**Pore characteristics of Longmaxi shale gas reservoir in the Northwest of Guizhou, China:
Investigations using small-angle neutron scattering (SANS), helium pycnometry, and gas
sorption isotherm**

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Abstract: Small-angle neutron scattering (SANS), helium pycnometry, and low-pressure gas sorption isotherm experiments were used to investigate the pore characteristics of Longmaxi shale, the leading producing formation in China. This work is the first microstructural study of Chinese marine shales by neutron scattering technique. The polydisperse spheres model (PDSP) was used for SANS data analysis to obtain porosity and pore size distribution (PSD), and the results were compared with those from gas (CO₂, N₂) adsorption and helium pycnometry. By evaluating the difference of porosities determined from these methods, the closed fraction of shale pores are derived and discussed. Moreover, the porosity has a positive correlation with total organic carbon (TOC) in Longmaxi shale samples. The fractal dimension of shale samples was derived, and results indicated that Longmaxi shale is a mass fractal. A lower mass fractal dimension means less closed pores with correspondingly more open structures.

Keyword: shale gas; pore characteristics; small-angle neutron scattering; helium pycnometry; gas

adsorption

1.Introduction

With a growing worldwide interest in alternative energy sources, natural gas and oil stored in shale are being actively explored (Kerr, 2010; Kargbo et al., 2010). Understanding pore characteristics of shale is essential to evaluating hydrocarbon storage capacity and flow capability in shale reservoir (Chalmers and Bustin, 2012; Slatt and O'Brien, 2011). A variety of techniques have been applied to study the pore characteristics of shale, which are divided into two categories: 1) imaging methods including field emission-scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM) and helium ion microscopy (HIM) (Loucks et al., 2012; Jiao et al., 2014; King et al., 2015); 2) small-molecule fluid immersion methods consisting of helium pycnometry, low-pressure gas (N_2 and CO_2) sorption isotherm analysis and mercury injection capillary pressure (MICP) analysis (Mastalerz et al., 2013; Yang et al., 2014; Sun et al., 2016). Samples used for the imaging methods need to be polished and coated, and are often examined for a limited area (on the order of hundreds of μm^2). Similarly, the immersion methods has a drawback of inability of detecting closed pores as the probing fluids can only reach surface-accessible pore spaces.

Small-angle x-ray and neutron scattering (SAXS and SANS), as well as ultra-small-angle x-ray and neutron scattering (USAXS and USANS), are well suited for the investigation of porous materials and provide complementary pore structure information such as porosity, pore size distribution and specific surface area (Hu et al., 2011; Schmidt, 1991). Neutron scattering techniques can nondestructively measure pores from nanometer to micrometer scale and detects total porosity (both closed and open pores), which presents an obvious advantage over traditional techniques (Bahadur, 2014). In addition, the characteristics of pore-matrix interface are also revealed by SANS techniques (Wong and Howard, 1986). In 2004, Radlinski et al. demonstrated the applicability of SANS and SAXS to determine the porosity, pore size and surface area in coals and sedimentary rocks (Radlinski et al., 2004a,b). Later, Radlinski et al. (2009) and Melnichenko et al. (2009) started working on capillary condensation of CO_2 in coals using x-ray and neutron scattering which was relevant to CO_2 sequestration by deserted underground coal mines. They

were able to present the average density of CO₂ confined in pores and accessibility of pores as a function of pore sizes which are difficult to obtain by other techniques (Melnichenko et al., 2009, 2012). Meanwhile, Sakurovs et al. (2009) tested the stability of pores in bituminous coal upon exposure to pressures up to 155 bar using SANS and USANS. These early studies provide a convenient basis for SANS and USANS studies on pore structures in shale, and ensuing work of interactions between methane and shale (Mastalerz et al., 2012; Clarkson et al., 2013; Jin et al., 2013; Ruppert et al., 2013; Lee et al., 2014; Bahadur et al., 2014, 2015; Anovitz and Cole, 2015; Zhang et al., 2015; Gu et al., 2015, 2016).

