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A novel method for the synthesis of hierarchical ZSM-5 zeolite in proton form

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Synthesis of hierarchical HZSM-5 zeolite

To synthesize hierarchical ZSM-5 zeolites directly in the proton form, a mixture containing tetraethylortosilicate $Si(O\text{-}Et)_4$, (99%, SIGMA ALDRICH), distilled water, tetra-n-propylammonium hydroxide TPA-OH (1M, MACKLIN)), aluminum isopropoxide $Al(O\text{-}i\text{-}Pr)_3$ (>98%, TOKYO CHEMICAL INDUSTRY), glucose (RUSHIM) was used. The molar ratio of reagents $Si(O\text{-}Et)_4$: H_2O : TPA-OH : $Al(O\text{-}i\text{-}Pr)_3$: glucose was 1 : 9.8 : 0.14 : 0.007 : 0.065.

Tetra-n-propylammonium hydroxide and tetraethylortosilicate were dissolved in distilled water and intensively stirred at 70 °C for 5 h. Then aluminum isopropoxide and glucose were added to the resulting gel and stirred until the mixture was completely homogenized, but without subsequent aging. The resulting suspension was placed in a Teflon autoclave and subjected to microwave treatment in an M6 system (Preekem Instruments, China). Crystallization was carried out at 200 °C for 2 h. The obtained particles of hierarchical zeolite, designated as H-ZSM-5-G, were isolated by centrifugation in an ultracentrifuge at 1800 rpm, washed with distilled water and dried at 170 °C for 2 h, then they were calcined at 550 °C for 6 h to remove the organic template.

For comparison, HZSM-5 zeolite was synthesized with the same molar ratio of reagents without addition of glucose.

Material characterization. Powder X-ray diffraction (XRD) data were collected using a Haoyuan DX 27MINI (Cu – K_α radiation, λ = 1.54187 Å) diffractometer operated at 40 kV and 12 mA.

The shape and size of the particles, as well as the structure of the synthesized zeolite, were determined with a Hitachi Regulus8230 field-emission scanning electron microscope (FE-SEM), operating voltage 5 kV and a scanning electron microscope NEWTONS NT3200 with a tungsten cathode, operating voltage 10 kV, and transmission (JEOL JEM-2100, operating voltage 200 kV) electron microscopy.

The textural properties of the zeolite were determined by a low-temperature N_2 physisorption on a Micromeritics Instrument Corporation ASAP 2020 Plus system. The specific surface area was calculated by the BET model. The total pore volume was calculated at $p/p_0 = 0.99$ from the adsorption branch of the isotherm. The micropore volume was calculated by the t-plot method. The mesopore size distribution, volume of mesopores were determined according to the Barrett–Joyner–Halenda (BJH) desorption curve.

The acidic properties of the zeolites were evaluated by temperature programmed desorption of ammonia (NH_3-TPD) using an USGA-101 chemisorption analyzer. Each sample was pretreated at 550 °C in dry air for 4 h, then at 550 °C in N_2 for 1 h. After that the sample was cooled to room temperature and exposed to an N_2–NH_3 flow (1 : 1 ratio) for 30 min. Subsequently, the physically adsorbed NH_3 on the sample was purged by N_2 at 100 °C. The signal of NH_3 desorption was recorded in the temperature range up to 650 °C with a heating rate of 8 °C min^{-1}.