Short Synthesis of Ordered Silicas with Very Large Mesopores

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Supporting Table S1. Synthesis conditions and structural parameters for samples.[a]

synthesis conditions	$d_{100\,\mathrm{UC}}$ (nm)	d_{100} (nm)	Extent of shrinkage	S_{BET} ($m^2\ g^{-1}$)	V_t ($cm^3\ g^{-1}$)	V_{mi} ($cm^3\ g^{-1}$)	V_p ($cm^3\ g^{-1}$)	W_{KJS} (nm)	w_d (nm)
0g+40C/1H+170C/3H	10.6	10.5	1%	850	1.26	0.05	1.04	11.0	10.5
1.0g+17C/1H	14.5	12.7	12%	622	0.88	0.08	0.66	10.9	11.4
1g+17C/1H+100C/3H	14.3	13.8	3%	903	1.31	0.16	0.99	13.7	13.1
1g+17C/1H+130C/3H	14.3	13.9	3%	764	1.37	0.10	1.08	14.9	13.7
1g+17C/1H+150C/3H	14.3	14.0	2%	674	1.34	0.07	1.03	14.9	13.8
1g+17C/1H+170C/3H	14.2	14.0	1%	456	1.34	0.02	0.95	17.2	13.9
1g+17C/1H+190C/3H	14.1	14.1	0%	449	1.22	0.06	0.82	16.7	13.4
1g+17C/1H+170C/1H	14.5	13.6	6%	978	1.26	0.21	0.90	13.3	12.5
1g+17C/1H+170C/2H	14.3	14.1	1%	725	1.37	0.07	1.09	14.5	14.1
1g+14C/1H+170C/3H	16.4	16.2	1%	405	1.12	0.04	(0.84)[c]	18.4	15.6
[d]1g+14C/24H+100C/48H	19.4	18.6	4%	570	1.13	0.05	0.81	20.4	17.7
2g+13C/1H+170C/4.5H	22.7	22.1	3%	425	1.63	0.02	(1.28)[c]	27.3	22.9
[d]2g+13C/24H+130C/24H	20.8	20.1	3%	350	1.34	0.02	(1.03)[c]	25.5	20.2
3g+12.5C/1H+170C/4.5H	e	28.9	f	330	1.76	0.00	(1.38)[c]	36.1	30.5
3g+12.5C/24H+130C/24H	27.2	25.2	7%	332	1.60	0.00	(1.25)[c]	33.4	26.4
3g+12.25C/1H+170C/6H	e	29.0	f	290	1.65	0.00	(1.29)[c]	39.2	30.2
3g+12.25C/24H+130C/24H	e	30.5	f	309	1.67	0.00	(1.31)[c]	38.6	31.9
3g+12.5C/1H	30.5	23.6	23%	296	0.87	0.09	(0.59)[c]	24.1	20.6
3g+12.5C/1H+100C/6H	27.2	25.6	6%	518	1.79	0.11	(1.29)[c]	29.2	25.9
3g+12.5C/1H+130C/6H	27.6	26.0	6%	411	1.48	0.05	(1.11)[c]	30.7	26.1
3g+12.5C/1H+150C/6H	27.6	26.8	3%	380	1.71	0.03	(1.31)[c]	32.5	27.8
3g+12.5C/1H+170C/6H	e	28.5	f	297	1.78	0.00	(1.39)[c]	37.7	30.0
3g+12.5C/1H+170C/3H	27.2	26.8	1%	386	1.65	0.02	(1.27)[c]	32.8	27.7
3g+12.25C/1H+170C/3H	e	28.0	f	326	1.27	0.02	(0.97)[c]	37.2	27.8

[b]14C/3H+170C/4.5H	[g]19.6	[g]19.0	3%	426	0.87	0	0.84	17.4	22.3

[a] Data taken in part from L. Cao, Ph.D. Dissertation, Graduate Center, City University of New York, 2010. Sample Notation: Xg+YC/Y'H+ZC/Z'H, sample was synthesized with X grams of micelle expander per 2.4 g of Pluronic P123 at initial temperature Y (°C) for Y' hours and at the hydrothermal treatment temperature Z (°C) for Z' hours;
$d_{100\,\mathrm{UC}}$ and d_{100}, (100) interplanar spacing for uncalcined sample and calcined sample;
shrinkage, calculated as 1- d_{100}/ $d_{100\,\mathrm{UC}}$ for SBA-15 or 1- d_{111}/ $d_{111\mathrm{UC}}$ for LP-FDU-12;
S_{BET}, BET specific surface area;
V_t, total pore volume;
V_{mi}, micropore volume;
V_p, primary mesopore volume;
W_{KJS}, BJH-KJS pore diameter;
w_d, pore diameter calculated using geometrical Equations 1 (SBA-15) or 2 (FDU-12).

