SPE 144050

Submicron-Pore Characterization of Shale Gas Plays

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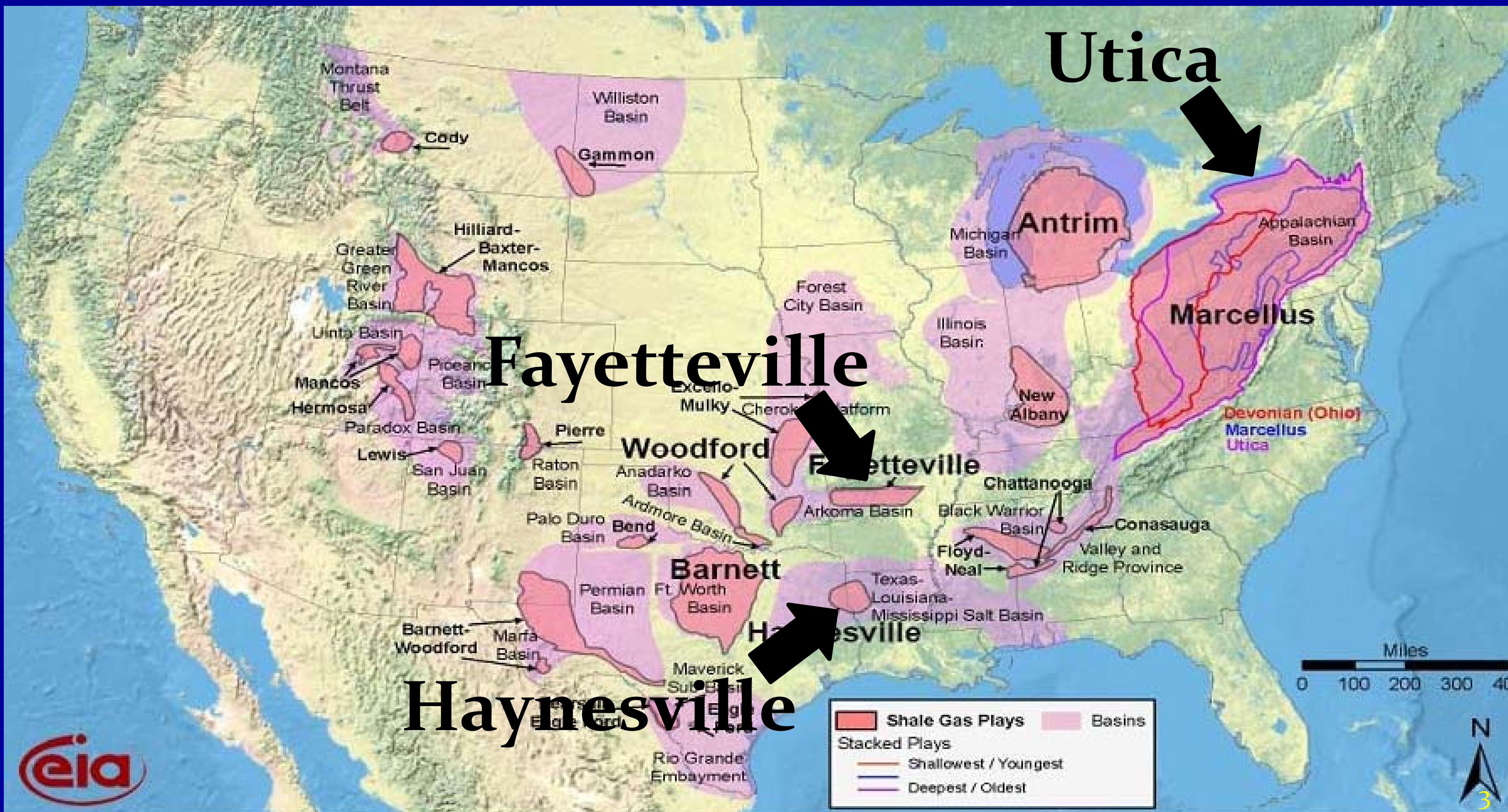
Talk Outlines

- US Natural Gas Shale Importance
- Research Scope & Workflow
- MICP Results
- Submicron Pore Imaging & Reconstruction
- XRD Clay Mineralogy Results
- Shale Gas Wettability Results
- Conclusions
- Acknowledgments

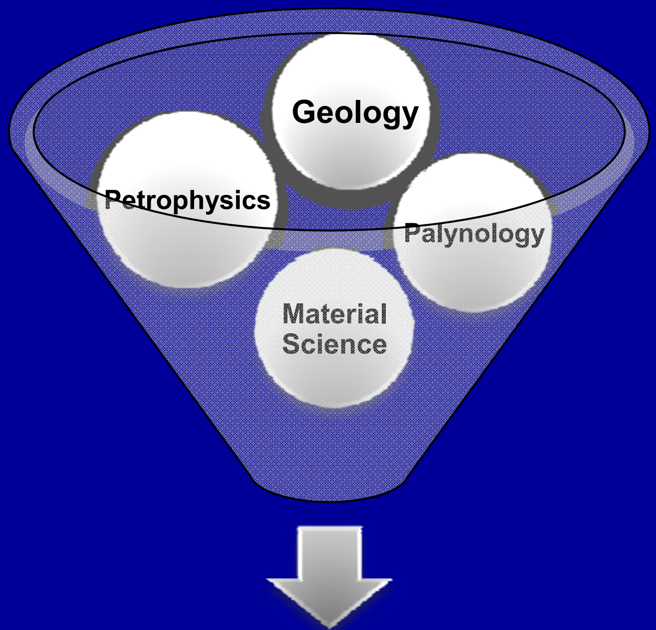
US Natural Gas Importance

87% of the supplied NG in US was produced domestically (EIA, 2009).
US possesses 2,552 Tcf of potential NG (EIA , 2011)
Shale gas est. reserve represent 32.4%

