

# **PS Multiple Approaches to Pore Structure Characterization of Bakken Petroleum System \***

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## **Abstract**

Ultra- and small-angle neutron scattering (USANS/SANS) techniques have been increasingly utilized to study pore structures of shale. The total porosity and pore-size distribution obtained from fitting a polydisperse spherical pore (PDSP) model to combined (U)SANS scattering profiles are evaluated against analyses from mercury injection capillary pressure (MICP) analysis and low-pressure nitrogen adsorption isotherms. This study focuses on shale samples from a self-sourced shale reservoir - the Bakken Shale. Differences in porosity and pore size distribution are observed between organic-rich source rocks and organic-lean reservoir rocks. Besides, a correlation between pore size distribution and mineralogy composition is investigated. Results show that the pore structure characteristics obtained from USANS/SANS, MICP and gas adsorptions are complementary and consistent; results obtained from different techniques with different principles, interpretation theories, and sample size are contrasted and discussed.

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# Multiple Approaches to Pore Structure Characterization of Bakken Petroleum System

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## Abstract

Ultra- and small-angle neutron scattering (USANS/SANS) techniques have been increasingly utilized to study pore structures of shale. The total porosity and pore-size distribution obtained from fitting a polydisperse spherical pore (PDSP) model to combined (U)SANS scattering profiles are evaluated against analyses from mercury injection capillary pressure (MICP) analysis and low-pressure nitrogen adsorption isotherms.

This study focuses on shale samples from a self-sourced shale reservoir - the Bakken Shale. Differences in porosity and pore size distribution are observed between organic-rich source rocks and organic-lean reservoir rocks. Besides, a correlation between pore size distribution and mineralogy composition is investigated.

Results show that the pore structure characteristics obtained from USANS/SANS, MICP and gas adsorptions are complementary and consistent; results obtained from different techniques with different principles, interpretation theories, and sample size are contrasted and discussed.

## Introduction

The Bakken Shale consists of three members: an upper shale member, a middle silty dolomite member and a lower shale member. Both the upper and lower shale members are organic-rich source rocks, which contribute hydrocarbons to the middle member; the whole Bakken Shale is considered a self-sourced petroleum system.

In order to study the effect of shale pore structure on the producibility of the Bakken Shale, we selected and obtained samples from a producing well (Kubas, 46.94N, -103.12W, API: 33-089-00586). In 2010, Kubas well was drilled horizontally and hydraulic fractured in the middle member of the Bakken Shale, but suffered sharp production decrease in the following years. This phenomenon is probably caused by both geometrical ( $\mu\text{m}$ -nm pore sizes) and topological (connectivity) aspects of pore structure of these shale members.

