Synthetic routes and crystallographic and thermal study of the microporous titanosilicate ETS10

E. Cazzini,[a,b] M. Catti,[a] and G. Ferraris[b]

[a]*Dipartimen.to di Scienza dei Materiali, Università di Milano Bicocca, Milano, Italy.* [b]*Dipartimento di Scienze Mineralogiche e Petrologiche, Università di Torino, Italy*

Keywords: **Titanosilicates, Structural disorder, Microporous materials**

Within the silicon-based microporous materials, titanosilicates are comparatively less studied systems characterized by a zeolite-like $[SiO_2]_\infty$ framework interwoven with $[TiO_3]_\infty$ chains, where Ti is octahedrally coordinated. We present here some results on ETS10, which has chemical formula $(Na_{1.5}K_{0.5})TiSi_5O_{13}.xH_2O$, and is characterized by a disordered mixture of two polymorphs with tetragonal and monoclinic symmetry [1], giving rise to a system of channels defined by 12-atoms rings. ETS10 is structurally related to another titanosilicate, ETS4, synthetic analogue of the mineral zorite. Samples of ETS10 were prepared according to the traditional method based on the $TiCl_3$ precursor, and also following a synthetic route employing TiO_2 as initial titanium source. In both cases, the synthesis was carried out in hydrothermal vessels at 230 °C, with carefully controlled pH and gel formation from the sodium silicate solution. The samples obtained were studied by SEM, TGA-DTA and X-ray powder diffraction techniques. The X-ray powder pattern is consistent with the simulation obtained by the DIFFAX programme [2], on the basis of a completely disordered stacking sequence of the monoclinic and tetragonal polymorphs. However, the sample prepared by the TiO_2 route is much better crystallized than the other one, according to diffraction intensities and peak widths. This is confirmed also by SEM images, which show crystallites with larger size in the former case. The thermal study revealed a weight loss of about 12% below 500 °C, corresponding to the dehydration process. A fraction of water was regained reversibly on cooling, so that a part of H_2O only appears to be strongly bonded inside the channels. On further heating, ETS10 decomposes at 750 °C into a mixture of phases, which were identified by X-ray diffraction to be quartz and narsarsukite ($NaTiSi_4O_{11}$).

[1] J. Rocha and M.W. Anderson, Eur. J. Inorg. Chem. 801 (2000)

[2] M.M. J. Treacy, J.M. Newsam and M.W. Deem, Proc. Roy. Soc. London A 433, 499 (1991)