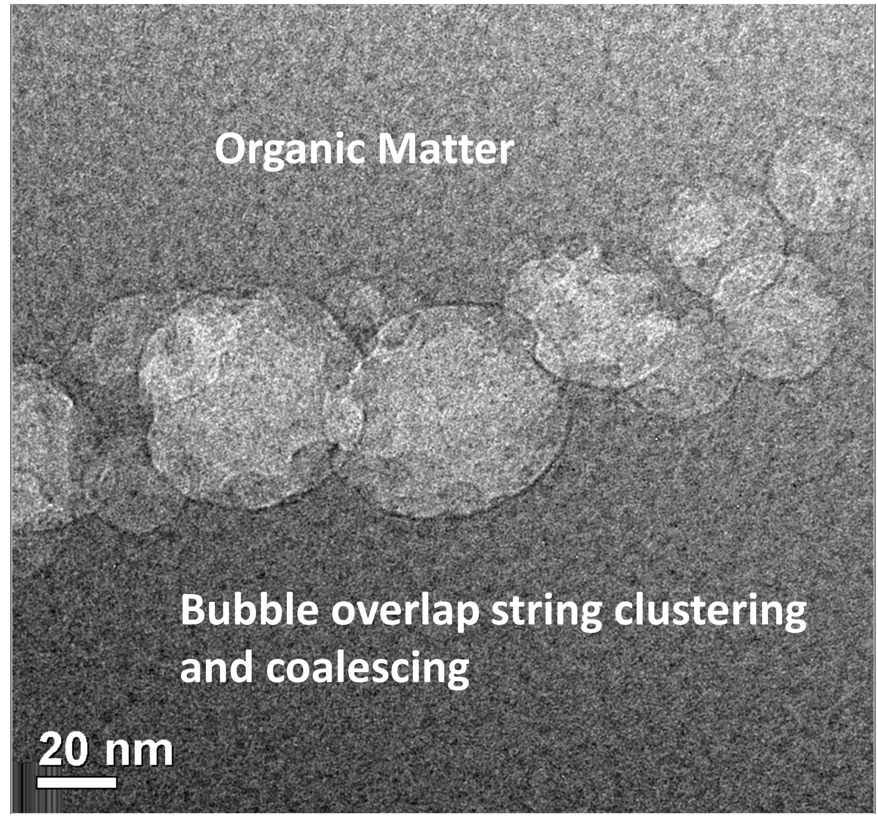
10 um

1567 TEM images

Light areas in Bubbles are lower density than surrounding organic matter (OM)

