## **Supporting Information**

## Synthesis of Ultra-large-pore FDU-12 Silica with Face-centered Cubic Structure

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**Supporting Figure S1.** SAXS pattern acquired using synchrotron X-ray source for ULP-FDU-12 silica.



**Supporting Figure S2.** Comparison of SAXS patterns, nitrogen adsorption isotherms and pore size distributions for LP-FDU-12 silicas synthesized (without the acid treatment) using TMB and xylene as a micelle expander.



Supporting Figure S3. SAXS pattern of FDU-12 synthesized with different amounts of xylene.



**Supporting Figure S4.** N<sub>2</sub> adsorption isotherms and pore size distributions for FDU-12 silica synthesized with different amounts of xylene.



Supporting Figure S5. SAXS pattern for EH5 samples.



Supporting Figure S6. N<sub>2</sub> adsorption isotherms and pore size distributions for EH5 samples.

Sample	Mass of TMOS (g)	Stirring speed (rpm)	Mass of xylenes (g)	Unit cell parameter							
				a <sub>as</sub> (nm)	a <sub>c</sub> (nm)	a <sub>c</sub> /a <sub>as</sub>	w <sub>KJS</sub> (nm)	$S_{BET}$ (m <sup>2</sup> /g)	$V_t$ (cm <sup>3</sup> /g)	V <sub>mi</sub> (cm <sup>3</sup> /g)	w <sub>KJS</sub> (nm)
MH1	3.3	350	4.5	50.8	42.2	0.83	21.8	211.0	0.34		
MH1-A- 130C-2d				51.8	49.9	0.96	26.6	297.1	0.85	0.011	33.5
MH1-A- 130C-4d				52.1	50.4	0.97	28.4	253.4	0.85	0.005	33.9
MH2	3.7	300	4.1	52.8	44.2	0.84	22.0	231.5	0.30		
MH2-A- 130C-1d				51.8	49.5	0.96	27.0	424.6	0.83	0.051	32.5
MH2-A- 130C-2d				52.1	51.1	0.98	27.2	317.0	0.83	0.014	34.1
MH2-A- 130C-4d				52.6	51.3	0.98	29.1	255.6	0.86	0.005	34.6

Supporting Table S1. Structural parameters for FDU-12 samples prepared from TMOS.



Supporting Figure S7. SAXS pattern for MH2 samples



Supporting Figure S8. N<sub>2</sub> adsorption isotherms and pore size distributions for MH2 samples



**Supporting Figure S9.** Pore volume vs. unit-cell volume for E4' sample calcined at different temperatures.

Regression function:  $y = 1.41 \times 10^{-5} x - 0.539$