Electronic Supplementary Information

Synthesis of zeolites using highly amphiphilic cations as organic structure-directing agents by hydrothermal treatment of dense silicate gel

Takahiko Moteki, Sye Hoe Keoh, Tatsuya Okubo

Experimental

Zeolite crystals were synthesized by the conventional hydrothermal method. The series of amphiphilic cations were added to the reactant mixture in bromide forms, with sodium hydroxide used as mineralizer. The final chemical composition was adjusted to SiO₂:0.125Na₂O:0.025SDA:17.5H₂O. Briefly, sodium hydroxide (Tokyo Kasei) was dissolved in distilled water, followed by the addition of the SDA (Tokyo Kasei). Then, fumed silica (Cab-O-sil M5, Cabot) was added to the mixture and mixed by hand with mortar and pestle. The homogenized mixture was transferred into a 23 ml Teflon©-lined autoclave (#4749, Parr) and subjected to the hydrothermal treatment at 333 K for 3 d under static condition. The obtained products were filtered, and washed with distilled water, then dried at 353 K for 12 h in an oven.

Characterization

Powder X-ray diffraction (XRD) patterns were collected on an Ultima IV (Rigaku) using CuKa radiation (40 kV, 40 mA) at a scanning rate of 4 degree/min over a range of 5 to 40 degree for high-angle measurement and at a scanning rate of 0.05 degree/min over a range of 1 to 5 degree for low angle measurement (2 theta). Thermogravimetric (TG) analysis

was performed on a ThermoPlus (Rigaku) with heating rate of 5 K/min using a mixture of 10% O₂ and 90% He as a combustion gas. Size and morphology of the samples were observed on a field-emission scanning electron microscope S-4800 (Hitachi).

Figures

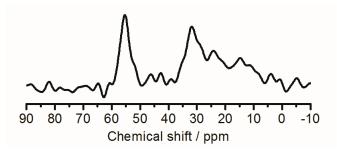


Figure S1. Solid-state 13-carbon MAS NMR spectrum of as-made silicalite-1 crystal synthesized with cetyltrimethylammonium cation.

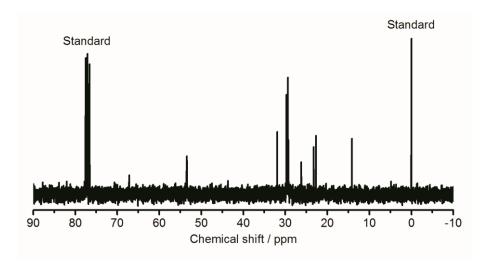


Figure S2. Liquid-state 13-carbon MAS NMR spectrum of cetyltrimethylammonium bromide solution. The signals at 0 and 77 ppm are from TMS and CDCl₃, respectively.

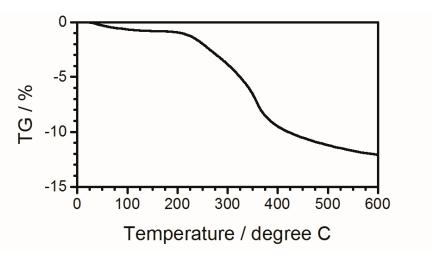


Figure S3. TG curve of silicalite-1 zeolite synthesized with cetyltrimethylammonium cation.

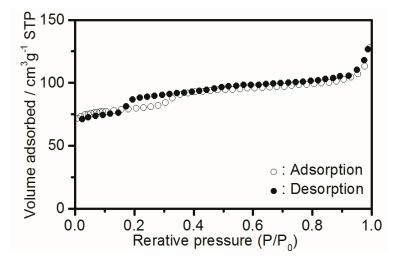


Figure S4. Nitrogen adsorption—desorption isotherm for calcined silicalite-1 crystal synthesized with cetyltrimethylammonium cation. Open and filled symbols indicate adsorption and desorption points, respectively.

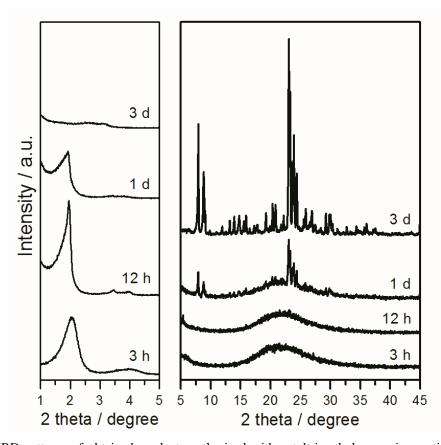


Figure S5. XRD patterns of obtained product synthesized with cetyltrimethylammonium cation for various period of time.