

Electronic supplementary information (ESI)

A hybrid mesoporous material with uniform distribution of carboxy groups assembled from a cubic siloxane-based precursor

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Spectroscopic data for 1

Selected data for **1**: ^1H NMR (500 MHz, CDCl_3): δ (ppm) 0.86–0.90 (t, 3H; CH_3), 1.04–1.08 (2H), 1.23–1.32 (m, 47H), 1.59–1.65 (2H), 2.39–2.44 (2H), 3.86–3.93 (m, 14H; SiOCH_2), 4.05–4.08 (t, 2H; COCH_2); ^{13}C NMR (125.7 MHz, CDCl_3): δ (ppm) 6.67, 14.12, 17.80, 17.81, 22.74, 26.00, 27.48, 28.71, 29.34, 29.42, 29.61, 29.65, 29.71, 29.75, 31.99, 59.92, 59.93, 59.95, 64.81, 173.81; ^{29}Si NMR (99.3 MHz, CDCl_3): δ (ppm) –65.26 (T^3), –102.65, –102.75, –102.80 (Q^3); MS (FAB): m/z : 1029.18 [$\text{M}+\text{H}]^+$.

Characterization: Solid-state ^{29}Si magic angle spinning (MAS)NMR spectra were recorded on a JEOL JNM-CMX-400 spectrometer at a resonance frequency of 79.42 MHz with a 45° pulse and a recycle delay of 100 s. Solid-state ^{13}C cross-polarization (CP)/MAS NMR spectra were recorded on the same spectrometer at a resonance frequency of 100.40 MHz at a resonance frequency of 100.53 MHz with a contact time of 1.5 ms and a recycle delay of 5 s. For solid-state NMR measurements, the samples were put into 5 mm zirconia rotors and spun at 5 kHz. Chemical shifts for both ^{29}Si

and ^{13}C NMR were referenced to tetramethylsilane at 0 ppm. The XRD patterns of the products were obtained on a Mac Science M03XHF22 diffractometer with Mn-filtered Fe K α radiation. Transmission electron microscopy (TEM) observations were carried out on a JEOL JEM-2010 microscope operated at 200 kV. Nitrogen adsorption measurements were performed by an Autosorb-1 instrument (Quantachrome Instruments, Inc) at 77 K. The Brunauer–Emmett–Teller (BET) surface area was calculated from the adsorption branch in the relative pressure range from 0.02 to 0.05. The pore size distribution was evaluated using the non-local density functional theory (NLDFT) equilibrium model (N_2 at 77 K on silica, cylindrical pore).

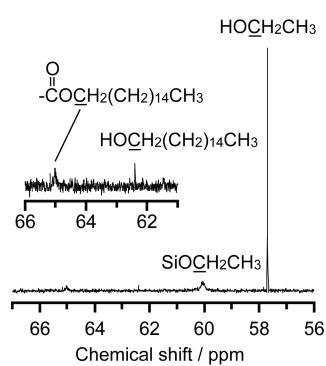


Fig. S1 Liquid-state ^{13}C NMR spectrum of the hydrolyzed solution of **1** after the reaction for 3 days.

