Supplementary Material (ESI) for Chemical Communications

Electronic Supplementary Information for:

Chiral enhancement in diethyl malonate addition by morphosynthesized l-proline mesoporous silica

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1. Experimental

1.1. General procedure for synthesizing proline mesoporous silica

In a typical synthesis, 10.0 g of amphiphilic surfactant, P123 was dissolved in 256.0 g of distilled water at room temperature followed by addition of sodium metasilicate nonahydrate and proline precursor. To the clear solution, concentrated hydrochloric (37.6%) was quickly added into the solution. The composition of final mixture is (1+x)SiO2 : x proline precursor : 0.018P123 : 11HCL : 117.1H2O, with x = Proline precursor/(Proline precursor + SiO2) = 2.5%, 5.0% and 7.5%.. The resultant solution mixture was stirred at 40 C for 1 h. The reaction gel mixture was aged at 100 C for 2 h in microwave irradiation (300 W, 100%, CEM Mars 5) under static conditions. The solid product was filtered and washed with copious amount of water and ethanol separately. The surfactant was removed by using Soxhlet extraction method with ethanol for 24 h.

1.2. Catalysts characterization

The powder Xray powder diffraction (XRD) patterns were obtained on a Rigaku diffractometer using CuKα radiation (λ = 0.1547 nm). The diffractograms were recorded in the 2Θ range of 0.7-5 at the rate 0.1/min (40kV, 20mA). BET measurements including surface areas

and pore volumes were performed using a Micromeritics porosimeter (model ASAP-2400). The samples were degassed at 300° C for 3 hours. The pore size distribution (PSD) was calculated using the Barrett-Joyner-Halenda (BJH) formula. The scanning electron microscopic (SEM) images were collected using a JEOL 630-F microscope. Before the measurement, the samples were dispersed onto a steal plate surface and coated with Pt metal. Transmission electron microscopic (TEM) images were taken using a JEM-3011 instrument (JEOL) equipped with a slow-scan CCD camera operating at 300 keV. HPLC analysis was performed on Agilent Technologies (model 1200 series). All solvents were HPLC purity grade and used without further purification.

1.3. Typical procedure for dethyl malonate addition reaction

Diethyl malonate addition was carried out at atmospheric environment with 1 mmole of substrate and 1.8 mole % of catalyst using Pirex batch reactor in thermal reactor system (Eyela Chemi Station). The reaction was done in several condition of reaction system with variation on temperature and solvents. The aliquots of the reaction mixture were withdrawn and subjected to GC analysis (Agilent 6890N, HP-5 capillary column, FID detector). When catalysts were object of recyclability test, used catalyst were washed several times by ethanol before reused again.

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2. TGA analysis of proline functionalized mesoporous silica