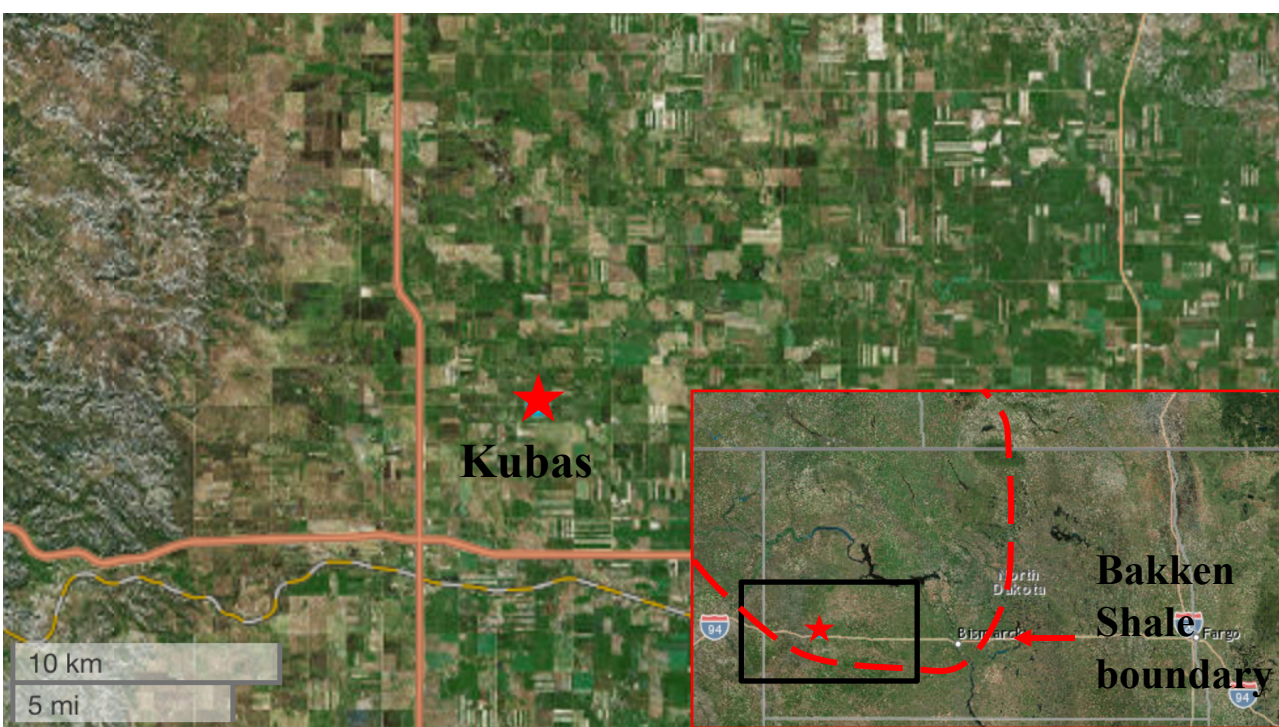


Figure 1. Location of Kubas well (46.94N, -103.12W) (DrillingInfo, 2017)

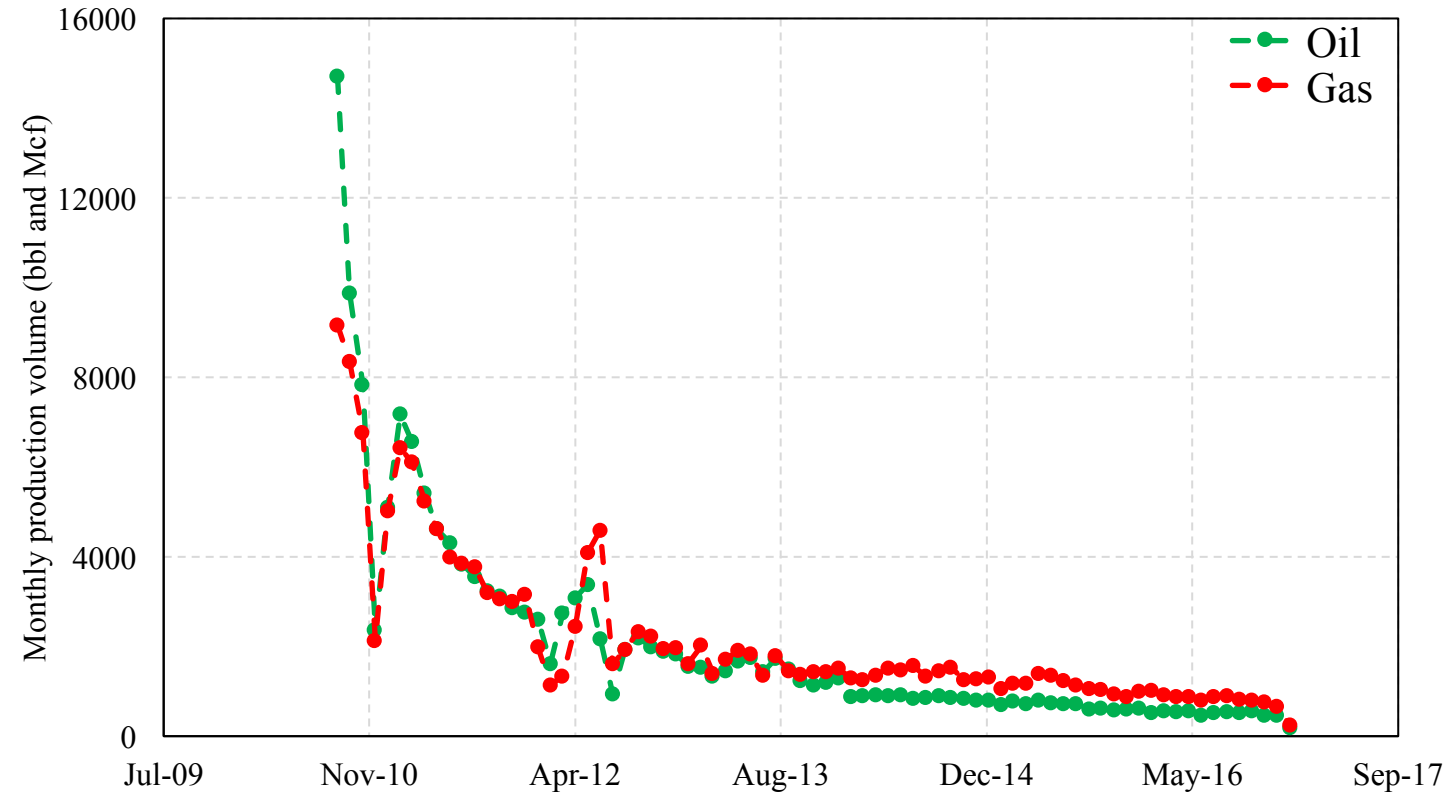


Figure 2. Production history of oil and gas in Kubas well (DrillingInfo, 2017)

## Materials and Methods

Table 1. Sample depth, mineral composition and raw material photos

Sample	Kubas U (upper) (10626 ft)	Kubas M (middle) (10636 ft)	Kubas M/L (lower) (10638 ft)
Mineral composition	QF (59.1%) Carb (9.8%) Clay (14.6%)	QF (34.6%) Carb (50.3%) Clay (13.8%)	QF (22.6%) Carb (64.7%) Clay (10.2%)
Organic Matter	TOC (9.8 wt.%)	TOC (0.6 wt.%)	TOC (1.4 wt.%)
Pictures			

Table 2. Description of method, sample size, and data interpretation

Methods	Sample size	Real sample	Model	Data Interpretation
MICP	Cube (1cm×1cm×1cm)			Washburn Equation: $D = -\frac{4\gamma \cos \theta}{P}$
N <sub>2</sub> adsorption	Fraction (500-850 $\mu\text{m}$ )			Barrett, Joyner, and Halenda (BJH) method: $Q(p) = \int dHq(p, H)f(H)$
(U)SANS	Thin-section (150 $\mu\text{m}$ thick)			PDSP Model: $I(Q) = (\rho_1 - \rho_2)^2 \frac{\phi}{V_v} \int_{R_{min}}^{R_{max}} V_r^2 f(r) F_{sph}(Qr) dr$

## Results 1

Table 3. Basic characteristics of Bakken Shale from multiple techniques

Sample	Bulk density (g/cm <sup>3</sup> ) <sup>1</sup>	Total pore area (m <sup>2</sup> /g) <sup>1</sup>	Median pore-throat diameter (nm) <sup>1</sup>	MICP Porosity (%) <sup>1</sup>	(U)SANS porosity (%) <sup>2</sup>
Kubas U (10626 ft)	2.44	3.41	8.4	1.62	3.03
Kubas M (10636 ft)	2.74	2.29	22.8	2.76	5.79
Kubas L (10638 ft)	2.70	2.12	6.2	0.98	3.88

<sup>1</sup> Measured by MICP;

<sup>2</sup> Measured in (U)SANS and fitted in PDSP model.

<sup>3</sup> Measured by low-pressure N<sub>2</sub> sorption isotherm.

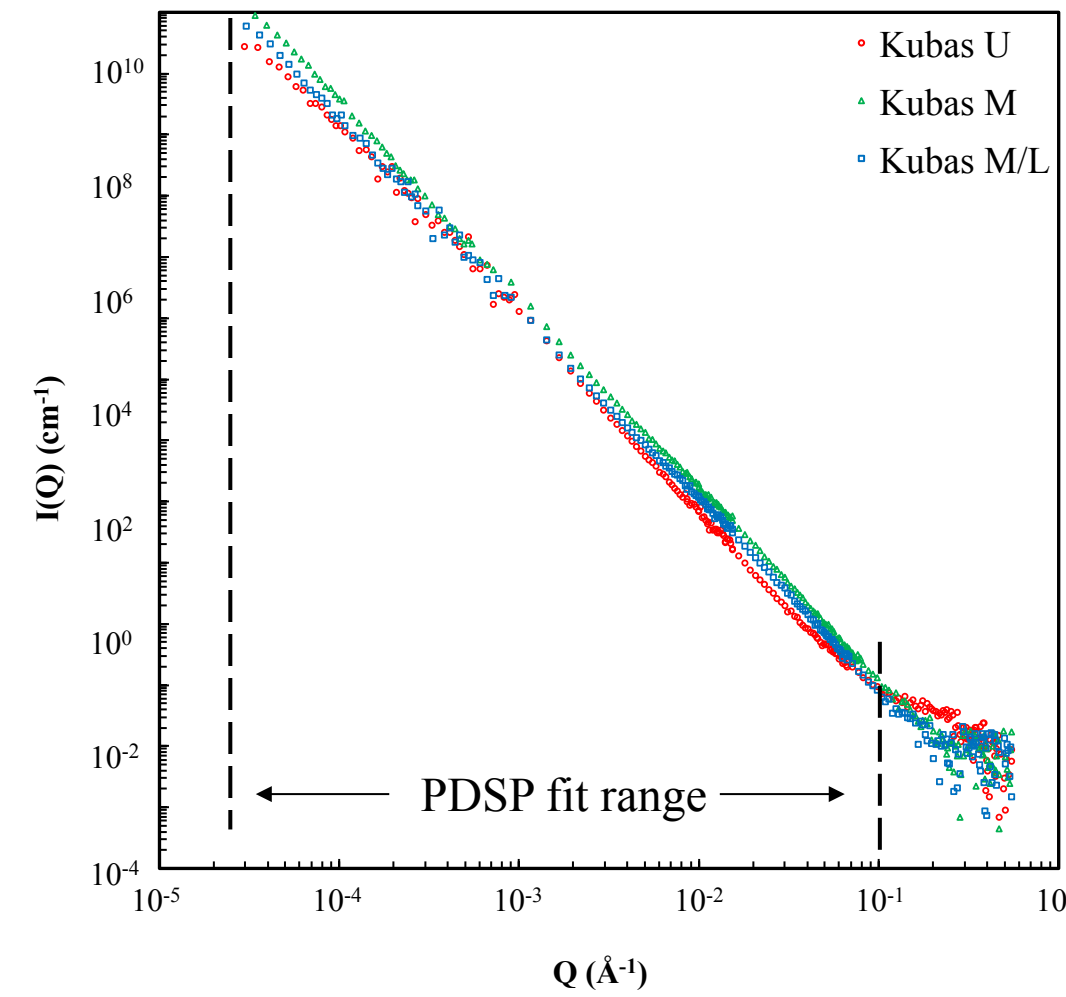


Figure 3. Background-subtracted USANS/SANS profiles and PDSP model fitted range

## Results 2

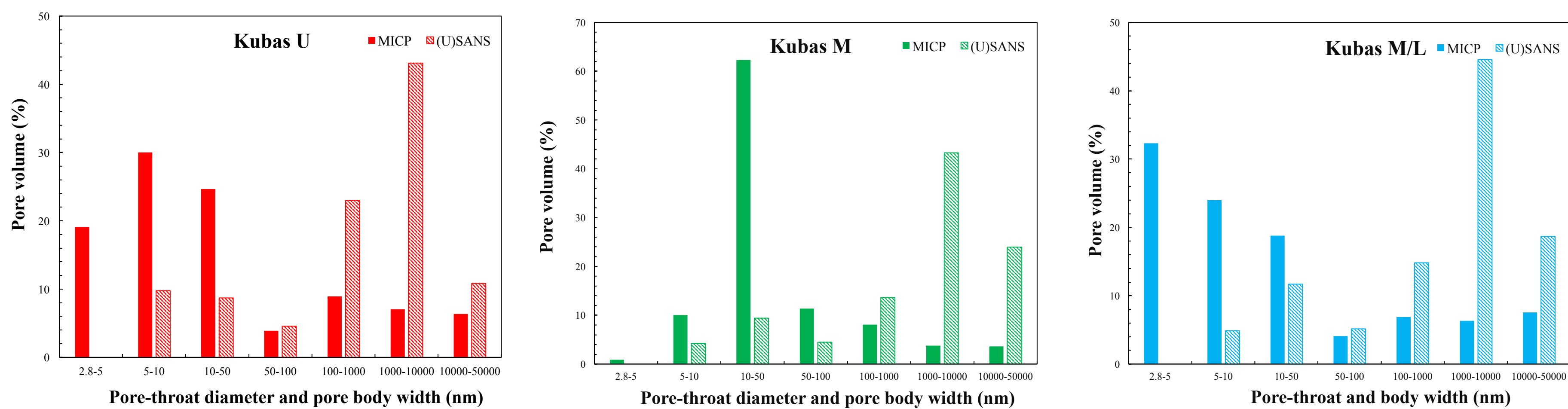


Figure 4. Pore-throat size distribution from MICP and pore-width distribution from (U)SANS

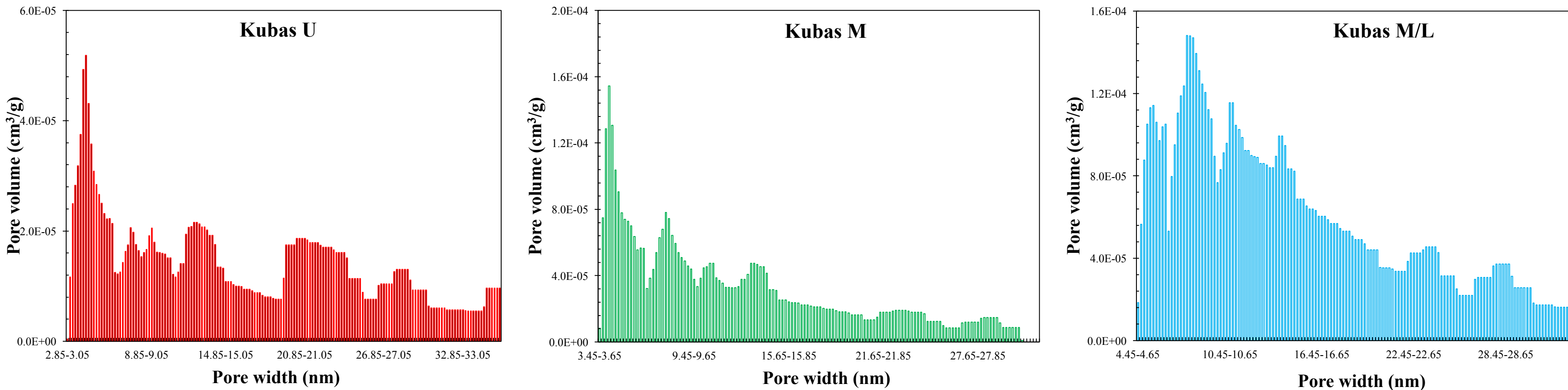


Figure 5. Pore width distribution of mesopores from N<sub>2</sub> sorption isotherm

## Discussion

- MICP results suggest a large portion of nm-sized pore-throats, and (U)SANS results indicate a dominant presence of  $\mu\text{m}$ -sized pores (Fig. 4), indicating the whole pore system, covering both large pores ( $\mu\text{m}$ -size) and small pore (nm-size), is primarily connected by nm-scale pore-throats. (U)SANS probes all (i.e., both body and throat) pore surfaces, while MICP only measures the throat size of pores that are accessible from sample surface, making PDSP model-derived (U)SANS porosity larger than MICP porosity. However, the organic porosity ( $<10$  nm) is more or less underestimated, as PDSP model is only fitted to the power-law portion of the data, while the bumps at high-Q, which represents organic-pores, are not included.
- Low-pressure nitrogen sorption isotherm has a narrow measurement range of 2-30 nm for pore width (Fig. 5). It proves the existence of lots of small pores ( $<15$  nm), and compensates the defect of PDSP model. However, this method is limited to its detecting pore size range, especially in  $\mu\text{m}$ -scale.
- Micropore ( $<2$  nm) volume contributions are not currently interrogated: (a) the upper limit of MICP injection pressure is 60,000 psi (corresponding to 2.8 nm in pore throat) (b) during (U)SANS data analyses, we fit the PDSP model to a high Q  $\sim 0.1 \text{ \AA}^{-1}$  (corresponding to  $\sim 5$  nm); we are applying suitable models in higher Q range to obtain pore-size distribution below 5 nm.

## On-going Work

We are focusing on samples from another producing Bakken well Anderson (Fig. 6), as well as Utica and Macos shales with different maturities, with improved methodologies for (U)SANS technique.

We aim to quantify (1) the influence of wettability on shale pore accessibility and (2) the fraction of closed pores inside the shale through contrast matching method using different deuterated fluids (Fig. 7).

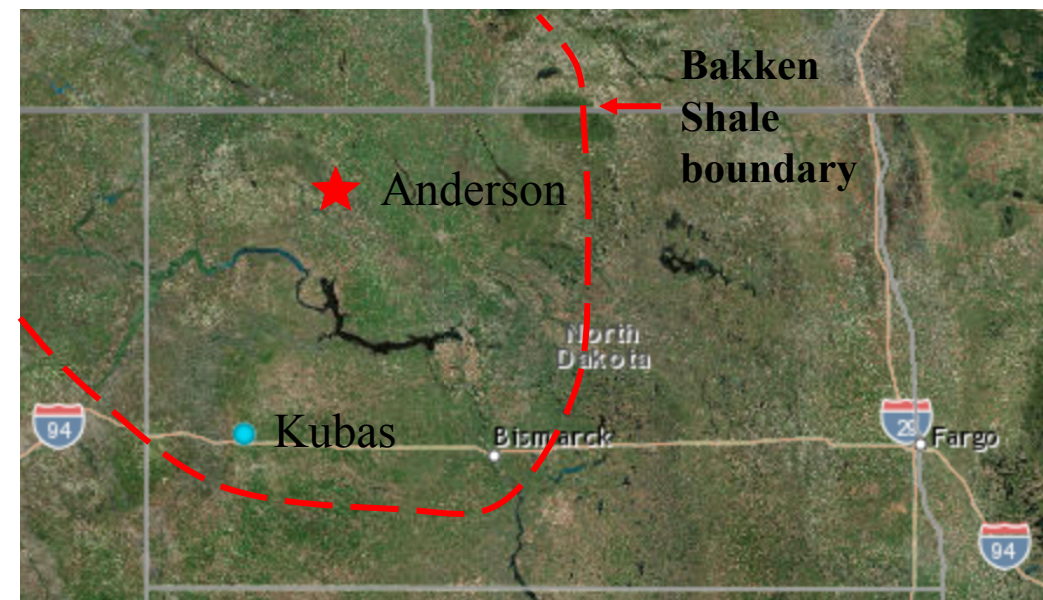


Figure 6. Location of Anderson well

Table 4. Current work progress on samples from Anderson well

Sample	Photo	XRD	MICP	N <sub>2</sub>	TOC	SANS/USANS		
						Dry	Mixed n-decane and deuterated n-decane	Mixed water and deuterated water
Anderson U (10064 ft)		✓	✓	TBD	✓	✓	✓	TBD
Anderson M (10121 ft)		✓	✓	TBD	✓	✓	✓	TBD
Anderson L (10124 ft)		✓	✓	TBD	✓	✓	✓	TBD

Figure 7. Cells loaded with dry sample (left) and sample saturated with mixed n-decane and deuterated n-decane (right)

## Conclusions

- Both MICP and (U)SANS measurements indicate that Bakken Shale has a wide range of pore lengths across  $\mu\text{m}$ -nm spectrum. Large pores of  $\mu\text{m}$ -scale dominate the pore systems, while pore-throats are basically of nm-scale. High-Q data of (U)SANS and N<sub>2</sub> adsorption exhibit the presence of a large amount of small pores in the range below 5 nm.
- The organic-rich upper Bakken shale (Kubas U) is of more complicated pore structure and more small pores than the middle Bakken (Kubas M) dolomites, while the bottom of middle Bakken (Kubas M/L) shows more similarity with upper Bakken because of organic matter intrusion from the lower Bakken.

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