

Mesoporous silica MCM-41 as highly active, recoverable and reusable catalyst for direct amidation of fatty acids and long-chain amines

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Experimental

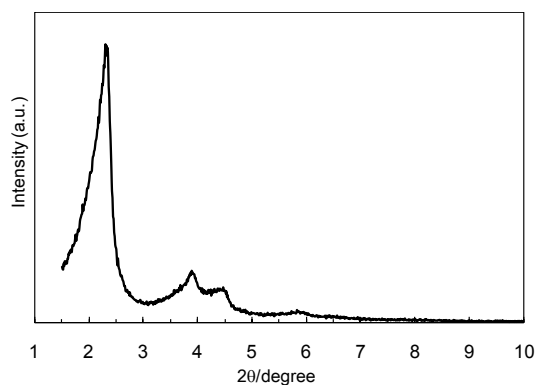
Materials: Mesoporous silicas, MCM-41 and aluminosilicate [Al]-MCM-41 were synthesized in our laboratory by reported procedure in literatures.¹ FSM-16 was gifted from Toyota Central R&D LABS., INC. H-Y zeolite (SiO₂/Al₂O₃ = 5.6, HSZ-320 HOA) was purchased from Tosoh Corporation, Japan. Ion-exchanged resin, Nafion[®] (NR50, 0.8 mmol H⁺/g) was purchased from Aldrich Japan. Mesoporous silica was calcined at 550 °C for 6 h prior to use, and all of organic reagents were commercial available and used without purification.

Measurements: Powder X-ray diffractograms were recorded on a XRD-6000 diffractometer with Cu K α radiation ($\lambda = 0.15418$ nm) (Shimadzu Corporation, Japan). Nitrogen adsorption isotherms were obtained at 77 K using a Belsorp 28SA apparatus (Bel Japan Inc., Japan). TG/DTA measurements were performed on a Shimadzu DTG-50 apparatus with temperature programmed rate of 10 °C/min in air stream. ¹H and ¹³C NMR spectra were measured by an ECA-500 FT-NMR (JEOL Ltd., Japan)

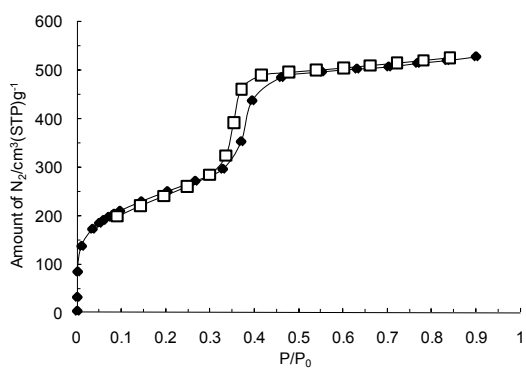
Preparation of MCM-41: A 25.1 g of hexadecyltrimethylammonium bromide (HDTMABr) was completely resolved in 77.0 g of distilled water at 50 °C (solution A). In a 300 mL beaker, a 28.1 g of sodium silicate (SiO₂: 29.3%, Na₂O: 9.3% in water) was added into an aqueous H₂SO₄ solution (0.31 mmol g⁻¹), then was added into the solution A. After stirring for 1 h at ambient temperature, the pH of the solution was adjusted to 9.98 by adding H₂SO₄ solution. The resulting white suspension was transferred into a 500 mL of bottle (polypropylene), and stood at 100 °C for 9 days. The obtained white powder was washed with distilled water and EtOH thoroughly and dried at 50 °C for overnight. The calcination of as-synthesized MCM-41 was taken place at 550 °C for 6 h.

1. H. -P. Lin, S. Cheng, C. -Y. Mou, *Micropor. Mater.*, 1997, **10**, 111-121.

Powder-XRD chart of MCM-41



Nitrogen isotherm of MCM-41



BJH plot of MCM-41

