Dealumination of zeolite Y by H_4EDTA: further comments

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Treatment with H_4EDTA is a well-established method of removing aluminium from zeolitic frameworks [1–4] which has been particularly useful for the structural modification of zeolite Y. The nature of the dealuminated product has been reported to depend on the rate of addition of H_4EDTA to the slurry of the zeolite in water [1,4]. According to this, when H_4EDTA is added slowly, the rate of removal of Al from zeolite is lower than the rate of migration of siliceous species (the nature of which is unclear) and the tetrahedral vacancies created by the extraction of Al are subsequently healed by silicon. By contrast, when H_4EDTA is added rapidly, the vacancies are not so healed and, when the amount of Al removed is large, the framework may collapse. This happens, for example, upon fast removal of 60% of Al atoms [1].

Our results [5] are in general agreement with those of Kerr et al. [1–4]. Our two samples were obtained by *fast* dealumination of zeolite Y (1 h treatment with a dilute aqueous solution of H_4EDTA), and 16 and 30% of Al was removed in samples 2 and 3 respectively. Most vacancies created by Al extraction were not healed. The existence of such vacancies is demonstrated by the fact that the intensity of the Si(0Al) peak in ^{29}Si magic-angle-spinning (MAS) NMR spectra upon dealumination is constant, and especially by ^{29}Si CP/MAS spectra in which the intensity of the Si(2Al, *n*OH) peaks increases. Further support for this conclusion comes from X-ray diffraction which reveals that the contraction of the unit cell upon dealumination is smaller than that reported by Kerr et al. [4], and from infrared spectra which show that nests of Si–OH groups have been created. At the same time, ^{27}Al MAS NMR detects no extra-framework Al. The only difference between our results and those of Kerr et al. is that in our case dealumination does not lead to an appreciable destruction of the zeolitic framework. This is demonstrated by X-ray diffraction, and is due to the fact that only small amounts of Al were extracted

(16 and 29% of Al atoms, equivalent to 4 and 7% of T atoms) as compared with 60% of Al atoms or 16% of T atoms in ref. [1]. Pichat et al. [6] report that 50% of Al must be removed to destroy the framework.

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