SANS and USANS intensity of shale samples mainly come from the neutron scattering contrast between pores and the matrix, with the minerals and organic phases have the scattering length density (SLD) of similar magnitude (Radlinski et al., 1996; King et al., 2015). The shape of neutron scattering curves is determined by the pore size distribution and porosity in shale samples. In principle, the shape of the pores also affects the scattering curve; however the wide distribution of pores in shale often smears out the form factors and a polydisperse spherical model is used in almost every neutron scattering studies on shale (Melnichenko, 2015). Combined SANS and USANS curve from shale samples is usually linear on a log-log scale over a wide Q ($= 4\pi\lambda^{-1} \sin(\theta)$ and 2θ is the scattering angle and λ is neutron wavelength) range which is attributed to a power law size distribution of pore sizes or fractal pore-matrix interface (Radlinski et al., 2004b; Melnichenko, 2015).

In a prior work, the pore size distributions over a size range of 5 to 1000 Å obtained from CO₂/N₂ adsorption were in reasonable agreement with those extracted from SANS/USANS studies of North American shale samples (Clarkson et al., 2013), suggesting that most of the pores in this size range were open to the surface. Bahadur et al. (2014) estimated the closed porosity fraction of Canadian Cretaceous shale samples by comparing porosity obtained from helium pycnometry with that from SANS. The pore connectivity was assessed from the combined SANS and MICP analysis (King et al., 2015). Moreover, some researchers also studied the fractal dimension of shale reservoir by SANS to characterize the irregularities of shale's pores (Mastalerz et al., 2012; Clarkson et al., 2013; Lee et al., 2014).

In this work, we focused on a representative shale formation in China: the Lower Silurian

Longmaxi Shale. This is the first time to quantitatively characterize the porosity and pore size distribution (PSD) of Chinese marine shale samples using a combination of SANS, helium pycnometry, and gas adsorption. Moreover, this work discussed the pore connectivity and fractal dimension, as well as their relations to total organic carbon (TOC) and thermal maturity.

2 Shale samples and analyses

2.1. Sample collection

A total of 9 Lower Silurian Longmaxi shale samples from Well XY1 and Well TY1 were used for analyses in this work. Both the wells distribute in the northwest of Guizhou province, Southwest China, where is the southern margin of Sichuan basin (Fig.1). The sample descriptions are listed in Table 1.

2.2 Organic petrology and X-ray diffraction

TOC (total organic content) was measured by a Leco carbon/sulfur analyzer. Samples were crushed to less than 100 mesh, and treated by hydrochloric acid to remove carbonates until no bubbles were produced. Then, the samples were washed by deionized-water until the water became neutral. Dried samples of about 110mg were pyrolyzed up to 540°C. The vitrinite reflectance (VRr) of shale samples were calculated by bitumen reflectance (BRr) conversion with the equation of $VRr = (BRr + 0.2443)/1.0495$ (Schoenherr et al., 2007).

X-ray diffraction was employed for mineral composition analysis of shale samples. Crushed samples (less than 300 mesh size) were measured with BRUKER (D8 DISCOVER) X-ray diffractometer and semi-quantified by Jade[®] 6.0 software.

2.3 SANS experiments

SANS experiments were performed at Oak Ridge National Laboratory (ORNL) on the General Purpose SANS (GP-SANS) instrument using neutron wavelengths of $\lambda=12 \text{ \AA}$ and $\lambda=4.72 \text{ \AA}$ ($\Delta\lambda/\lambda\sim 0.15$). Before the SANS experiments, samples were prepared by cutting as 10mm×10mm×2mm slices which parallel to bedding. The samples were then polished to their final thickness of less than 1.0 mm using crocus paper and dried under vacuum at 60°C for at least 24h. The sample-to-detector distances were used, 18.5m, 10m and 1m, covering a Q range of 0.001 to 0.5 $\text{\AA}^{-1}$. For polydisperse porous media, an appropriate relationship between pore radius

and the Q is used: $r \approx 2.5/Q$ (Ruppert et al., 2013; Bahadur et al., 2015; Clarkson et al., 2012; Radlinski et al., 2000). Therefore, SANS experiments in our work allowed to measure the pore radius from approximately 250nm to 0.5nm (500nm to 1nm in diameter). The isotropic raw data were reduced to one dimensional data by IGOR Pro software, and they were analyzed using PRINSAS software (Hinde, 2004).

2.4 Helium pycnometry

The helium porosity and density of shale samples were conducted by KXD-II porometer following SY/5336-2006 standard (Li et al., 2016). Shale samples were prepared by coring cylindrical plugs with 2.5cm in diameter and a height of 3cm. Porosity was obtained from the difference between bulk density and skeletal density with a helium expansion method (Mastalerz et al., 2012; Li et al., 2016).