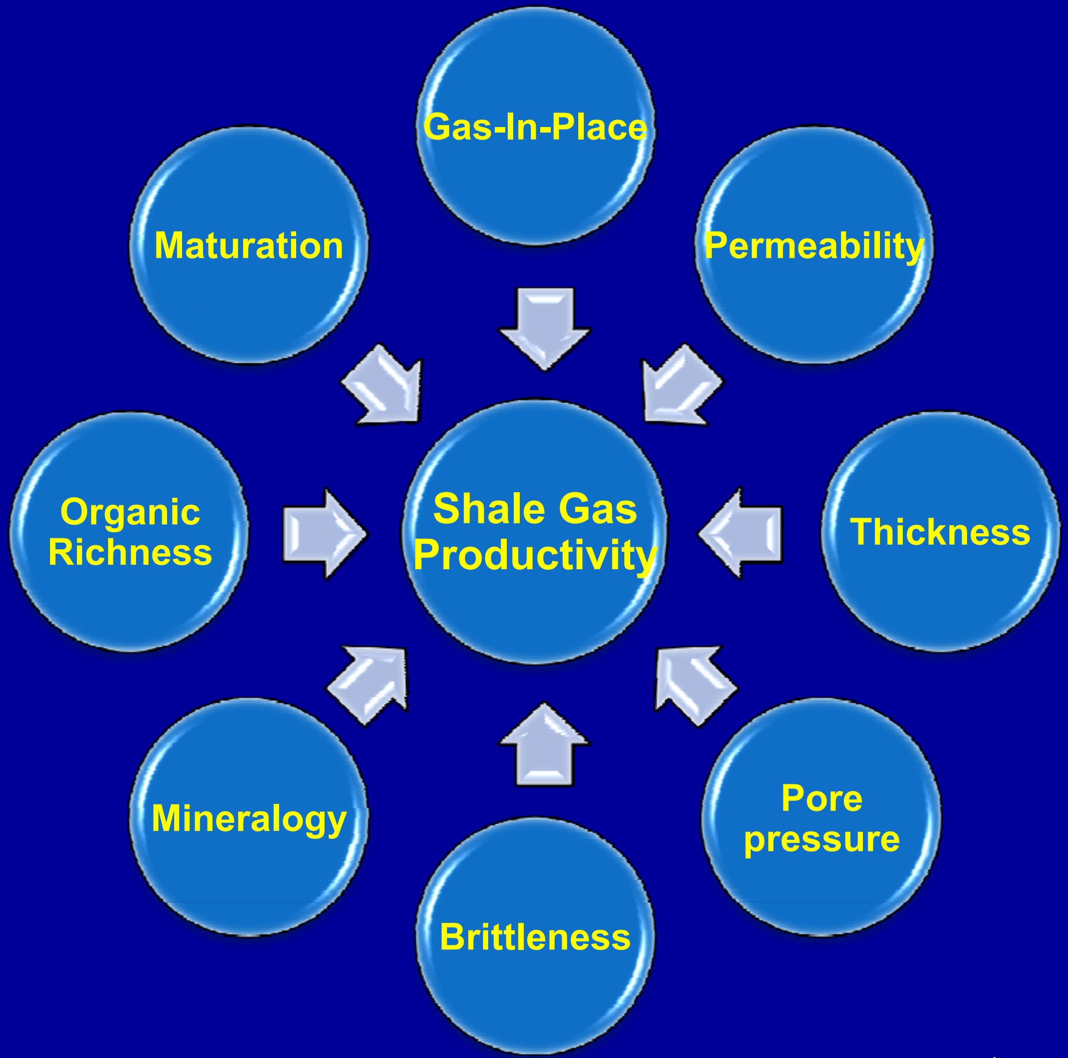


Essential Shale Gas Parameters

Demystifying Shale Gas Complexity by…

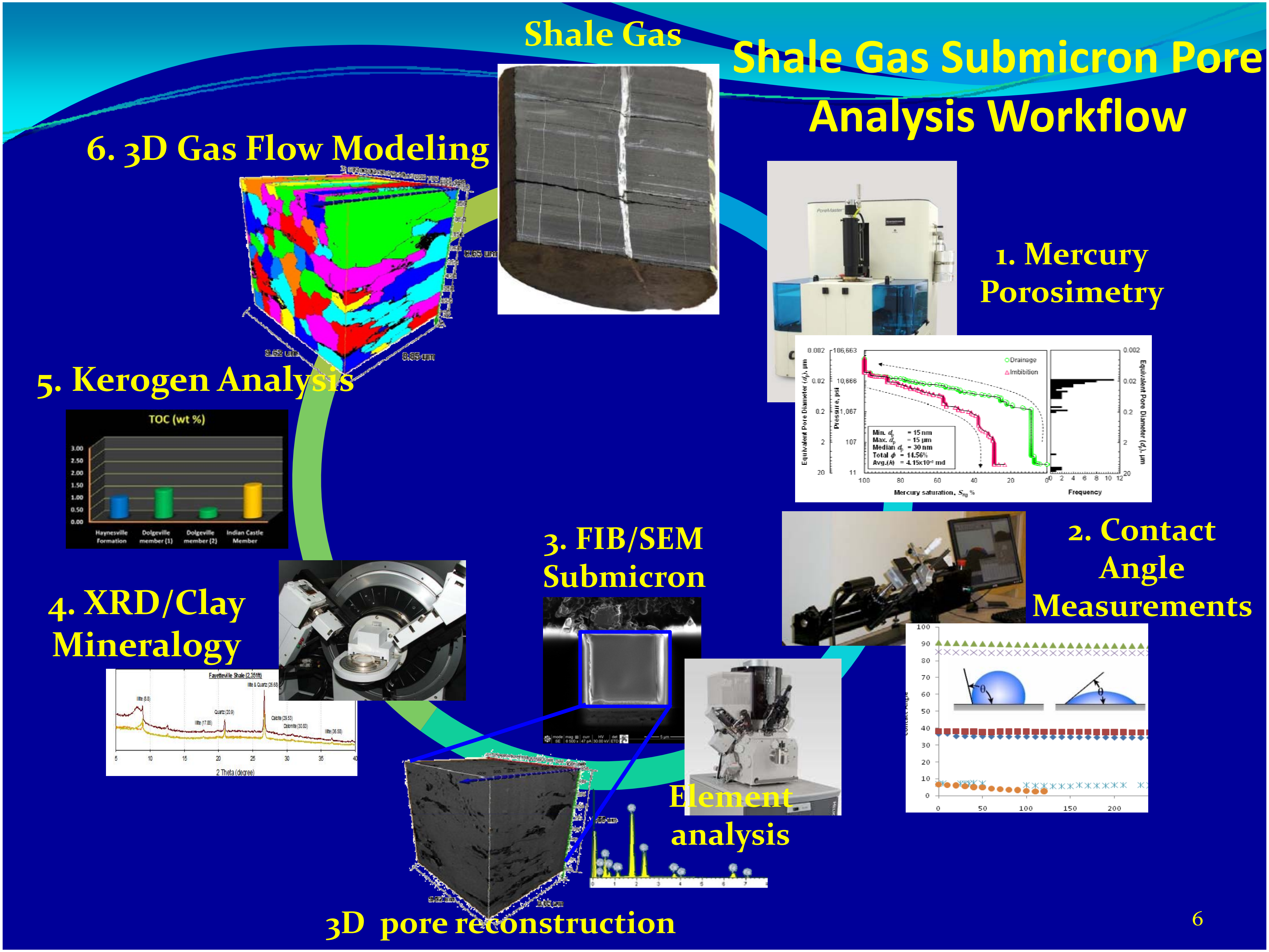


Shale Gas Characterization Interdisciplinary Tools



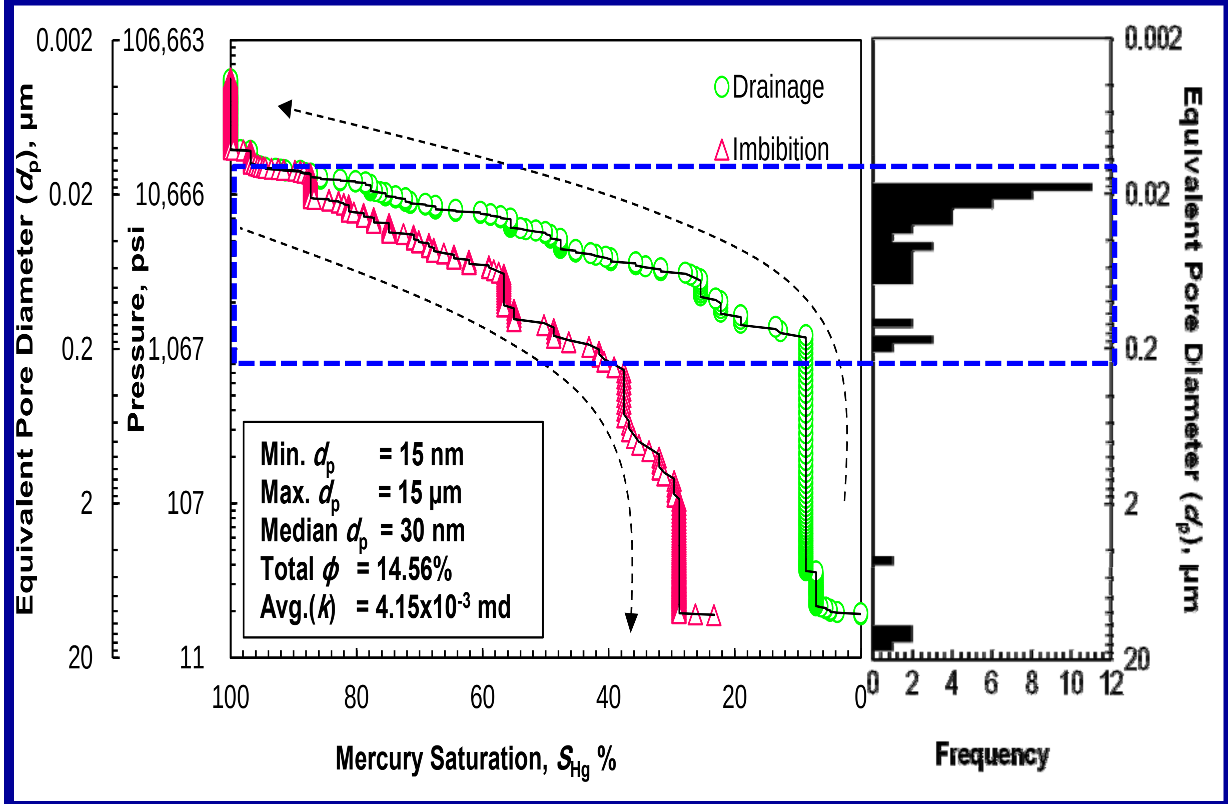
Research Objectives

- Look into the submicron pore structure of shale gas to extract the petrophysical properties.
- Determine the potential effects of organic matter, rock mineralogy on pore types and permeability.
- Identify shale gas porosity types with their magnitudes.
