Submicron-Pore Characterization of Shale Gas Plays

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SPE Americas Unconventional Gas Conference and Exhibition
12–16 June 2011
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
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

- Petrophysics
- Geology
- Palynology
- Material Science

Shale Gas Productivity

- Gas-In-Place
- Permeability
- Maturation
- Organic Richness
- Mineralogy
- Brittleness
- Pore pressure
- Thickness
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

1. Mercury Porosimetry
2. Contact Angle Measurements
3. FIB/SEM Submicron Imaging
4. XRD/Clay Mineralogy
5. Kerogen Analysis
6. 3D Gas Flow Modeling

Element analysis
3D pore reconstruction
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
- $\phi = 14.5\%$
- $k = 4.15$ 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. \( d_p = 2\text{–}20 \text{ nm} \)
- Median \( d_p = 6.5 \text{ nm} \)
- \( \phi = 10\% \)
- \( k = 138 \text{ nano-darcy} \)
- 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 framboisds.

- 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.
- $\phi_{\text{Total}} = 28\%$, $k_{\text{av.}} = 0.01$ md

- $\phi_{\text{OM}} = 11.3\%$
- 1.44 tortuosity
- 0.28 anisotropy

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Min. $d_p = 27$ nm
Max. $d_p = 4.33$ µm
Mean = 40 nm
Total $\phi = 28.22\%$
Kerogen $\phi = 11.29\%$
Tortuosity ($\tau$) = 1.44
Anisotropy ($\beta$) = 0.28
Avg. perm. ($k$) = 0.01 md
$k_x = k_y = k_z = 0.018$ md
$k_x = k_z = 0.005$ md
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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

- 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)

70% illite content
Low TOC 0.81 wt.%
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, $\phi=10\%$.
- Utica shale offered better matrix permeability, with a pore throat size of 20–200 nm, $\phi=14.5\%$.
- Fayetteville shale resulted in the highest $k=0.01$ md and $\phi=28\%$.
- 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 frambooids, 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)
- Materials Research Center (Missouri S&T)
- Southwestern Energy
- Baker-Hughes Co.
- RPSEA & DOE
Thank You
Questions?
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