2.5 Low-pressure gas adsorption analyses

Low-pressure N_2 adsorption measurement was conducted by Quantachrome Autosorb-1 to obtain the pore volume and PSD of pores with diameters larger than 2nm in shales. Meanwhile, due to higher accessibility of CO_2 than N_2 , low-pressure CO_2 adsorption measurement were performed by Quantachrome Nova Station A for characterizing pores with diameters less than 2nm. Shale samples were crushed into 40-80 mesh and degassed at approximately 110°C under vacuum for about 5 hours. Nitrogen and carbon dioxide adsorption/desorption isotherms were obtained at 77.3K and 273.1K, respectively. The density functional theory (DFT) method was used for the pore volume and PSD calculation; more details about this method can be found in Gregg and Sing (1982) and Lastoskie et al. (1993).

3 Results and Discussion

3.1 Calculation of Scattering Length Density (SLD)

The sample information are listed in Table 1, including depth, mineral composition, TOC, and thermal maturity. The volume of each component in a sample was calculated based on its density. The SLDs of each component are presented in Table 2. They were calculated using Equation 1 (Radlinski et al., 1996; King et al., 2015; Bahadur et al., 2014, 2015):

$$\rho_n = \frac{N_A d}{M} \sum_j p_j (\sum_i s_i b_i)_j \quad (1)$$

where N_A is Avogadro's number ($N_A = 6.022 \times 10^{23}$), d is the density, s_i is the abundance of nucleus i in phase j , p_j is the proportion of phase j in the mixture and b_i is the coherent scattering amplitude for nucleus i .

The SLD of each shale sample (presented in Table 1) was estimated by a volume average over all components (including TOC), according to Equation 2 (Bahadur et al., 2014, 2015):

$$\text{SLD (shale)} = \frac{\sum_i^n \text{vol\%}(i) \text{SLD}(i)}{100} \quad (2)$$

where i means a component and n indicates the total number of components.

3.2 Analysis of SANS results

The SANS profiles of shale samples are shown in Figure 2A. The scattering curves follow power law over a Q range from 0.2 to $0.001 \text{ \AA}^{-1}$ which is similar to prior studies of shale samples (Clarkson et al., 2013; Ruppert et al., 2013; Bahadur et al., 2015;). The Polydisperse Spheres (PDSP) model was used to fit the data over the measured Q range, 0.5 to $0.001 \text{ \AA}^{-1}$. The obtained porosities of the shale samples are presented in Table 2. As shown in Table 2, they are much higher than typical porosity of shale samples (Bahadur et al., 2015; King et al., 2015). This suggests that the SANS data in the Q range of 0.2 to $0.5 \text{ \AA}^{-1}$ contain scattering from a separate population of pores, which have been reported in previous studies (King et al., 2015; Bahadur et al., 2014, 2015).

In the following analysis, we focused on the data in the Q range of 0.2 to $0.001 \text{ \AA}^{-1}$, corresponding to pore radii ranging from 1.25 to 250 nm. Figure 2B shows examples of the fits to PDSP model and the porosities are listed in Table 2. The obtained porosities are now lower than those obtained using the data over a Q range of 0.5 to $0.001 \text{ \AA}^{-1}$. The Porod invariant method has also been used to calculate the porosity (Bahadur et al., 2015; Clarkson et al., 2012, 2013). For a two-phase system consisting of shale matrix and pores, porosity (ϕ) can be calculated from the equation using the scattering contrast ($\Delta\rho$):

$$\int_0^\infty Q^2 I(Q) dQ = 2\pi^2 (\Delta\rho)^2 \phi (1 - \phi) \quad (3)$$

The Porod invariant (PI) porosities of shale samples obtained from PRINSAS are shown in Table 3. A comparison of porosities obtained from PDSP model and Porod invariant method suggests that

they are roughly consistent with each other.

3.3 Porosity Classes and PSD

Helium pycnometry measures the open pores with diameters larger than 0.2 nm due to size of helium gas molecules. Helium porosity and bulk density of shale samples are recorded in Table 3. Pore volume calculation in shales, based on the DFT model from cumulative adsorption data of nitrogen and carbon dioxide, has been commonly used in literature (Mastalerz et al., 2013; Yang et al., 2014; Sun et al., 2016). Combining with the bulk density, the N₂ and CO₂ porosities can be obtained and they are shown in Table 4. Carbon dioxide porosity contains the open pores with diameters from 0.3 to 1.4 nm. Meanwhile, N₂ porosity quantifies the open pores with diameters from 1.4 to 300 nm.