- Measure the impact of fracturing fluids on shale gas wettability.



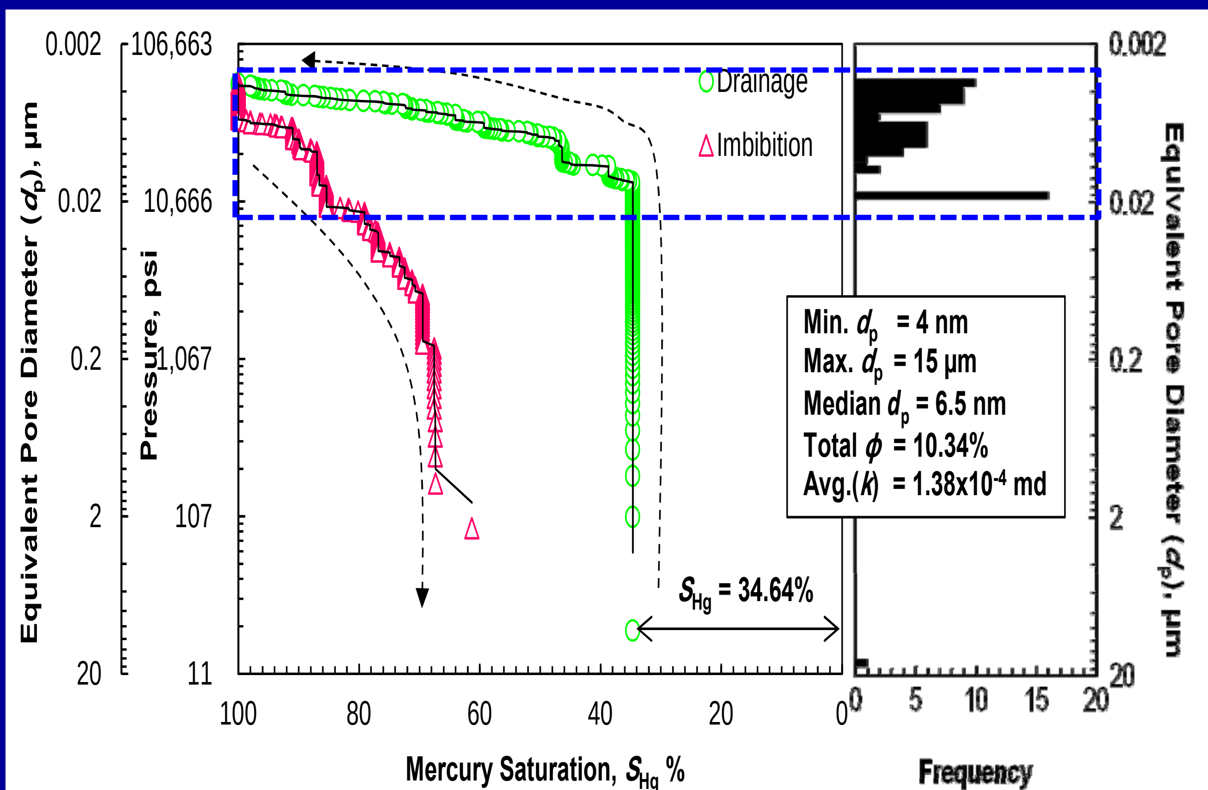
Shale Gas Submicron Pore Analysis Workflow
Shale Gas
1. Mercury Porosimetry
2. Contact Angle Measurements
3. FIB/SEM Submicron
Element analysis
3D pore reconstruction
4. XRD/Clay Mineralogy
5. Kerogen Analysis
6. 3D Gas Flow Modeling
TOC (wt %)
Haynesville Formation
Dolgeville member (1)
Dolgeville member (2)
Indian Castle Member
Drainage
Imbibition
Mercury saturation, S_{Hg} %
Frequency
Pressure, psi
Equivalent Pore Diameter (d_p), μm
2 Theta (degree)