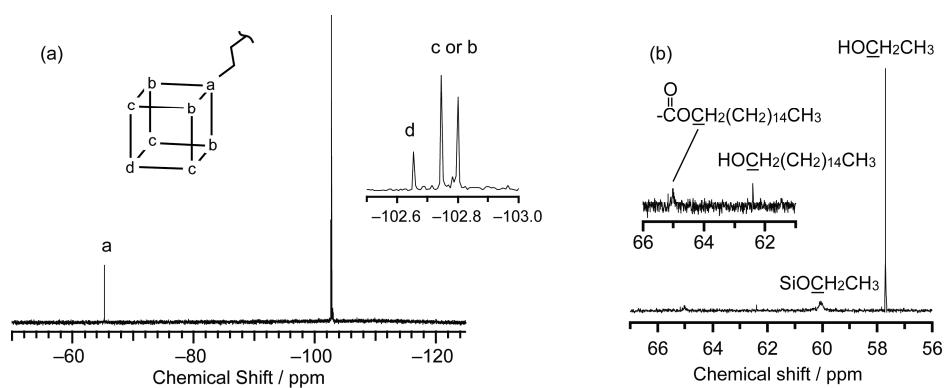


Fig. S2 Liquid-state ^{29}Si NMR spectra of (a) precursor **1** in CDCl_3 and (b) the hydrolyzed solution of **1** after the reaction for 3 days. The broadened signals after hydrolysis should be due to the presence of various partially hydrolyzed species with different numbers and positions of residual SiOEt groups. These spectra were recorded on a JEOL Lambda-500 spectrometer at a resonance frequency of 99.05 MHz with a pulse width of 6.5 μs , and a recycle delay of 30 s

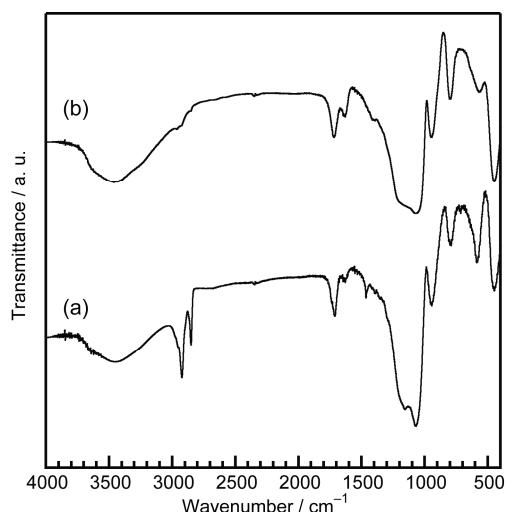


Fig. S3 FT-IR spectra of **1H** (a) before and (b) after the acid treatment. The spectra were obtained by the KBr disc technique using a Perkin Elmer Spectrum One spectrometer.

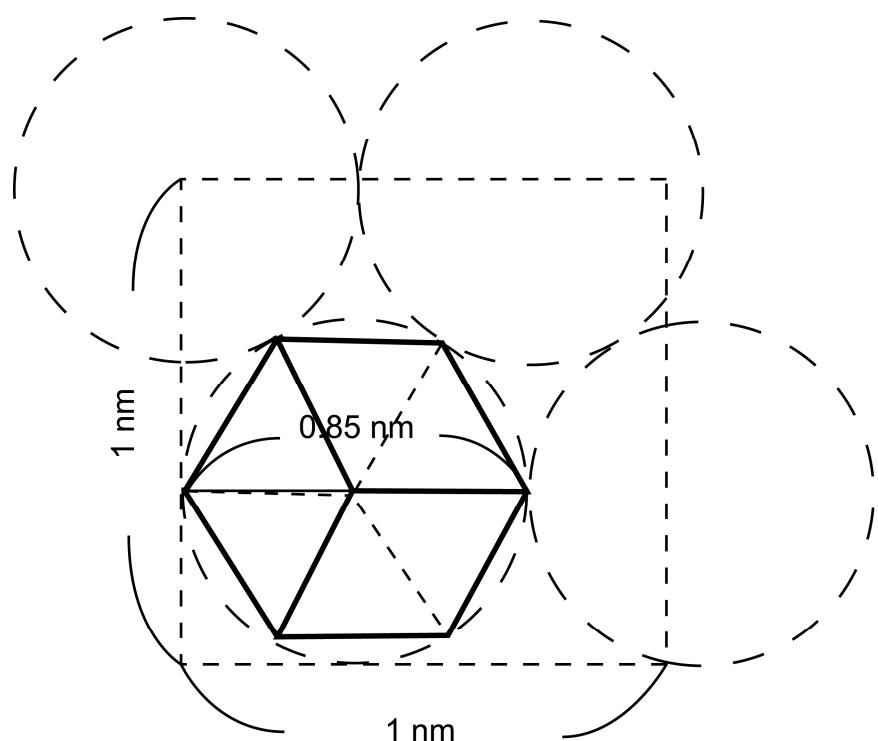


Fig. S4 Possible configuration of cubic siloxane cages on the mesopore channels.
(The circles with dotted lines show a hexagonal arrangement. The square with dotted lines shows the scale of 1 nm.)