Helium molecules can penetrate all open pores including those measured N₂ and CO₂ adsorption (i.e., pore body diameters of 0.3~300 nm). The porosity measured from three different methods (helium porosimetry, PDSP analysis of SANS, and N₂ - CO₂ adsorption) are shown in Figure 3. The porosity measured by N₂ and CO₂ adsorption (pores less than 300nm in diameter) is only slight less than that measured by He, suggesting that most of the open pores in Longmaxi shale samples is less than 300 nm in diameter. The control factors of pores which diameters larger than 300 nm will be discussed later in the following section.

The pore size distributions (PSD)s (pore radii range from 1.25 to 250 nm) obtained using PDSP model for all shale samples are shown in Figure 4 and those extracted from CO₂ and N₂ gas sorption data are presented in Figure 5. As expected, the PSDs obtained from SANS data follow power law decay (Figure 4). Shown in Figure 5, the PSDs extracted from CO₂ adsorption data exhibit three volumetric maxima (at approximately 0.35 nm, 0.5 nm, and 0.8 nm) in Longmaxi shales. The PSDs from N₂ adsorption data present volumetric maxima at pore sizes ranging from 2 to 4 nm. In order to provide direct comparison of PSDs derived from SANS and N₂ adsorption, we have converted volume-weighted, $f(r)$, in Figure 4 and pore volume distribution, dV/dD , in Figure 5, into pore volume distribution, dV/dD , and compared them in Figure 6. In general, the PSD from SANS and gas adsorption have a similar trend. The PSD of N₂ adsorption show fluid condensation in smallest mesopores, this discrepancy may be due to the sample preparation for N₂. Here the samples were crushed into 40-80 mesh for N₂ adsorption, whereas SANS is measured on thin slice

samples.

3.4 Relationships between porosity and TOC/clay content

Previous studies have shown that organic matter-hosted pores are an important part of the shale's pore-system (Loucks et al., 2012; Curtis et al., 2012; Shi et al., 2015; Yang et al., 2016). Organic matter contains most of the pores in the Longmaxi samples, as indicated by the linear correlation between the TOC and porosity and also revealed by FE-SEM images (Shi et al., 2015; Yang et al., 2016). Figure 7A shows that both He and PDSP porosities have a positive correlation with TOC in Longmaxi shale samples, which agrees with previous studies (Shi et al., 2015; Yang et al., 2016).

In this work, the correlation between the porosity and clay content was also discussed for Longmaxi samples. Using FE-SEM, previous studies showed that pores in clay minerals of Longmaxi shale were normally greater than 300nm (Shi et al., 2015; Yang et al., 2016). Therefore, the pores which diameters larger than 300nm are mainly composed of pores in clay minerals as well as part of organic pores. The porosities of these pores was estimated by subtraction of N₂/CO₂ porosities from He porosity (Δ porosity = He porosity - N₂ porosity - CO₂ porosity). In order to reduce the effects of organic pores, the Δ porosity was normalized to TOC content. In Figure 7B, the clay mineral content shows a positive correlation with the TOC-normalized Δ porosity, indicating that the content of clay minerals affects the proportion of macropores in the Longmaxi shales.

3.5 Contribution of closed porosity

The porosities of some shale samples estimated by SANS are larger than those derived from helium, this is because SANS porosity includes the closed pores. The fraction of closed porosity ($F_{\text{closed pore}}$) is therefore estimated by calculating the difference between porosity obtained by SANS and other techniques:

$$F_{\text{closed pore}} = \frac{\text{PDSP porosity} - (\text{He porosity} - \text{CO}_2 \text{ porosity} - \text{N}_2 \text{ porosity}_{(1.4-2.5)})}{\text{PDSP porosity}} \quad (4)$$

where the porosity of small pores with diameters less than 2.5nm was subtracted from the Helium porosity, matching the lower limit of the pore size measured by SANS. The open porosity thus calculated contained the pores with diameters larger than 500nm, which was not covered in PDSP

porosity. However, as shown in Figure 3, the contribution to porosity from pores with diameters larger than 300nm is already small. Therefore, the overall error associated with the result of Equation 4 is expected to be small.