MICP Results-Utica Shale



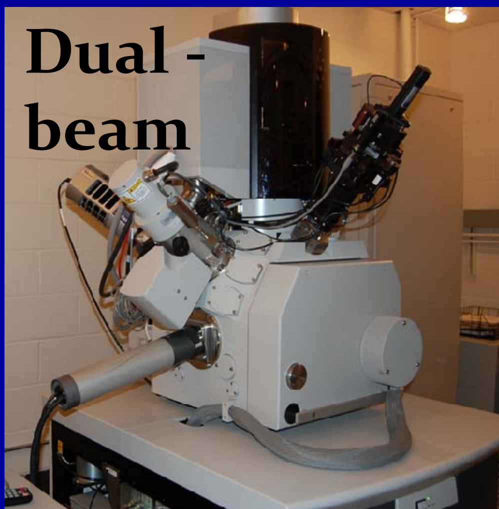
- Various pore sizes and/or different flow units.
- Major intrusion between 2,000 and 20,000 psi
- Eq. d_p= 20–200 nm
- Median d_p= 30 nm
- ϕ = 14.5%
- k =4.15 micro-darcy
- Resi. Hg is 23.3%

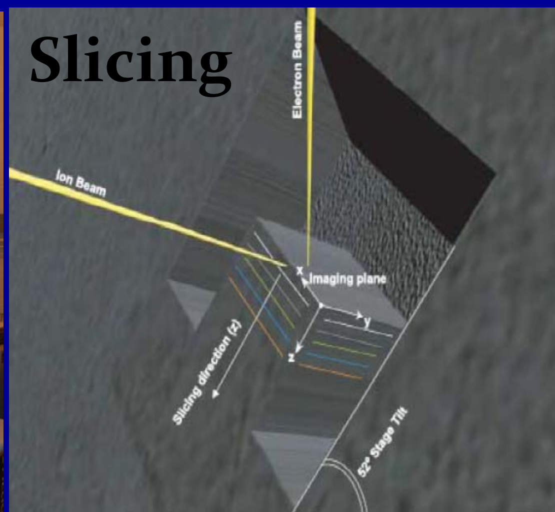
MICP Results-Haynesville Shale

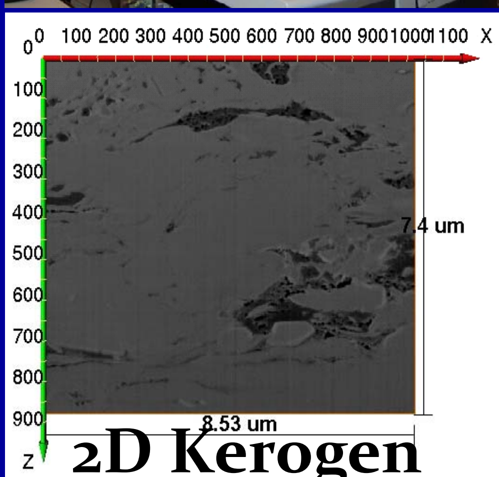


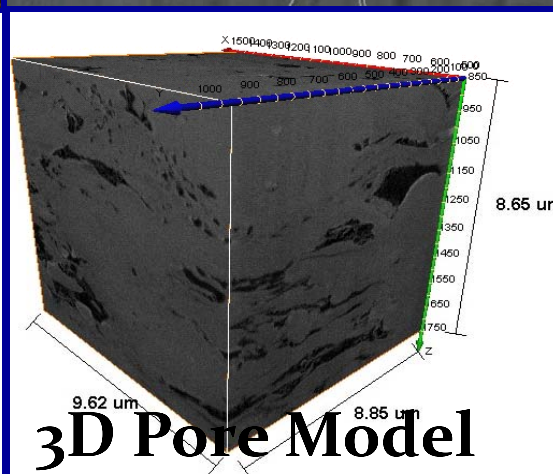
- One morphology is present.
- Major intrusion between 10,600 and 60,000 psi
- Eq. d_p= 2–20 nm
- Median d_p= 6.5 nm
- ϕ = 10 %
- k=138 nano-darcy
- Resi. Hg = 26.66%

SEM-FIB Tomography: 3D Submicron Pore-Scale Reconstruction



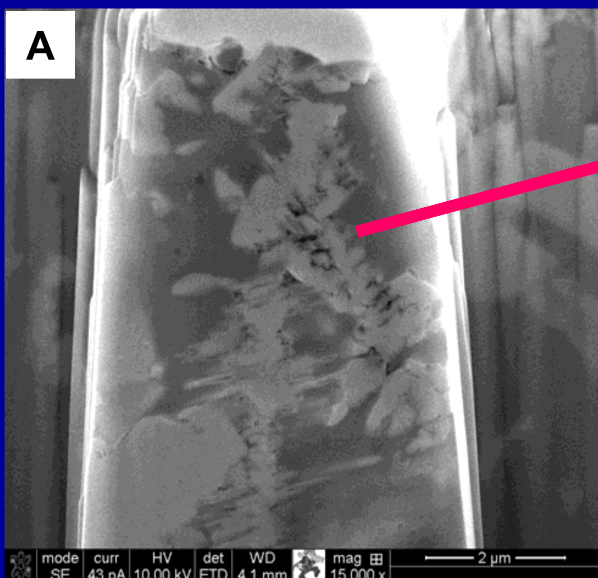




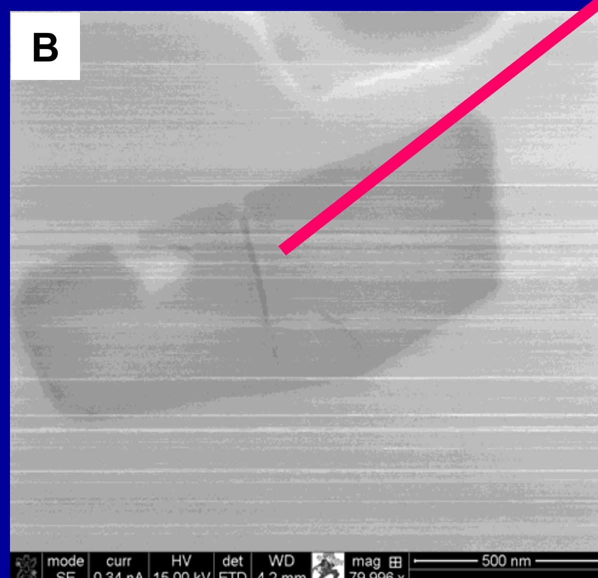


- Sample preparation
- In-situ specimen preparation
- Acquire 200 slices of 50nm thick
- Imaging processing programs.
- 2D Kerogen model
- 3D Structured pore model

SEM Images of Utica Shale Sample No. 3

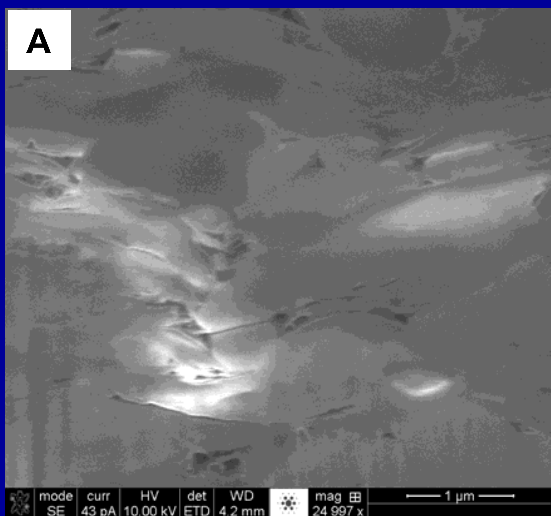


Intergranular pore sizes ranged from 15 to 50 nm.