[b] Synthesis conditions for LP-FDU-12

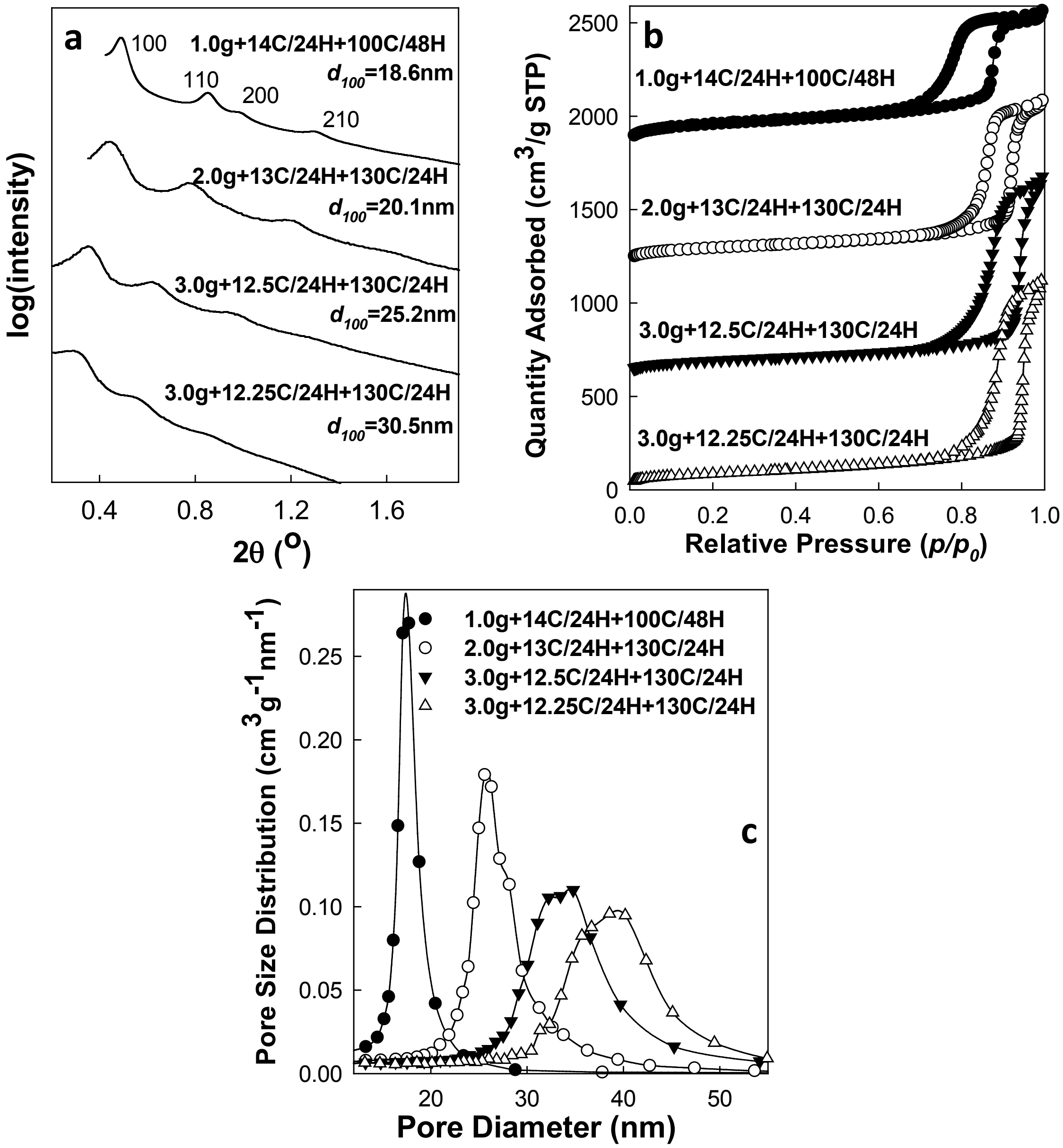
[c] Could not be calculated with acceptable accuracy using α_s plot method. Values in parentheses are estimates based on V_t values for the particular sample, and V_p data for samples with similar shape of the adsorption isotherms.

[d] Data taken from Cao, L.; Man, T.; Kruk, M., *Chem. Mater.* **2009,** *21*, 1144.

