Calorimetric study of the acidity of Y-zeolites treated with silicon tetrachloride


Introduction: The acidity of synthetic HY samples dealuminated with silicon tetrachloride vapor at various temperatures is investigated using microcalorimetry. The work covers sample preparation, characterization, and acidic properties. The differential heats of ammonia adsorption versus coverage and the acidity spectra are described. The catalytic performance is measured in the hydrogen transfer reaction.

Experimental
Dry ammonia was used as the molecular basic probe. Ammonia was adsorbed at 150°C to prevent physisorption. The zeolite sample in powder form, approximately 60mg in weight, was pretreated in a quartz cell, first with oxygen by heating at 2°C per minute and holding at 350°C for about 4 h and then under vacuum (1.33 mPa) at the desired temperature (350°C or 600°C) for about 2h. The pretreated sample was transferred to a Tian-Calvet microcalorimeter of the twin conduction type maintained at constant temperature (150°C).

Results
In this study, microcalorimetry was employed to measure the acid sites of a Y-zeolite quantitatively and also to determine their distribution. The gaseous probe (dry NH₃) was admitted to the zeolite sample in successive small doses of ~ 0.04 cm³, the gas pressure before expansion and at equilibrium being measured for every dose by a Datametrics Barocel gauge of maximum pressure 200Pa. Both the gas pressure and the thermogram were monitored graphically for every dose to determine the sorption equilibrium. The sorption process was followed both volumetrically and calorimetrically (with facilities for automation and computer processing) up to a final pressure of ca.150Pa. Data are shown on Fig. 1.

For more details ask for the publication B0618.