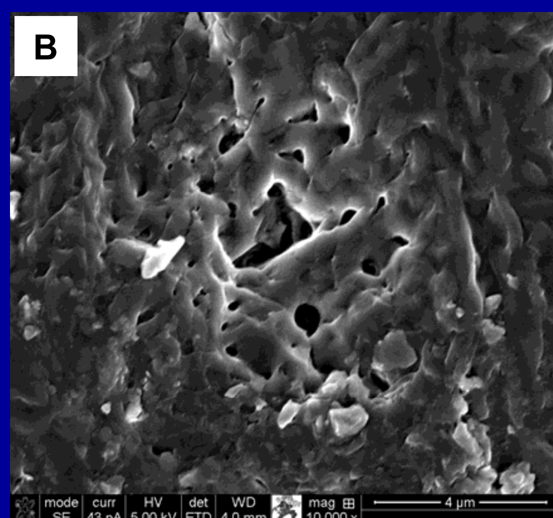


Intraparticular or mineral porosity with opening throat is about 5 nm.

SEM Images of Utica Shale Sample No. 2

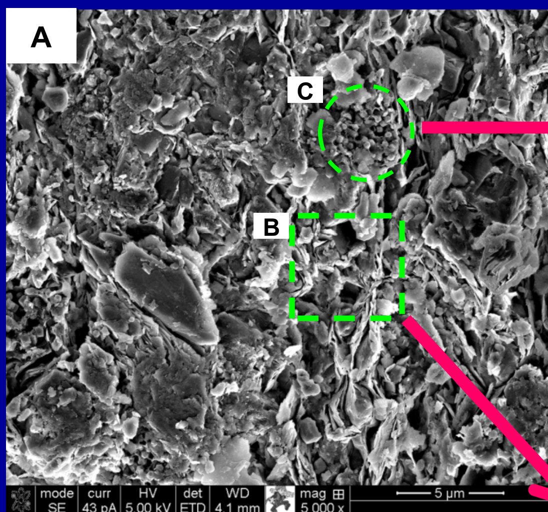


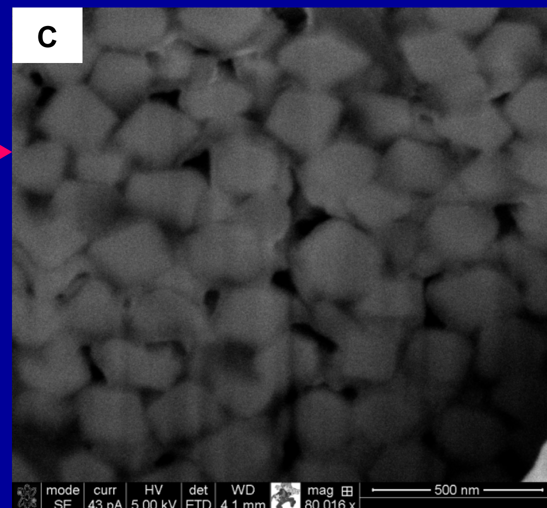
Clay platelets within the quartz grains which create inextricable pore structure with a variety of pore sizes due to diagenesis.



- Organic matter with a complex textile of pores.
- Morphology and genesis of OM control the permeability pathways of gas flow .

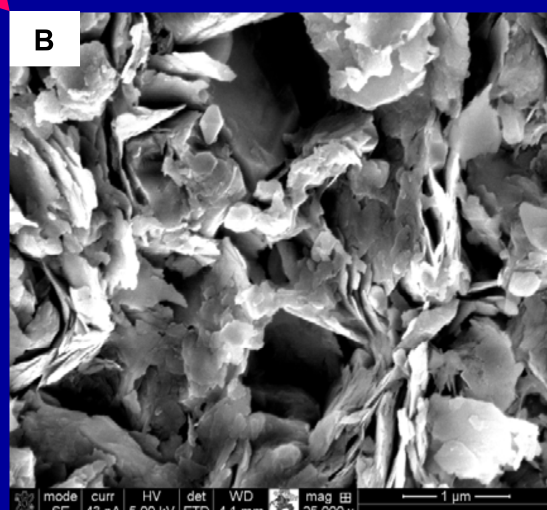
SE/BSE Images of Utica Shale Sample No. 2





- Nano-porosity is observed within pyrite framboids.

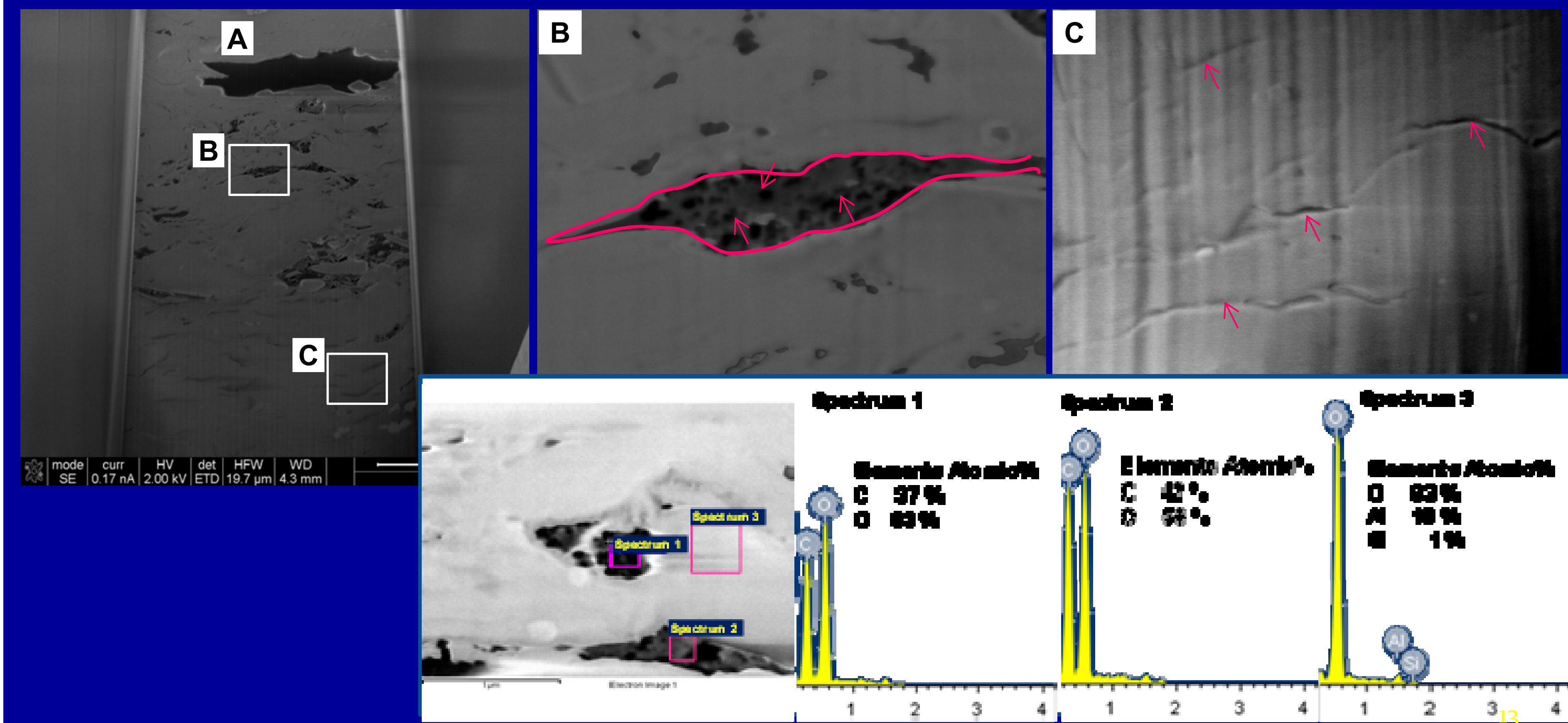
- Pore sizes of 20 and 100 nm.