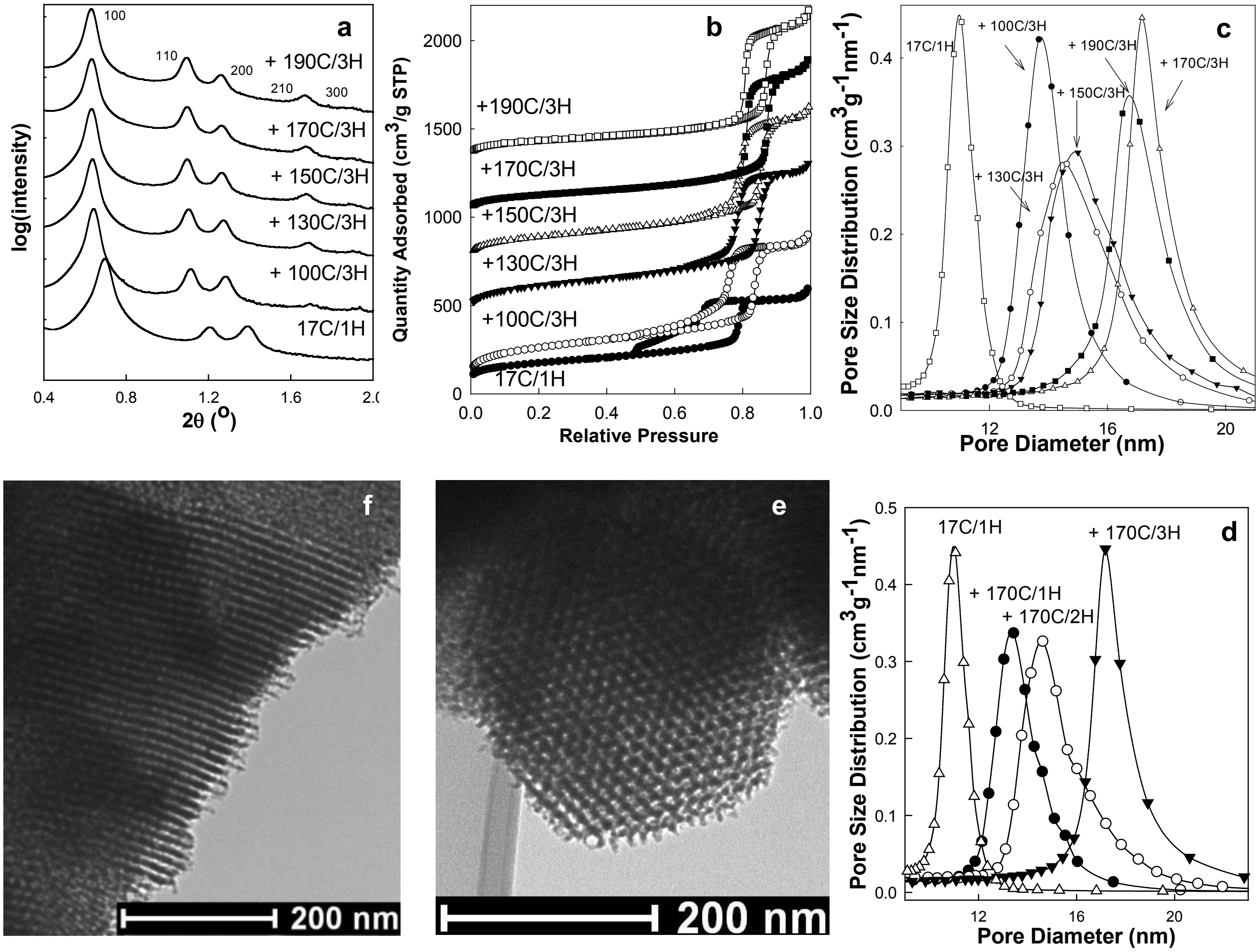
[e] No clear peak on SAXS pattern.