The fraction of closed porosity for each shale sample is given in Table 3. A lower value of closed porosity means that the sample contains more open structures in shale, with better pore connectivity (Melnichenko, 2015). Ruppert et al. (2013) reported that more than 85% of the pores were accessible to CD₄ and D₂O in Barnett Shale. Bahadur et al. (2014) found that 20-37% of pores were closed in Cretaceous Shale. If methane is stored in the closed pores, the hydraulic fracturing will lead to methane release and hence increase the shale gas production. Therefore, the fraction of closed pores is not only indicative of pore connectivity, but also directly impacts gas well production and ultimate recovery. Detailed investigations of closed pores are currently underway.

3.6 Fractal characteristics of Longmasi shale

As shown in Figure 2b, after subtracting the background (Table 3), the scattering curves follow power law over a Q range from 0.2 to 0.001 Å⁻¹. The power law exponent has been correlated with surface or mass fractal of the studied samples (Lee et al., 2014; Bahadur et al., 2015; Clarkson et al., 2013). When the power-law exponent (p) is greater than 3, it reflects the surface fractal characteristic of shale with the surface fractal dimension (D_s) is equal to 6- p . If the power-law exponent is less than 3, it represents the mass (aggregates) fractal behavior of shale with the mass fractal dimension (D_m) is equal to the value of p . In this work, all power-law exponents of shale samples are slightly less than 3. It means that Longmaxi shale samples present mass fractal character or transition from surface to mass fractal behavior. The mass fractal dimension (D_m) was given by the PRINSAS analysis and are presented in Table 3.

The fractal dimension has been used to characterize the irregularities of shale reservoir in recent years (Yang et al., 2014; Sun et al., 2016; Lee et al., 2014; Li et al., 2016). Previous studies demonstrated that the surface fractal dimension shifted to mass fractal dimension with an increasing maturity (Lee et al., 2014; Jin et al., 2011). Thus, the mass fractal dimension is expected to continue to decrease with the increase of maturity. Clarkson et al. reported that most north American shale (maturity less than 2.0%) corresponded to surface fractal dimensions from

2.7 to 3.0 (Clarkson et al., 2013). Lee et al. (2014) demonstrated the shift from surface to mass fractal after the heat treatment of shale samples. Therefore, the Longmaxi shale samples with maturity (R_o) higher than 2.0% likely possess mass fractal behavior.

The mass fractal dimension is essential to estimate the irregularity of shale and diffusive transport of fluid in shale. As shown in Figure 8, the higher values of mass fractal dimension correspond to more closed pores. The fraction of closed pores can reflect the pore connectivity in shale as discussed in Section 4.3. Therefore, the higher values of mass fractal dimension may show less open structures in shale, corresponding to worse pore connectivity.

5 Conclusions

- (1) The pore size distributions obtained from SANS and low-pressure gas (CO_2 and N_2) adsorption were compared.
- (2) Both He porosity and PDSP porosity have positive correlations with TOC in Longmaxi shale samples. The content of clay minerals affects the proportion of macropores in the Longmaxi shales.
- (3) The fraction of closed porosity are estimated to be 6.69-42.6% by calculating the difference between porosity obtained by SANS and gas adsorption techniques. Strong differences in fraction of closed porosity exist between samples.
- (4) The Longmaxi shale with maturity (R_o) higher than 2.0% already have mass fractal dimension behavior. The greater values of mass fractal dimension correspond to more closed pores.

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Figure 1. Location map of wells around Sichuan Basin [modified after Sun et al. (2016)]

Figure 2. SANS profiles of Longmaxi shale samples (A) and PDSP model (B) fit for two representative shale samples.

Figure 3. Porosity measured from three different methods: helium porosimetry (blue), PDSP analysis of SANS (red), and N₂ (yellow) / CO₂ (green) adsorption

Figure 4. Pore volume distribution vs. pore size, calculated using the PDSP model.

Figure 5. Pore volume distribution vs. pore size, derived from the adsorption branches of CO₂ and N₂ gas sorption isotherms with DFT model for Longmaxi shale samples.

Figure 6. Comparison of pore volume distributions obtained from combined N_2 adsorption data and SANS.

Figure 7. (A) Correlation between porosity and TOC; Red cycles: He porosity; Blue cycles: PDSP porosity. (B) Correlation between TOC-normalized Δ porosity (He porosity - N_2 porosity - CO_2 porosity) and clay content.

Figure 8. Relationships between fraction of closed pore and mass fractal dimension of shale samples.