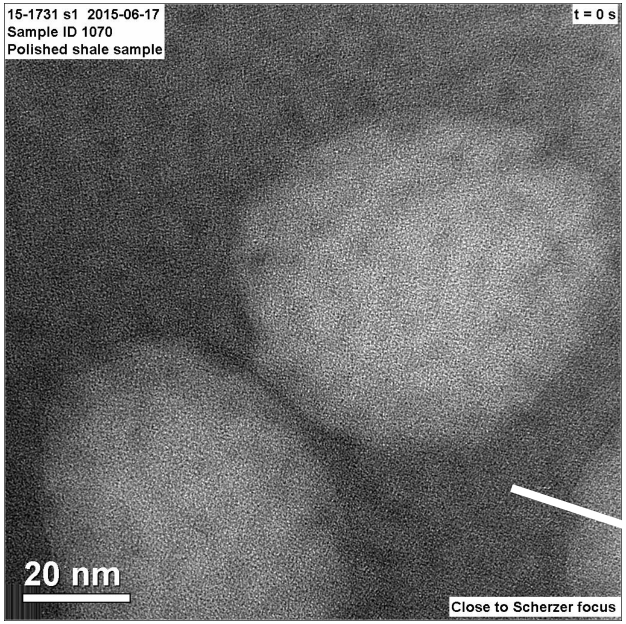


BEG



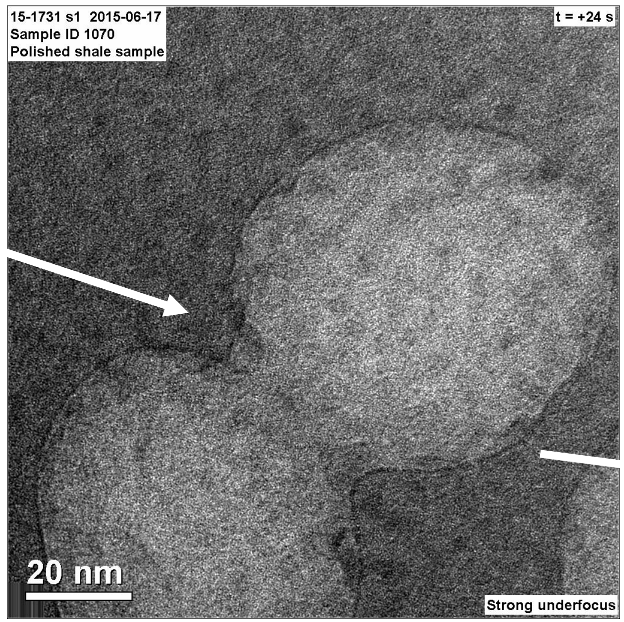
BEG

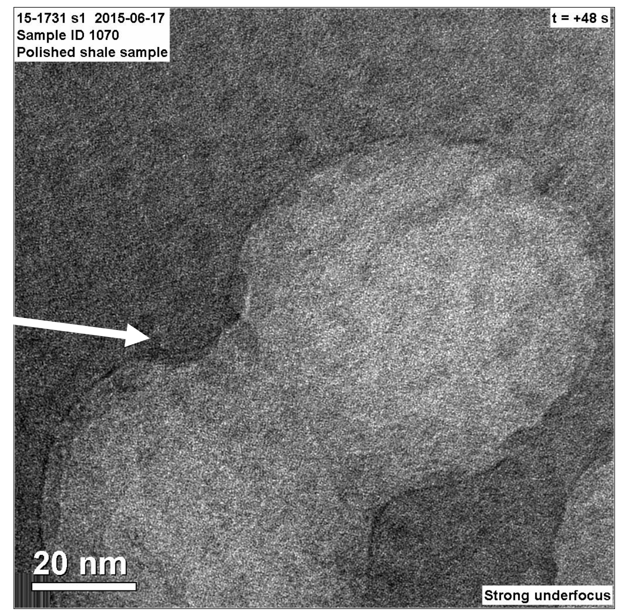
(Photo credit: Patrick Smith)



The expected outcome when you are not paying attention.

Bright field 300Kev TEM image in the organic matter of two bubbles coalescing caused by “beam heating” over ~ 5 minute period



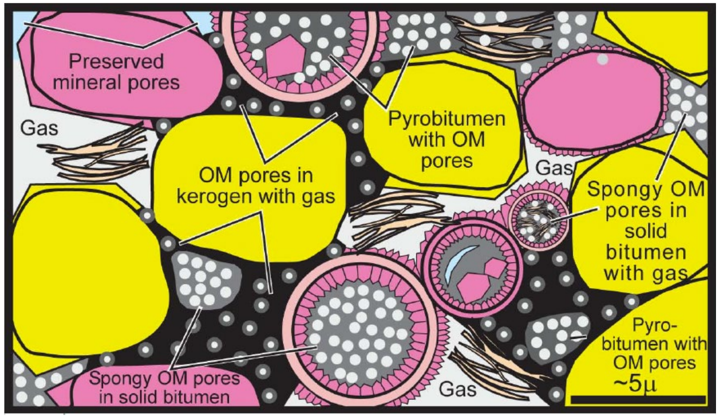


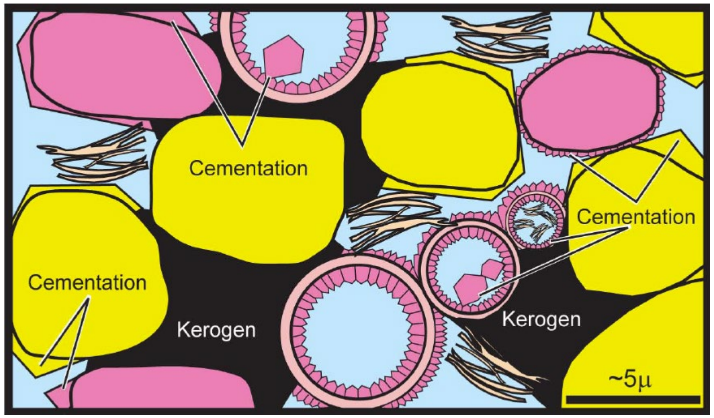
<u>Reference:</u>
“Bubble Breakup in porous Media”
Farzam Javadpour
Journal of Canadian Petrolum Technology

(Photo credit: Patrick Smith)

Workflow

- Identify the origins of mineral grains (detrital, biogenic, authigenic)
- Describe texture and fabric of the sample (depositional texture vs. diagenetic overprint)
- Quantitatively document pore types and pore systems
- Identify the distribution of OM in the system (kerogen vs. solid bitumen)
- Link grain assemblages, organic matter (OM), texture, fabric, and pore systems to lithofacies, kerogen and maceral type, thermal maturity, and depositional facies.





(Loucks and Reed, 2014)