Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supplementary information

Direct Synthesis of Tetraalkoxysilane from Silica and Alcohol

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Table of Contents **Page**

1. General Remarks

Silica (CARiACT Q-10) used as a starting material was supplied by Fuji Silysia Chemical Ltd. The surface area (BET), the average pore diameter (BJH), and pore volume ($P/P_0 = 0.99$) were 261 m^2/g, 24.5 nm, and 1.19 cm^3/g, respectively. The silica was dried at 300 °C for 10 h prior to use. Rice hull ash as a natural silica source was prepared by calcination of rice hull at 500–1000 °C for 6 h. A photograph of RHA-500 was shown in Figure S3. The quartz powder and the Celite 209 purchased from Sigma-Aldrich Japan Co. (Tokyo, Japan). The SiO_2 content in RHA was 91–93%, determined by energy-dispersive X-ray fluorescence analysis. The molecular sieves 3A, 4A, 5A and 13X (2mm beads) were purchased from Merck Co. (Darmstadt, Germany), and dried at 300 °C for 10 h prior to use. All chemicals were reagent grade and were used without further purification.

Isothermal nitrogen adsorption/desorption measurement of SiO_2 was carried out at -196 ºC on a BEL Japan BELSORP mini II. The reaction products were analyzed by gas chromatography (GC) and Gas chromatography–mass spectrometry (GC-MS). All volatile products were characterized by GC-MS using a Shimadzu GC-2010 gas chromatography connected to a GCMS-QP 2010 plus mass spectrometer. The yield of products were determined by GC using a Shimadzu GC-2014 equipped with a flame ionization detector.

2. Typical reaction procedure

Two 10 ml of stainless steel autoclaves were connected via a 1/4-inch stainless steel joint. The upper autoclave was filled by 8 g of molecular sieve 3A (2 mm beads). SiO_2 (0.09 g, 1.5 mmol), KOH (8.5 mg, 0.15 mmol) EtOH (8 g) were added to a bottom autoclave at room temperature under a N_2 atmosphere. The bottom autoclave was heated at 260 °C for 6 h. After cooling to room temperature, *p-tert*-butyltoluene (68 mg) was added to the reaction mixture as an internal standard to determine the yield of products using GC.

Figure S1. Photograph of RHA-500 used as a starting material.