Thermal Behaviour of tobermorite 11Å from Kalahari Manganese Field (Sud Africa)

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The term “tobermorite” indicates a group of minerals widely studied for their close relationship with C-S-H gel (Taylor, 1964, 1992, 1997), the principal binding agent formed during the hydration process of Portland cement; the technological importance of tobermorite is also related to its cation-exchange capacity, with potential application in the disposal of nuclear wastes (Komarneni & Roy, 1983; McCulloch et al., 1985).

The minerals of the tobermorite group vary in the content of molecular water, related to a different length of the c axis. As a function of the water content, it is possible to distinguish tobermorite 14Å, tobermorite 11Å, and tobermorite 9Å. In particular it was noted that the 14Å phase collapses to tobermorite 11Å at 80-100°C, while the latter shrinks to tobermorite 9Å at 300°C (McConnell, 1954); however, some specimens of the 11Å form do not collapse to the 9Å phase at 300°C and are called “anomalous”, whereas those tobermorites that shrink on heating are called “normal” (Mitsuda & Taylor, 1979).

The studied samples come from the Kalahari Manganese Field (South Africa) and were used by Merlino et al. (2001) to solve the crystal structure of tobermorite 11Å.

Its composition is Ca4Si6O15(OH)2.5H2O.

Thermogravimetric analysis was conducted from room temperature up to 1200°C. It showed two important steps of weight loss: a first, corresponding to a loss of about 10 wt.%, in the temperature range between 100°C and 300°C, and a second between 500°C and 600°C, with a loss of about 3 wt.%. In the DSC curve the first was associated to an endothermic peak, related to the loss of humidity and interlayer water.

The second one was correlated to an exothermic peak, attributed to the crystallization of a new phase. Finally, a broad exothermic band at 1000°C was observed. The products of this process were studied by XRD; the product formed at 500°C is a new phase with a periodicity of 10Å (here called tobermorite 10Å). At 1000°C tobermorite 10Å converts into wollastonite.

The kinetics of this process was followed at the GILDA (General Italian Line for Diffraction and Absorption) of the European Synchrotron Facility (ESRF), Grenoble (France). The first formation of tobermorite 10Å was observed at 420°C, coexisting with tobermorite 11Å. Le Bail refinement of the crystallographic parameters was performed with GSAS (Larson & Von Dreele, 1994) and their variations were studied as a function of temperature.

In order to obtain single crystals of tobermorite 10Å suitable for diffraction studies, some single crystals of tobermorite 11Å were heated at different temperatures and different period of times, in the effort to verify the nature of the heating products. XRD studies upon these products allow us to observe a transition from tobermorite 11Å to tobermorite 10Å, passing through a clinotobermorite-like phase.

Weissenberg studies on tobermorite 10Å show an extensive disorder, with diffuse and streaked reflections along c*.

The family cell is orthorhombic, with a 5.57 b 3.67 c 21 Å; the systematic absences suggest an A-centred cell. Further investigations are required to unravel the crystal structure of this phase.

References: