ESRF	**Experiment title:** **In situ powder diffraction study of the thermal behaviour of large-porous aluminosilicate ECR-1**	**Experiment number**: **08-02-638**
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Names and affiliations of applicants (* indicates experimentalists):

Arletti Rossella, Department of Earth Sciences, university of Modena and Reggio Emilia

Ori Silvia: Department of Earth Sciences, university of Modena and Reggio Emilia

De Giorgi Raffaella: Department of Earth Sciences, university of Modena and Reggio Emilia

Report:

Introduction

Zeolites and related microporous solids have a huge industrial and commercial value and find widespread application in many technologies. [1-2]. Their advantages over other solids stem directly from their surface areas and structure-dependent pore geometry and cation environments. One of the most remarkable properties of zeolites is to dehydrate reversibly upon heating but, sometimes however this process is not always completely fulfilled, at least in short time and the dehydrates phase does not transform reversibly into the natural hydrated one, but one or more heat – induced phases are stable over along period of time. The zeolite dehydration process has been widely studied because the knowledge of the structural modifications induced by temperature and the definition of the stability field of these materials is of prime importance to assure their persistence and effectiveness in technological applications.

In may 2005, the Framework Type Code EON was approved by IZA Structure Commission. It is based on the framework-type material ECR-1, with an orthorhombic space group *Pmmn* and cell parameters a= 7.5709 Å, b= 18.1480 Å, and c=25.9324 Å. This phase was synthesized for the first time by Vaughan and Strohmaier in 1987 but the small size of the crystals obtained prevented a single crystal study [3,4]. Two structural frameworks (ECR-1A and ECR1A), deduced on the basis high-resolution transmission electron microscopy data, composed of different stacking of MOR and MAZ sheets were proposed [5]. The first structural Rietveld refinement on this zeolite, performed only in 2006 [6] confirmed the structure model – firstly indicated by Leonowicz and Vaughan [5].

The aim of this study is to follow *in situ* the dehydration mechanism of the zeolite ECR1, in its as-synthesized and NH_4 exchanged form in order to determine its thermal stability and whether the structure undergoes phase transition during dehydration.

Experiment

Two samples were studied: an as synthesized Na–ECR1 and an NH_4- exchanged one. The temperature-resolved XRPD experiments were performed on powdered samples packed inside a 0.5 mm capillary. Data was acquired in parallel Debye-Scherrer geometry, with LaB_6 refined wavelength of 0.88849 Å; the rotating capillary sample was heated in-situ by means of hot air stream equipment, with heating rate of 4.0 °C/min, from r*T* up to 925°C. The powder diffraction patterns were continuously collected, during the whole heating process, on the 4 mm slit-delimited slice of a translating imaging plate detector [7,8] located at a sample-to-detector distance of 270 mm. A total of 45 powder patterns were extracted by integrating temperature slices of 20 °C width. **Due to the bad quality of the data collected only cell parameter were refined.**

Results

Both the studies samples show a strong thermal stability: Na- ECR-1 undergoes complete decomposition only at 780°C while the NH_4 exchanged form is stable at least up to 925 °C, as attested by the persistence of the diffraction peaks even at the higher temperatures. No phase transition occurs during the dehydration process.

Only small variation in the unit cell parameters are observed. For the Na-as synthesized phase, the final values obtained after the refinement at 715 °C accounted a variation of -0.25, +0.07, -0.77 and -0.95% for *a*, *b*, *c* and V respectively. For the NH_4 exchanged ECR1 is quite different the refinement performed at 925° show to final variations of -1.33%, -0.88%, -1.68% and -3.82% for *a*, *b*, *c*, and V respectively.

Future Perspectives

Better quality date will be collected in next experiment to refine the structure parameters. On the basis of these results, it will be possible to understand in details the mechanism of dehydration of this large pore aluminosilicate.