[f] Cannot be calculated because $d_{100\,\mathrm{UC}}$ was not available

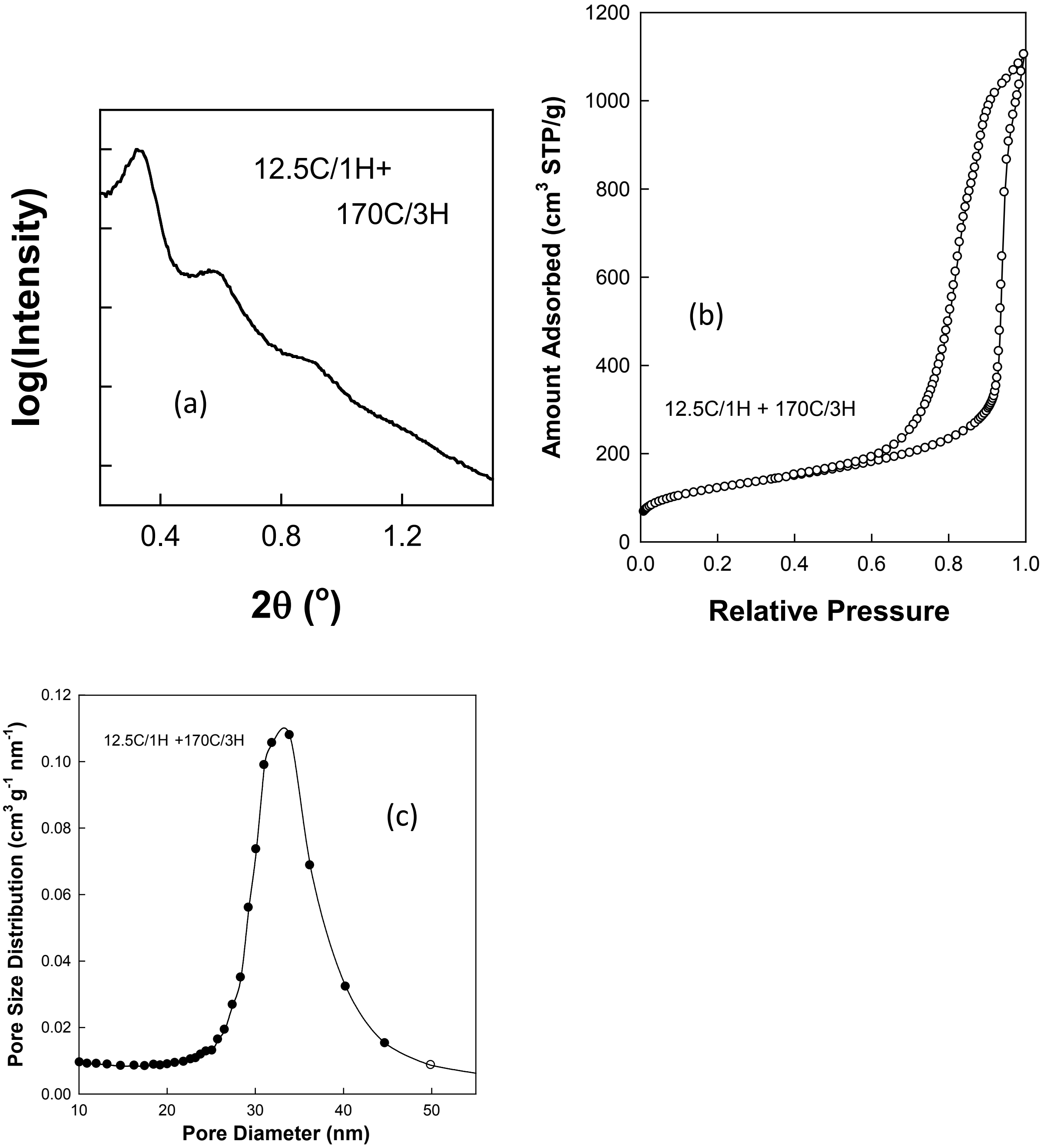
[g] d_{111} for sample LP-FDU-12



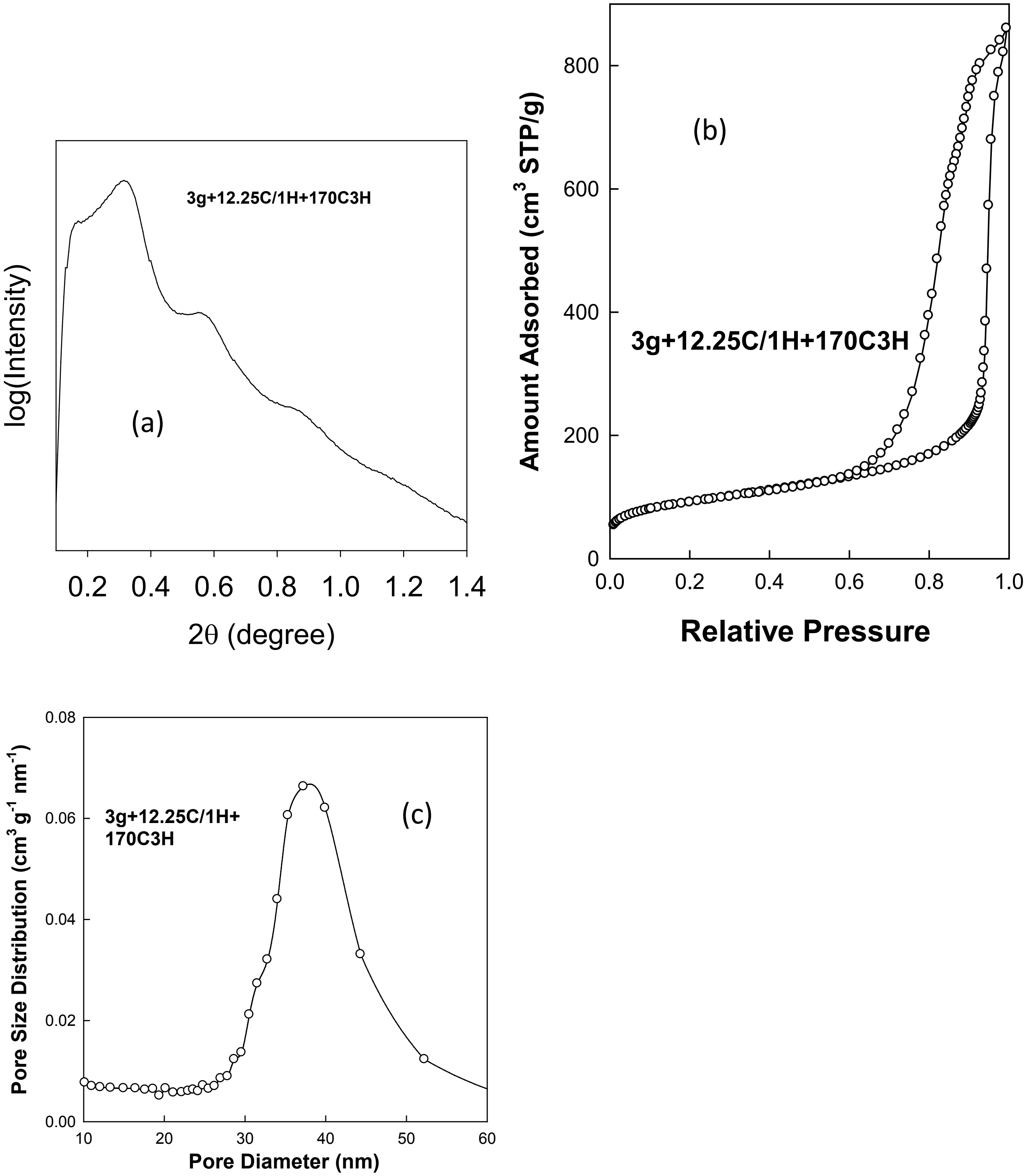
Supporting Figure S1. (a) SAXS patterns, (b) nitrogen adsorption isotherms and (c) pore diameter distributions of calcined samples prepared with various amount of TIPB and at different initial temperatures and hydrothermal treatment conditions. The SAXS patterns were offset vertically to facilitate comparison. The isotherms for samples 3.0g+12.5C/24H+130C/24H, 2.0g+13C/24H+130C/24H and 1.0g+14C/24H+100C/48H were shifted vertically 600, 1200 and 1800 cm^3 STP g^{-1}, respectively. Data for samples 2.0g+13C/24H+130C/24H and 1.0g+14C/24H+100C/48H are taken from Cao, L.; Man, T.; Kruk, M., *Chem. Mater.* **2009,** *21*, 1144.



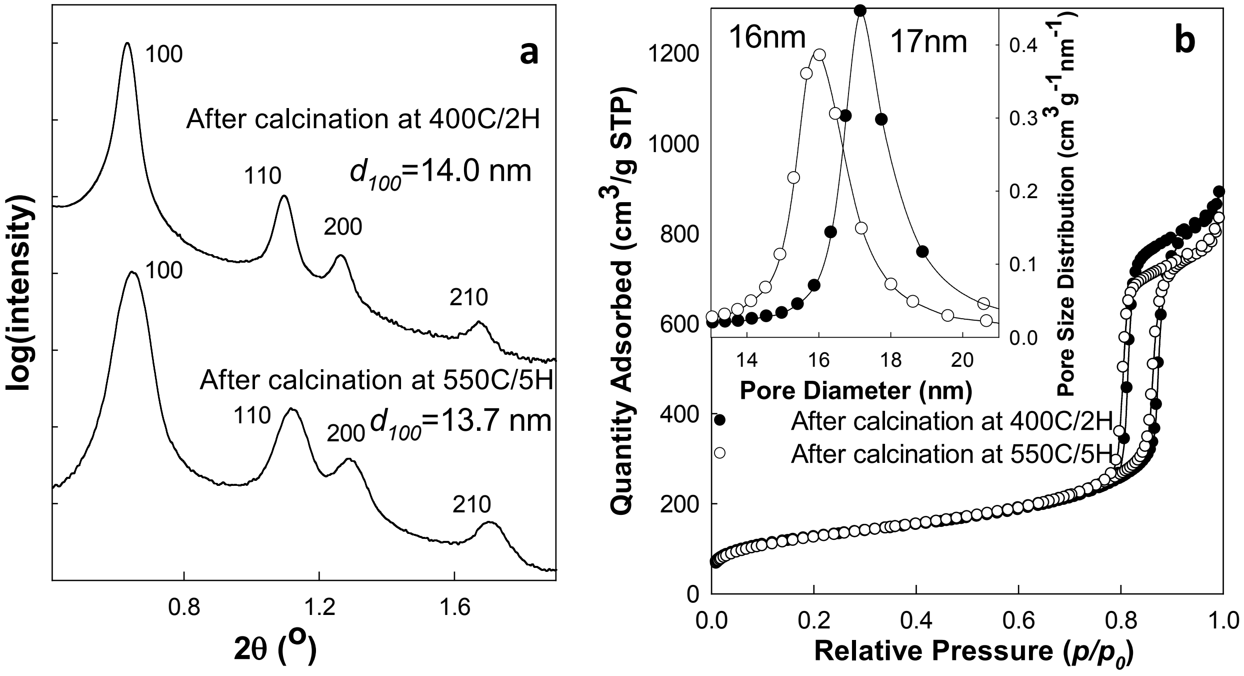
Supporting Figure S2. (a) SAXS patterns of calcined samples prepared at an initial temperature of 17 °C and hydrothermally treated at temperatures from 100 to 190 ^{o}C for 3 hours. The patterns were offset vertically to facilitate comparison; (b) and (c) nitrogen adsorption isotherms and pore size distributions of the above samples. The isotherm for samples +130C/3H, +150C/3H, +170C/3H, +190C/3H were shifted vertically 400, 700, 1000, and 1300 cm^{3} STP g^{-1}, respectively; (d) pore size distributions of samples hydrothermally treated at 170 ^{o}C from 1 to 3 hours compared with the sample prepared without hydrothermal treatment; (e) and (f) TEM images of calcined sample hydrothermally treated at 170 ^{o}C for 3 hours. Figures taken from L. Cao, Ph.D. Dissertation, Graduate Center, City University of New York, 2010.



