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A novel method for a nanosized ZSM-5 (MFI) zeolite synthesis in proton form

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Material characterization. Powder X-ray diffraction (XRD) data were collected at a scanning speed of 1.4 °/min on a Rigaku MiniFlex 600 (CuK_α radiation, λ = 1.54187 Å) diffractometer operated at 40 kV and 20 mA.

The shape and size of the particles, as well as the structure of the synthesized zeolite, were determined by scanning (CarlZeiss NVision 40, operating voltage 40 kV) and transmission (JEOL JEM-2100, operating voltage 200 kV) electron microscopy. The particle size distribution in the synthesized zeolite was calculated from the data for 360 particles in several TEM images.

The atomic force microscopy (AFM) was used to study the morphology of individual particles of synthesized nanosized HMFI zeolite. A Smart SPM scanning probe microscope (Horiba, France) with NSG30 ‘Golden’ Silicon cantilevers (TipsNano, Switzerland) was used in the in semi-contact mode. The peak-to-peak oscillation amplitude of the cantilever away from the sample surface was 20–25 nm. The tip radius of the probe was 5–10 nm (manufacturer's data). Before analysis, the zeolite powder was pressed, and the resulting tablet was fixed on the holder with a double-sided adhesive tape. Preliminary optical control of the surface was performed. The several the most homogeneous areas were analyzed. The scan size varied from 2.5 μm x 2.5 μm to 0.3 μm x 0.3 μm.

The elemental composition of zeolite was determined on Thermo ARL Perform'x Sequential XFR X-ray fluorescence spectrometer.

The textural properties of the zeolite were determined by a low-temperature N_2 physisorption on an 'AUTOSORB-1C' (Quantachrome Instruments, USA) 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 t-plot method. The pore size distribution was calculated from the adsorption isotherm using the NLDFT method in the approximation of a cylindrical pore shape.

The degree of incorporation of aluminum in the zeolite frame was determined by ^{27}Al MAS-NMR spectra [(Infinity UNOVA 500 NMR spectrometer (Varian inc., frequency 130 MHz)].

The acidic properties of the catalysts were evaluated by temperature programmed desorption of ammonia (NH_3-TPD) on a USGA-101M chemisorption analyzer. Each sample was pretreated at 550°C at 30 min in a He flow of 30 ml min^{-1}. After that the sample was cooled to 60°C and was saturated with 10 vol.% He-NH_3/He for 30 min. Subsequently, the physically adsorbed NH_3 on the sample was purged by He 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.