ON THE REACTION OF ALUMINOSILICATES WITH AQUEOUS SOLUTIONS OF NaOH AND KF

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Neutralization of H-mordenite with NaOH follows diffusion kinetics with the diffusion of sodium cations into the mordenite as rate-determining step; neutralization curves are determined by the $\text{H}_3\text{O}^+ \rightarrow \text{Na}^+$ ion exchange isotherm. With KF solutions, all aluminium can be extracted from the lattice of NaY-zeolite.

The reactions of aqueous solutions of NaOH and KF first described as a possible characterization method for amorphous aluminosilicates /1, 2/, are also proposed (and certainly much more reasonably as in the case of amorphous materials) for the characterization of zeolites. Breck and Skeels /3/ used the reaction with a KF solution for the determination of hydroxoaluminium cations in shallow bed calcined ammonium exchanged zeolite Y. Kühl /4/ titrated an H-mordenite in dilute NaCl solution with 0.1 N NaOH and obtained a sinoidal neutralization curve. In order to check both characterization methods, we examined the titration of HM with NaOH (the mordenite being especially suitable for this reaction on account of its high stability toward acids) as well as the reaction of NaY with aqueous solutions of KF.
In the neutralization experiments, H-mordenite (HM) with a SiO$_2$/Al$_2$O$_3$ mole ratio of 17 was used, which has been obtained by leaching with HCl a synthetic Na-mordenite. 1 g HM was slurried in 110 cm$^3$ of distilled water containing varying amounts of saturated NaCl solutions, and titrated with 0.1 N NaOH. The equilibrium pH and the pH as a function of time (t) were measured after each addition of NaOH using a glass electrode (OGA 50 N, Meinsberg). 1 g NaY (SiO$_2$/Al$_2$O$_3$ mole ratio 5.2) was slurried in 100 cm$^3$ 3 mol dm$^{-3}$ KF solution and after filtration the solution was titrated.

RESULTS AND DISCUSSION

The neutralization curves obtained are given in Fig. 1. With NaOH alone (curve 1, without NaCl addition) even at pH 7-8 some acidity of the mordenite remains unneutralized as is seen from a comparison with other titration curves, which were obtained in the presence of NaCl. The IR spectrum of the mordenite after filtration from a slurry of pH 7.6, still shows about 15% (relative to the starting HM) of the absorption of the OH vibration band at 3600 cm$^{-1}$, which is due to acidic OH groups. With increasing amounts of added NaCl, the amount of protons exchanged from the mordenite increases, giving rise to a shift of the titration curves. After all experiments, only negligible amounts of aluminium were found in the solution. Addition of KF to the mordenite slurry did not cause a basic reaction. This is to be expected in the presence of species like aluminium hydroxide within the mordenite. The HM thus selectively behaves as a Brønsted acid. The analysis of the set of data pH = f(t) which were obtained after each NaOH addition, revealed that the kinetics of this process in the initial step may be described by the well known diffusion law $q_t/q_\infty = 6/\pi^2 \frac{D}{\pi^2} t^{1/2}$ (r: radius of the particle). The quotient $q_t/q_\infty$ representing the exchanged amount at a given