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Synthesis and acid-base properties of Mg-saponite

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Experimental

XRD patterns were recorded with a DRON 3M diffractometer using CuKα radiation in the Bragg–Brentano reflecting and Debye–Scherrer transmission geometry (λ = 1.54 Å). Diffractograms were collected at room temperature over the range of 5–70°(2θ) with a step size of 0.02° and a count time of 0.6 s, and a scan speed of 2° per second. The XRD pattern of the Mg-saponite sample was compared with the JCPDS database of standard compounds (International Centre for Diffraction Data, 1999).

Diffuse reflectance infrared Fourier transform spectra (DRIFTS) was recorded at room temperature with a Nicolet 460 Protégé spectrometer equipped with a diffuse reflectance attachment. The sample was placed in an ampoule supplied with a KBr window. Before IR spectra collection, the sample was treated in a vacuum at 350 °C (heat rate 5.5 K min^{-1}) for 2 h. To obtain a satisfactory signal-to-noise ratio, 500 scans were collected per spectrum. The spectra were measured from 400 to 6000 cm^{-1} with a resolution of 4 cm^{-1}. The probe molecules for acid sites (CD_3CN) and for basic sites (CCl_3D) were adsorbed at room temperature and saturated vapor pressures of 96 and 140 Torr, respectively.

Toluene thermodesorption experiments were carried out in a flow system by heating the sample up to 350–450 °C with a heating rate of 10 K min^{-1} using He as a carrier gas. The helium flow (40 ml min^{-1}) passed through the zeolite trap for water removal and then *via* the gas supply system was introduced to a quartz reactor containing 100 mg of the sample. The helium flow rate was monitored by the gas system supplier and measured at the outlet by a flow meter. The temperature measured by a chromel-alumel thermocouple contacted with the sample surface. Toluene was placed into the evaporator by a syringe and its concentration was measured by means of a thermo-conductivity detector (TCD) connected to the reactor outlet.

Prior to carry out the toluene adsorption, the sample was heated in a He flow (40 ml min^{-1}) at 300–450 °C for 1 h and cooled down to 50 °C. Then the sample was saturated with toluene by manifold injection system. It was then purged with pure He for 30–50 min and simultaneously cooled to room temperature.