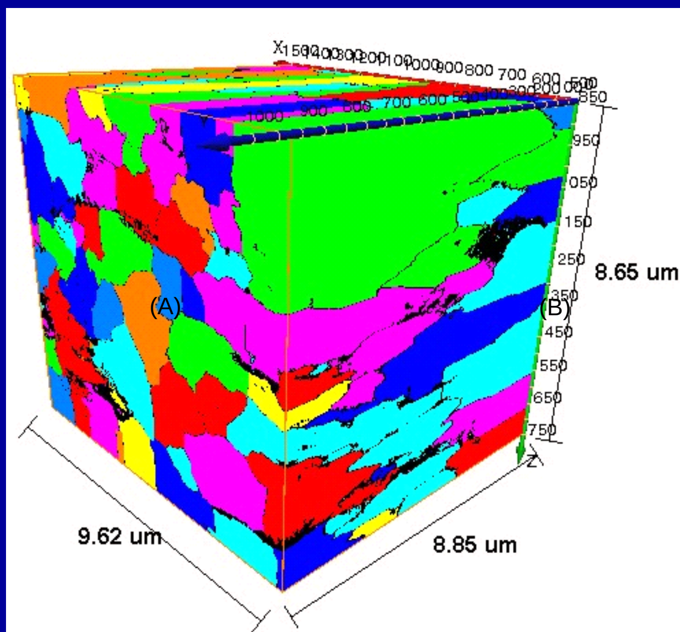
Clay platelets are closely packed together and form a variety of micron and nano pores (< 2 µm in diameter)

SEM Images of Fayetteville Shale Sample

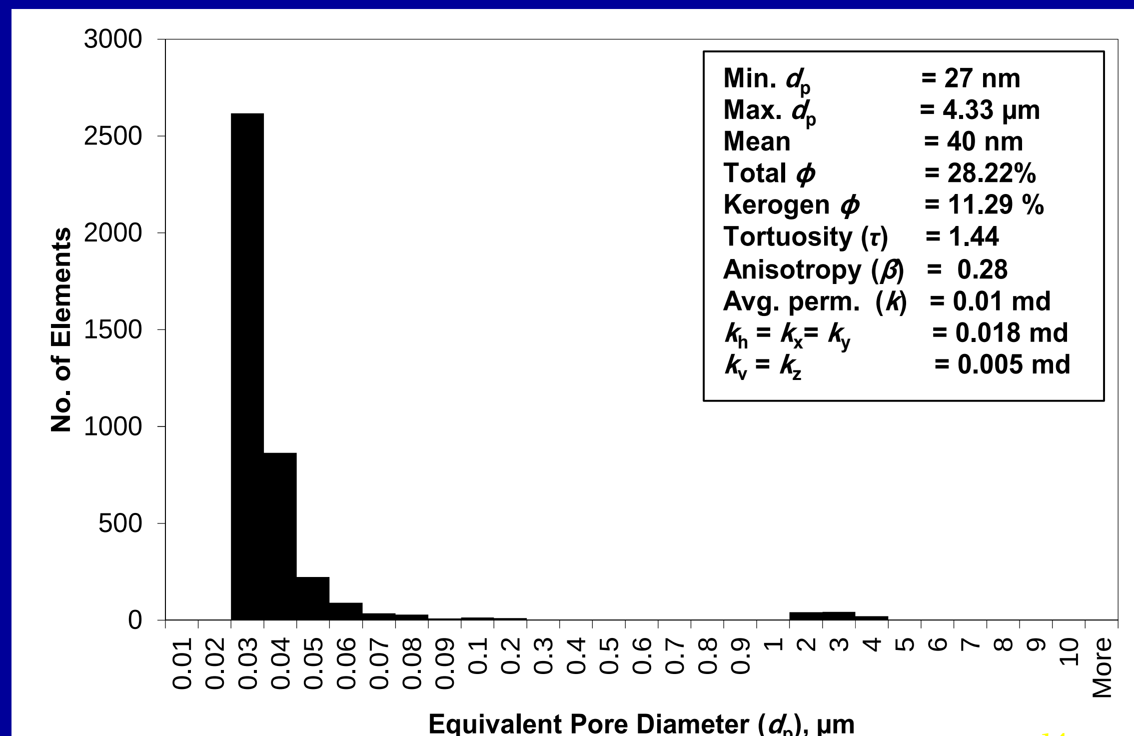
(A) Vuggy Micro-porosity, (B) Kerogen Nano-porosity Occupies about 40–50% of the Organic Matter, and (C) Nano Fractures



