

Structural study on the phase transformation of natural scolecite with increasing temperature

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Scolecite, $\text{CaAl}_2\text{Si}_3\text{O}_{10}\cdot 3\text{H}_2\text{O}$ is classified to fibrous zeolite group. The sequence of general phase transformation with increasing temperature has been reported for natural scolecite: scolecite \rightarrow meta-scolecite \rightarrow amorphous phase and decomposes to An + Qtz (Rykl *et al.* 1986; Gottardi and Galli 1985).

In this study, the high-T evolution of the structure of natural scolecite from Poona, India were studied up to 573 K to reevaluate the dehydration process of scolecite using TG-DTA and in situ single crystal X-ray diffraction experiments.

As the results from structural refinement at room temperature, the lattice constants of the sample are determined as follows: $a = 18.504(3)\text{Å}$, $b = 18.971(2)\text{Å}$, $c = 6.5262(9)\text{Å}$ and $\beta = 90.558(5)^\circ$. The crystal structure of scolecite, $\text{CaAl}_2\text{Si}_3\text{O}_{10}\cdot 3\text{H}_2\text{O}$, was refined with the space group $F1d1$ from 3567 reflections with $I_o > 2\sigma(I)$, yielding $R = 4.62\%$, $wR = 11.41\%$. At $\sim 423\text{ K}$, the space group was changed to $Fd11$ from $F1d1$, and scolecite underwent a phase transformation to meta-scolecite phase.

As the results from structural refinement at 523 K, the lattice constants of the sample are determined as follows: $a = 18.122(3)\text{Å}$, $b = 18.847(3)\text{Å}$, $c = 6.5408(11)\text{Å}$ and $\alpha = 88.948(7)^\circ$. The crystal structure of scolecite, $\text{CaAl}_2\text{Si}_3\text{O}_{10}\cdot 2\text{H}_2\text{O}$, was refined with the space group $Fd11$ from 2782 reflections with $I_o > 2\sigma(I)$, yielding $R = 10.72\%$, $wR = 28.85\%$. When phase transformation occurs, OH_2 in scolecite is expelled and then the half of Ca ions move by $\sim 1/2c$. At 573 K, the number of observed reflections was decreased dramatically.

Under high-T experiments from 423 to 523 K, two reciprocal lattices were observed, each lattice is corresponding to twin component with the [00-1] twin law. The [00-1] twinning could be associated with the dehydration mechanism. The X-ray diffraction data suggest the possibility of exist of a new H_2O site in meta-scolecite phase. This may be a key to solve the dehydration process of scolecite.

Keywords: scolecite, dehydration, phase transformation, single crystal X-ray diffraction, twin, high temperature