Nano-scale Pore Structure of Middle Devonian Organic-Rich Black Shale and its Evolution through Thermal Maturation

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Abstract

The pore structures and the evolution of porosity were analyzed in samples from three wells that penetrated the Marcellus Shale in Appalachian basin. The thermal maturity ranges from Ro (vitrinite reflectance) 1.36% to Ro 2.89%. Total organic carbon (TOC) of Mahantango Formation to Marcellus Shale samples from West Virginia and Pennsylvania used in this study varies from 0.25 to 9.12 wt.%. Subcritical N2 adsorption and Scanning Electron Microscope (SEM) techniques were utilized to test nano-scale pore structures (pore sizes, pore volumes, and pore-size distributions) qualitatively and quantitatively. Also, X-ray fluorescence (XRF) and X-ray diffraction (XRD) analysis were conducted to study the heterogeneity of mineral composition and its influence on the pore structures. Before running N2 adsorption, shale samples were crushed, then degassed under high vacuum with heating. In order to find an appropriate procedure for sample preparation, one set of rock sample was crushed and hand-grounded, then degassed under five different temperatures (25°C, 80°C, 120°C, 200°C, 300°C). Based on the results, size and more critically temperature for degassing are important factors. A size of <250 microns (60 mesh) degassed under 120°C for 24 hours is the recommended procedure. After the measurements, BET, t-Plot, H-K, and BJH models were used to interpret the results. The results, combined with SEM image analysis indicate that, as thermal maturity increases, specific surface areas (SSA), pore volumes, and pore-size distributions varies significantly. Among all the factors we tested, thermal maturity appears to be the major control on evolution of pore structures of organic-rich mudrocks.

References Cited


**Geological Background and Introduction**

**Pore Micro Texture**

- Digitized SEM Analysis
- Porosity
- Organic Matter

**Specific Surface Area and Pore Volume**

- Pore size distribution, and its contribution to pore volume and surface area

**Pore Structure and Storage Capacity**

**Observations**

- Specific surface area and pore volume of higher thermal maturity samples are less than that of lower thermal maturity samples.
- Pores of diameter less than 5 nm make the greatest contribution to SSA, whereas pore volumes are affected by larger pores. Samples with higher thermal maturity have less smaller pores (less diameter less than 5 nm).

**Pore Size Distribution**

Samples with higher thermal maturity (well CSI) have less smaller pores

No difference of vertical scale

**Conclusions**

1. Four pore micro texture facies are picked, Organic-rich, Organic-lean, Clay-rich, Carbonate-rich.
2. SEM analysis can be correlated to bulk petrophysical properties of cores.
3. Significant part of the pore system in mudrock is beyond SEM resolution, which is 10 nm in this research.
4. There is a general positive correlation between richness of organic matter and specific surface area and pore volume.
5. Organic matter makes the majority of the micropores and mesopores space.
6. As thermal maturity increasing from dry-gas zone to post-mature zone, pore volume and surface area decreases.

**Reference**