3D Volume Pore Size Histogram

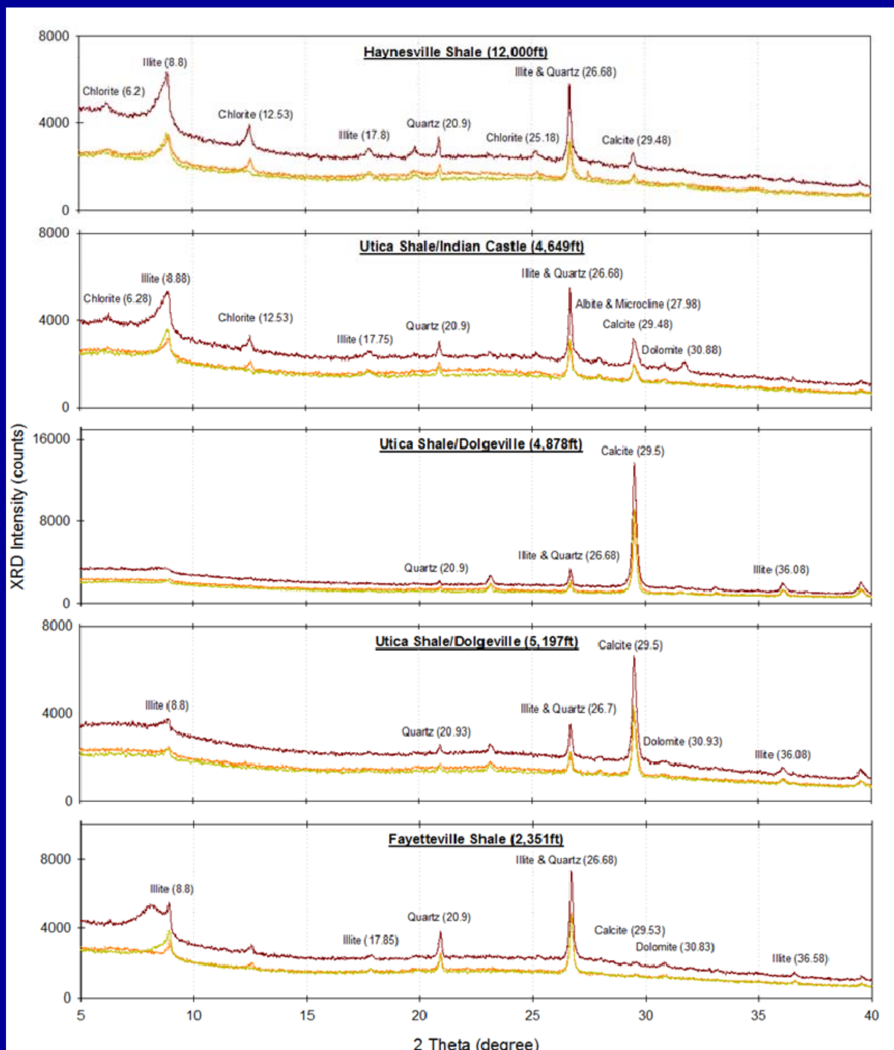


- Major pore size is 30nm
- Few micron-cracks or vugs of 3 μm.
- ϕ_{Total} = 28%, $k_{av.}$ = 0.01 md



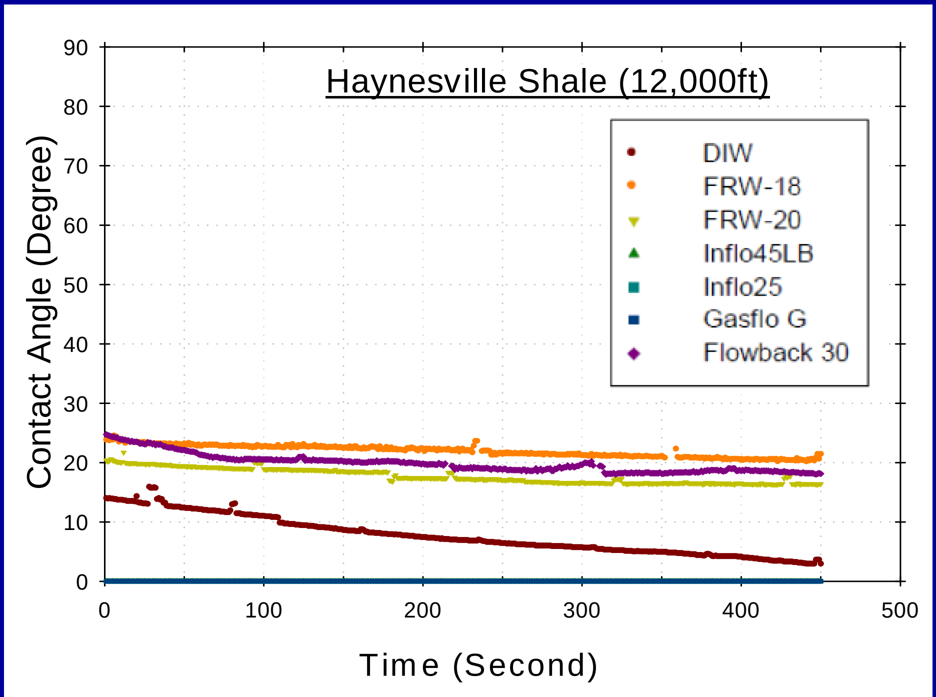
- ϕ_{OM} = 11.3%
- 1.44 tortuosity
- 0.28 anisotropy

XRD Clay Mineralogy Results



- Semi-quantitative clay minerals analysis
- Haynesville & Utica-Indian Castle Fm. have illite%
- Utica-Dolgeville Fm. exhibits high calcite%
- Fayetteville compositionally comprise of high quartz%