Table 1. Parameters of Lower Silurian Longmaxi shale samples

Sam ple	W ell	De pth (m)	Quantitative analysis of whole-rock minerals (wt.%)							TOC (wt. %)	R _o (%)	SLD ($\times 10^{10}$ c m ⁻²)
			Cla ys	Qua rtz	K-feld spar	Plagioc lase	Calc ite	Dolo mite	Pyr ite			
XY1 -66	X Y1	610	53	32	0	6	5	3	1	1.05	2. 34	3.55
XY1 -77	X Y1	621 .5	28	20	1	5	3	41	2	1.09	2. 67	3.64
XY1 -90	X Y1	637 .2	31	45	2	6	10	3	3	3.72	2. 17	3.67
XY1 -94	X Y1	641 .1	15	70	0	2	8	3	2	3.61	2. 01	3.86
XY1 -96	X Y1	643 .2	18	26	0	3	10	40	3	4.88	2. 21	3.63
TY1- 12	T Y1	667 .3	27	18	1	4	45	4	1	1.93	2. 04	3.62
TY1- 14	T Y1	670	21	30	2	10	28	5	5	7.58	2. 12	3.57
TY1- 17	T Y1	673	13	75	1	3	8	0	1	4.43	2. 21	3.87
TY1- 18	T Y1	674	16	61	0	7	7	5	3	3.86	2. 45	3.81

Table 2. Parameters used in the calculation of Scattering Length Density (SLD)

Mineral name	Density, d	Molecular weight, M	Molecular scattering amplitude, $\Sigma s_i b_i$	SLD=($N_A d/M$) $\Sigma s_i b_i$
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	(g/cm ³)	(g/mol)	($\times 10^{-12}$ cm)	($\times 10^{10}$ cm ⁻²)
Quartz	2.65	60.085	1.5755	4.18
Carbonates	2.80	100.0872	2.2309	3.76
Plagioclase	2.61	262.2237	6.595	3.95
K-feldspar	2.6	278.332	6.603	3.71
Pyrite	5.0	119.977	1.5234	3.62
Clays	2.63	263.161	5.2467	3.16
TOC	1.3	1908	60.32	2.50

Table 3. Sample and pore characteristics from helium pycnometry and SANS analyses

Sample	Bulk Density (g/cm ³)	He Porosity (%)	PI Porosity (%)	PDSP Porosity (%) Q<0.5 Å ⁻¹	PDSP Porosity (%) Q<0.2 Å ⁻¹	SANS flat background (cm ⁻¹)	Fractal dimension D _m	Fraction of closed pore (%)
XY1-6	2.69	3.51	2.2	16.1	3.1	0.066	2.79	6.7
XY1-7	2.68	3.66	2.2	12.9	2.8	0.049	2.81	11.3
XY1-9	2.53	4.52	3.5	11.5	4.5	0.039	2.89	27.7
XY1-9	2.53	4.96	5.4	14.2	5.5	0.053	2.93	36.2
XY1-9	2.38	6.59	6.7	19.4	6.7	0.056	2.92	27.0
TY1-12	2.67	4.43	5.3	15.3	5.3	0.045	2.98	29.8
TY1-14	2.27	8.14	8.7	31.9	9.2	0.102	3.00	42.6
TY1-17	2.46	4.91	4.5	17.8	4.8	0.065	2.85	22.4
TY1-18	2.4	6.65	2.3	37.6	6.9	0.225	2.99	34.5

*PI= Porod Invariant; PDSP= Polydisperse Spheres

Table 4. Pore volumes (PV) and porosities determined via gas adsorption

Sample	CO ₂ sorption (0.3-1.4 nm)		N ₂ sorption (1.4-300 nm)		
	PV (cm ³ /kg)	Porosity (%)	PV (cm ³ /kg)	Porosity (%)	Porosity _(1.4-2.5 nm) (%)
XY1-66	1.28	0.34	10.5	2.82	0.28
XY1-77	3.54	0.95	9.46	2.54	0.25
XY1-90	3.79	0.96	11.9	3.01	0.30
XY1-94	4.5	1.14	13.7	3.47	0.35
XY1-96	4.91	1.17	21.3	5.07	0.51
TY1-12	1.23	0.33	14.6	3.90	0.39
TY1-14	9.99	2.27	25.2	5.72	0.57
TY1-17	3.22	0.79	15.7	3.86	0.39
TY1-18	6.96	1.67	20.2	4.85	0.48