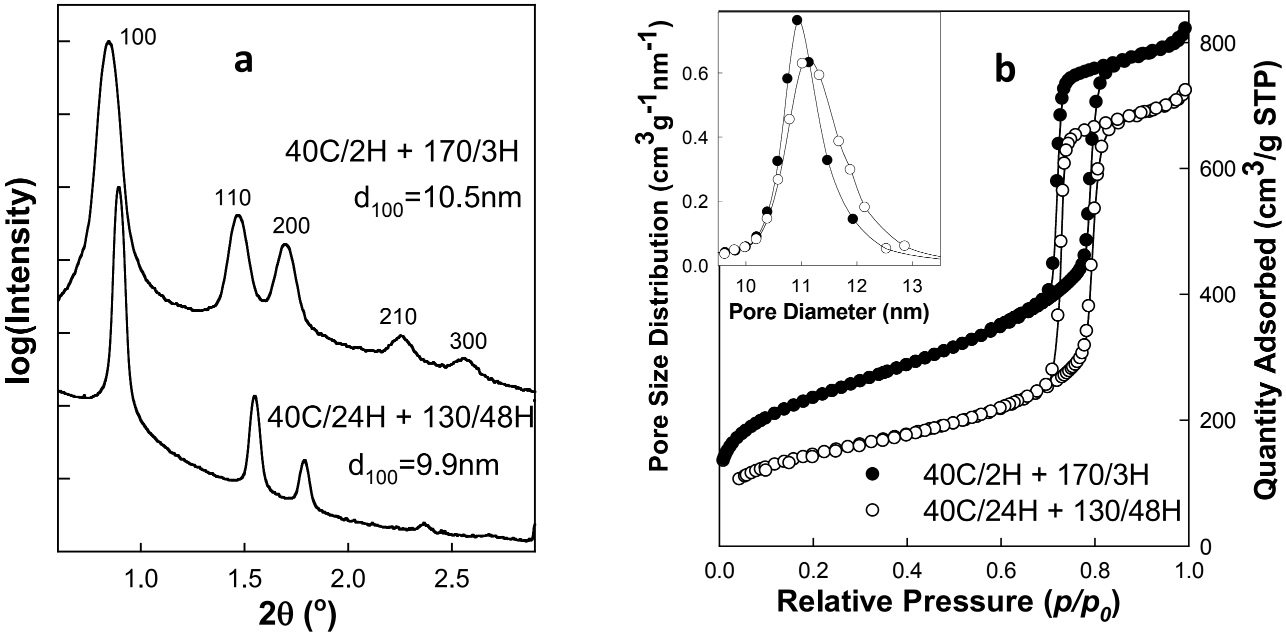
Supporting Figure S3. (a) SAXS pattern, (b) nitrogen adsorption isotherm and (c) pore size distribution of a calcined SBA-15 sample prepared at an initial synthesis temperature of 12.5 °C and hydrothermally treated at 170 °C for 3 hours.