Shale Gas Wettability

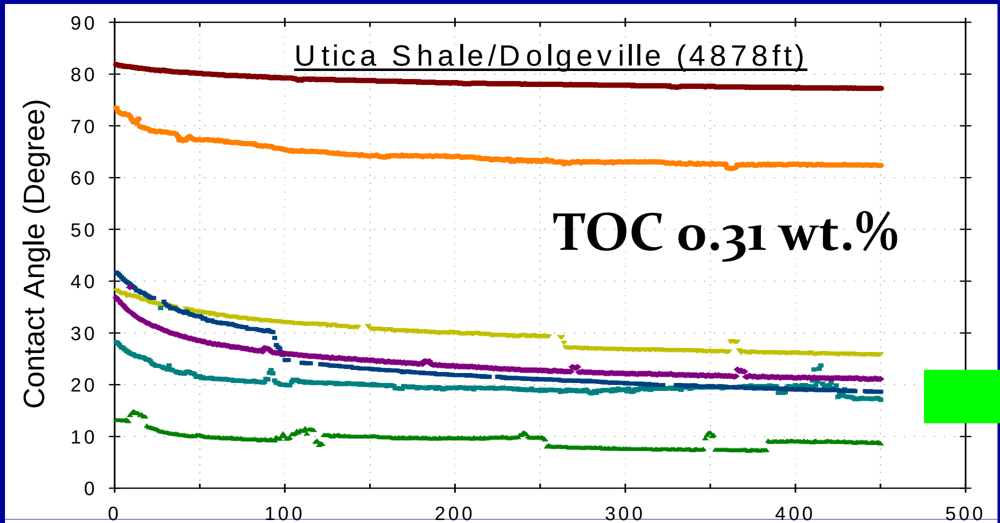


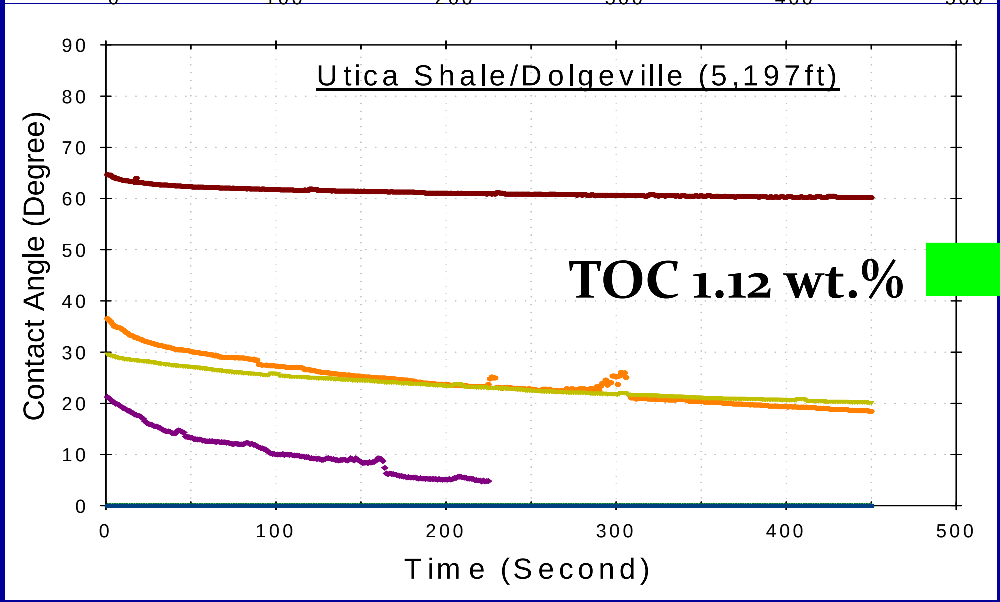
70% illite content
Low TOC 0.81 wt.%

- Six fracturing fluids
  - 2 Polymers
  - 4 Surfactants
- 0.1% concentrations
- Polished surfaces
- For 7.5 minutes
- Results compared to DIW

- Haynesville becomes hydrophobic with polymers and Flow-back surfactant.
- Surfactants makes all shales hydrophilic (strongly water-wet)

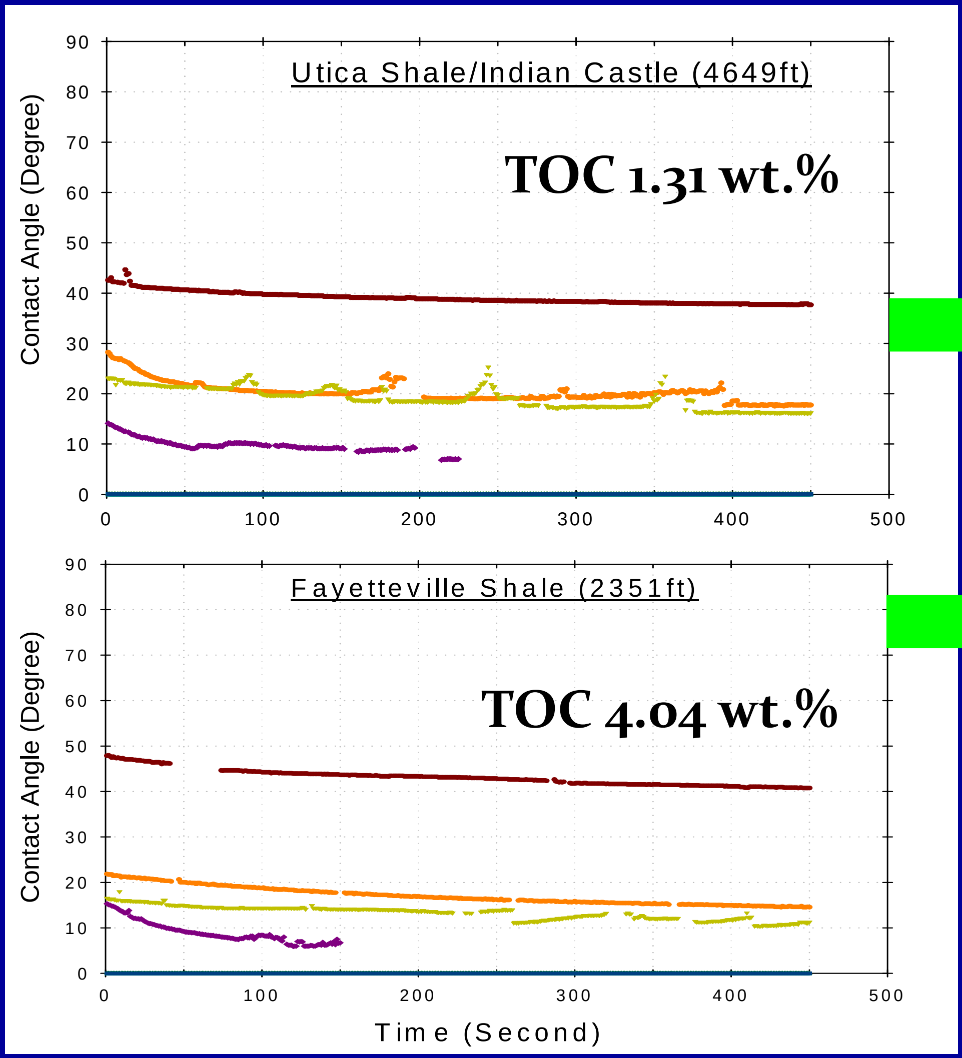
Utica Shale-Dolgeville Fm.





- High contact angle near oil-wet (60-80°)
- High calcite content %
- FRW-18 polymer slightly impairs surface wettability toward water-wet .
- FRW-18 & 20 resulted in similar impairment to intermediate water-wet

Utica-Indian Castle Fm./Fayetteville Shale



- Both shales' have similar wettability trends.
- High illite content%
- Low TOC content%

- High quartz or possibly illite
- High TOC content%

Conclusions

- Haynesville shale exhibited extremely low matrix permeability with a pore size of 2–20 nm, ϕ=10%.
- Utica shale offered better matrix permeability, with a pore throat size of 20–200 nm, ϕ=14.5%.
- Fayetteville shale resulted in the highest k=0.01 md and ϕ=28%.
- The organic matter of Fayetteville shale has abundance of nanopores with size of 5–100 nm and occupy 40-50% of the kerogen body.

Conclusions (cont.)

- A robust, detailed sequential milling and imaging procedure using SEM-FIB proved successful.
- Various types of porosities (e.g., interparticular, intergranular, kerogen, vuggy, pyrite framboids, and fractures) were observed.
- Numerous petrophysical properties were extracted from reconstructed 3D submicron pore model.
- Calcite content proved the dominant factor changing the wettability of shale gas rock to oil-wet.
- Organic matter content governed the wettability alterations of calcareous samples.

Acknowledgments

- Dr. Timothy Kneafsey (BNL)
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- Southwestern Energy
- Baker-Hughes Co.
- RPSEA & DOE

Thank You

Questions?

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