

Supporting information

In-Situ Assembly of Layered Double Hydroxide Nano-crystallites within Silica Mesopores and Its High Solid Base Catalytic Activity

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Preparation of hydrothermally stable SBA-15:

A hydrothermally stable mesoporous material SBA-15 was synthesized using P123 (EO₂₀PO₇₀EO₂₀) as template in acidic conditions.¹² Zeolite precursor seeds,¹³ P123, hydrochloric acid, and deionized water were mixed to give a reaction mixture with a molar composition of 1 Al₂O₃ : 100 SiO₂ : 1.8 P123 : 10 TPAOH : 10 Na₂O : 590 HCl : 17,680 H₂O. The mixture was aged at 60 °C for 24 h and afterwards heated at 100 °C for 48 h. The resultant solid was filtered, extensively washed with deionized water, and then dried at 100 °C.

Preparation of SBA-CH₃:

SBA-15 (1.5 g) was dispersed in dry toluene (50 ml) at 70 °C under N₂ atmosphere and then (CH₃)SiCl (10 ml) was added whilst stirring. The mixture was stirred for 24 h, filtered, and the residue dried in vacuum at 90 °C for more than 8 h. After modification of the outer surface, the surfactant template was removed by solvent extraction in refluxing ethanol whilst stirring for 2 days.

Preparation of LDH crystallites confined in SBA-15:

SBA-CH₃ (1.5 g) was immersed in 100 ml 4 M Mg(NO₃)₂ and 2 M Al(NO₃)₃ mixed solution at room temperature. After stirring for 10 min, the sample was filtrated and calcined at 600 °C for 6h to get the oxide loaded SBA-15 (SBA-oxide). The resulting product was placed in an autoclave (50 ml in volum) with 40 ml deionized water. The reaction vessel was tightly capped after purging with nitrogen gas and then hydrothermally treated at 110 °C for 5 days.

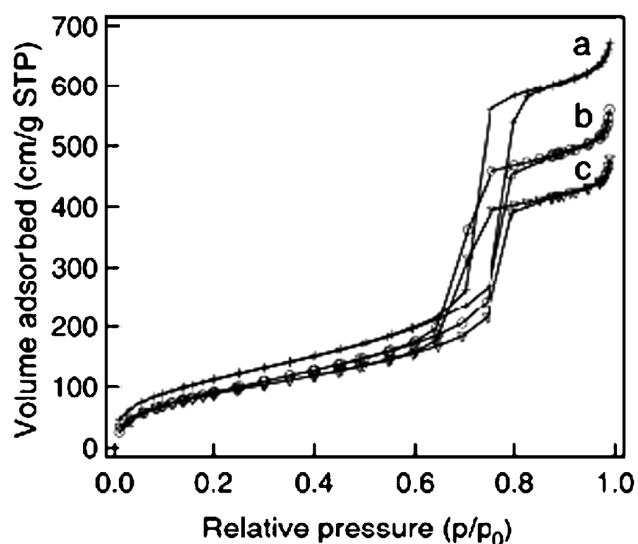
Catalytic testing

Aldol condensation reactions were preformed in a 100 ml round-bottom flask equipped with a magnetic stirrer and a condenser system in nitrogen under CO₂-free conditions. For the

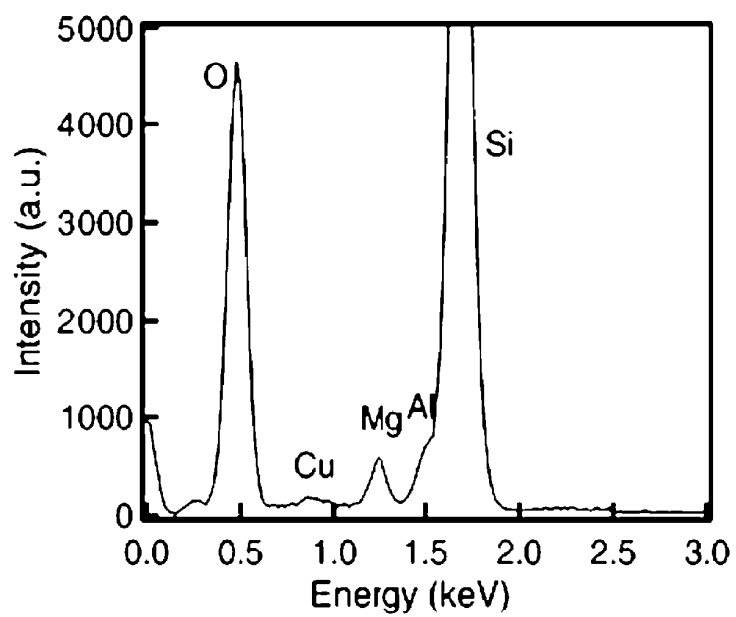
self-condensation of acetone, 0.75 mol acetone, 0.5 g of SBA-LDH catalyst were added and the reaction was conducted at 273 K. Samples were taken at regular time intervals and were analyzed off-line by GC-MS. A blank experiment (SBA-15 as catalyst) was also tested in the same condition.

Characterization:

The basic properties of the sample were measured by the adsorption and temperature-programmed desorption (TPD) of CO₂ with Thermo Finnigan TPDRO 1100. The samples were previously outgassed at in argon at 373 K for 1 h, and then cooled to 353 K. After absorbing of CO₂ (3 vol.% CO₂ in He; 20 cm³ STP min⁻¹) at this temperature for 60 min, the catalysts were treated in He (20 cm³ STP min⁻¹) for 45 min at 373 K to remove the physically adsorbed CO₂. The CO₂ uptake was measured by treating the sample from room temperature up to 1173 K at a heating rate of 10 K min⁻¹ and recording the results with a TCD detector. The number of Brønsted basic sites was determined assuming that one molecule of CO₂ adsorbs on each basic site.



Nitrogen adsorption-desorption isotherms of (a) SBA-CH₃,
(b) SBA-oxide and (c) SBA-LDH.



EDAX results of SBA-LDH corresponding to Fig. 3.