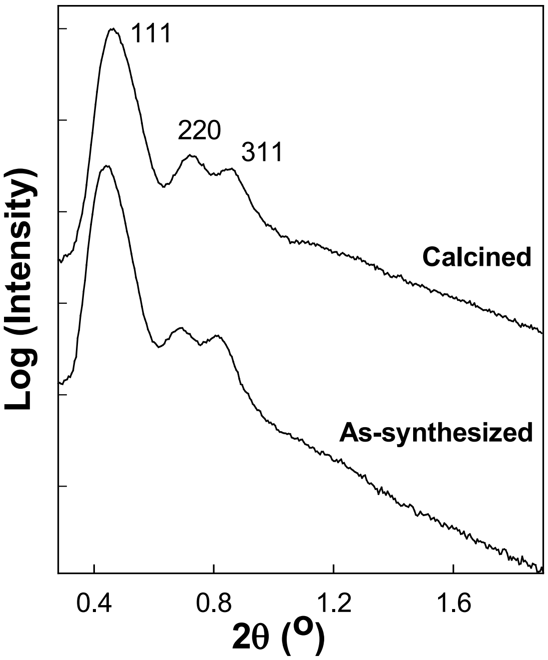
Supporting Figure S4. (a) SAXS pattern, (b) nitrogen adsorption isotherm and (c) pore size distribution of a calcined SBA-15 sample prepared at an initial synthesis temperature of 12.25 ^{o}C and hydrothermally treated at 170 ^{o}C for 3 hours.



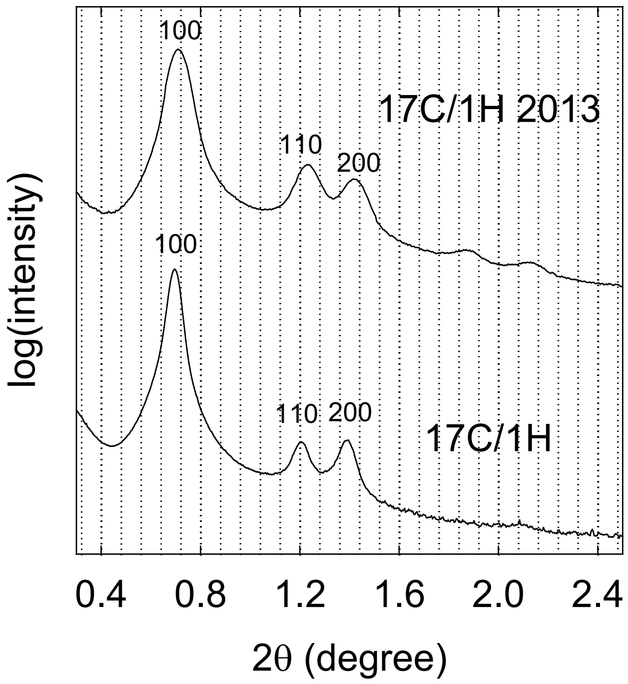
Supporting Figure S5. (a) SAXS patterns and (b) nitrogen adsorption isotherm (and inset with pore size distributions) of LP-SBA-15 silica after a shortened and standard calcination. LP-SBA-15 samples were synthesized with the condition of 17C/1H+130C/3H, 1 g TIPB per 2.4 g Pluronic P123. SAXS patterns were offset vertically to facilitate comparison. Figures taken from L. Cao, Ph.D. Dissertation, Graduate Center, City University of New York, 2010

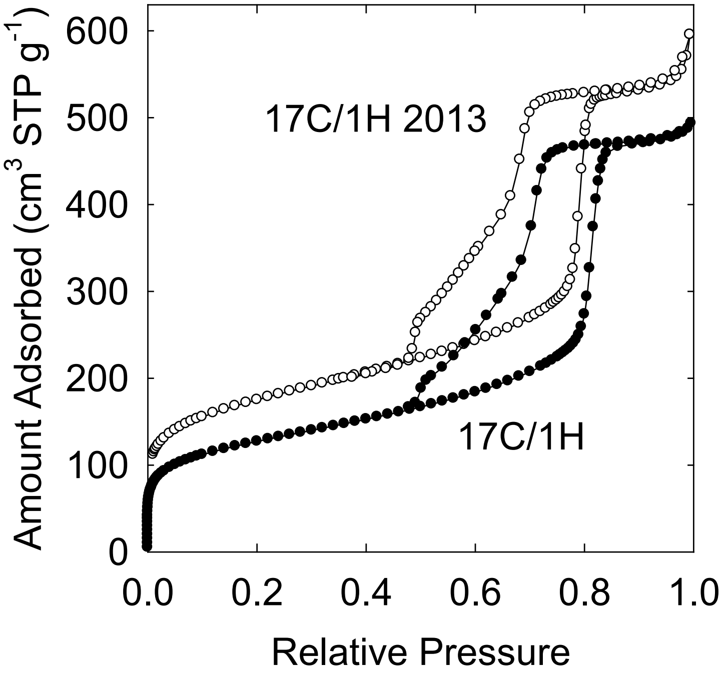


Supporting Figure S6. (a) SAXS patterns, and (b) nitrogen isotherms (and inset with pore size distributions) of calcined SBA-15 prepared via a short synthesis and a standard synthesis. The SAXS pattern for SBA-15 synthesized using standard synthesis was acquired in a synchrotron facility CHESS at Cornell University and because of that, the peaks are more narrow. The SAXS patterns were offset vertically to facilitate comparison. Figure 6(b) taken from L. Cao, Ph.D. Dissertation, Graduate Center, City University of New York, 2010.

