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Partially dealuminated heulandite produced by acidic $REECl_3$ solution: A chemical and single-crystal X-ray study

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ABSTRACT

Single crystals (0.1–0.5 mm) of natural heulandite from Nasik (India) were treated in 4 *M* NaCl solution at 423 K for 12 weeks, yielding almost fully Na-exchanged heulandite (composition: $Na_{8.44}Ca_{0.09}K_{0.01}[Al_{8.63}Si_{27.37}O_{72}]\cdot nH_2O$). This precursor phase was subsequently treated in a Teflon autoclave with 0.5 *M* rare-earth element (REE) solution (0.25 *M* $ErCl_3\cdot 6H_2O$ and 0.25 *M* $LaCl_3\cdot 7H_2O$; pH of 2.8) for 15 weeks also at 423 K. Er and La in the zeolite were subsequently measured by inductively coupled plasma (ICP) mass spectrometry yielding only 656 ppm Er and 195 ppm La, whereas electron-microprobe (EMP) analyses indicated that the Na concentration decreased from originally 8.44 Na pfu to 0.25 Na pfu. The low REE content may be explained by the relatively small free diameter of the channel windows and the large size of hydrated REE ions. The low Na concentration is caused by partial dealumination of the tetrahedral framework where Si replaced some Al of the framework and Al migrated into the structural channel. Dealumination or more generally dissolution phenomena on the crystal surface occurring due to the acidic milieu in the exchange solution were observed as etch pits on scanning-electron microscope (SEM) images.

X-ray single-crystal data of $REECl_3$-treated heulandite were collected at 100 and 293 K and at 100 K after partial dehydration at 323 and 378 K. Structure refinement using all data sets suggested the presence of low concentrations of octahedrally coordinated Al^{3+} that was dissolved from the framework and incorporated into the channels. T-O distances in the framework, corrected for rotational disorder, are significantly shortened compared with the Na-exchanged precursor heulandite. This indicates that $REECl_3$-treated heulandite has a significantly lower Al concentration in the framework than the Na-exchanged precursor phase.