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Ultrafast microwave-assisted synthesis of nanosized HZSM-5 zeolite

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Synthesis of nanosized ZSM-5 zeolites with a crystallization time of 1-3 minutes.

To synthesize nanosized ZSM-5 zeolites directly in the proton form, a mixture containing tetraethyl orthosilicate $Si(OEt)_4$ (99%, Sigma-Aldrich), distilled water, tetra-*n*-propylammonium hydroxide TPAOH (1 M, Macklin) and aluminum isopropoxide $Al(OPr^i)_3$ (>98%, Tokyo Chemical Industry) was used. The molar ratio of reagents $Si(OEt)_4$: H_2O: TPAOH: $Al(OPr^i)_3$ was 1:4.8:0.19:0.007. Calculated SiO_2/Al_2O_3 ratio was 278.

Tetra-*n*-propylammonium hydroxide and tetraethyl orthosilicate were dissolved in distilled water and intensively stirred at 65 °C for 5.5 hours. Then aluminum isopropoxide was 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 for 1 and 3 minutes at 200 °C. The obtained particles of nanosized zeolite, designated as H-ZSM-5@1 and H-ZSM-5@3, were isolated by centrifugation in an ultracentrifuge at 1800 rpm, washed with distilled water and dried at 170 °C for 2 hours, and then they were calcined at 550 °C for 6 hours to remove the organic template.

Material characterization. Powder X-ray diffraction (XRD) data were collected using a Thermo Fisher Scientific ARL X' TRA (Cu K_α radiation, λ = 1.54187 Å) diffractometer in the range of 2θ angles from 5 to 50°.

The shape and size of the particles, as well as the structure of the synthesized zeolite, were determined by scanning (Newtons NT3200 with tungsten cathode, operating voltage 15 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 using a Belsorp Mini-X (MicrotracBEL, Japan) instrument. 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 MP method. Mesopore size distribution, volume of mesopores were determined according to the Barrett–Joyner–Halenda (BJH) method from the desorption curve; micropore size distribution was obtained according to the MP method from the adsorption curve; determination of the external surface area was carried out according to the *t*-plot method using the adsorption curve.

The acidic properties of the catalysts were evaluated by temperature programmed desorption of ammonia (NH_3-TPD) using a USGA-101 chemisorption analyzer. Each sample was pretreated at 550 °C for 4 hours in dry air and then for 1 h at 550 °C in N_2. 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}.