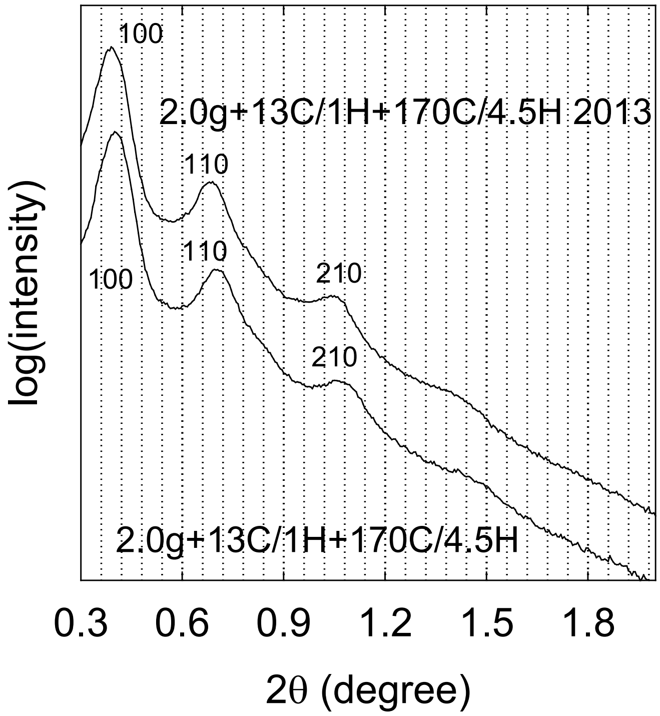


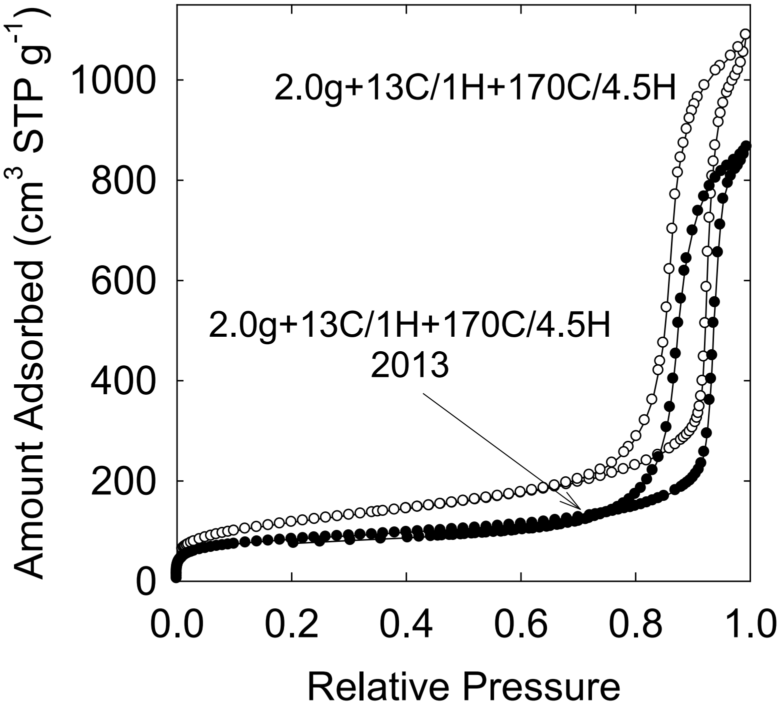
Supporting Figure S7. SAXS patterns of as-synthesized and calcined LP-FDU-12 prepared via a short synthesis. Figure taken from L. Cao, Ph.D. Dissertation, Graduate Center, City University of New York, 2010





Supporting Figure S8. (top) SAXS patterns of calcined sample prepared at an initial temperature of 17 °C. Bottom pattern (shown also in Supporting Figure S2) was acquired at a synchrotron facility CHESS at Cornell University and because of that, the peaks are more narrow. The other pattern was acquired after storage in the calcined form for 5 ½ years, using Bruker Nanostar U SAXS/WAXS instrument. Grid lines are included to allow for an easier comparison of the peak positions. (bottom) Nitrogen adsorption isotherms for this sample, collected initially and after 4 years of storage.





Supporting Figure S9. (top) SAXS patterns of calcined sample prepared at an initial temperature of 13 °C with subsequent hydrothermal treatment at 170 °C for 4.5 h. One pattern was shown in Figure 1. The pattern denoted “2013” was acquired after storage in the calcined form for 4 years. Grid lines are included to allow for an easier comparison of the peak positions. (bottom) Nitrogen adsorption isotherms for this sample, collected initially and after 4 years of storage.