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Synchrotron powder X-ray diffraction study of the structure and dehydration behavior of sepiolite

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ABSTRACT

Rietveld refinements using synchrotron powder X-ray diffraction data were used to study the crystal structure and dehydration behavior of sepiolite from Durango, Mexico. The room-temperature (RT) sepiolite structure in air compares well with previous models but reveals an additional zeolitic H_2O site. The RT structure under vacuum retained only ~1/8 of the zeolitic H_2O and the volume decreased by 1.3%. Real-time, temperature-resolved synchrotron powder X-ray diffraction data and Rietveld refinements were used to investigate the behavior of the sepiolite structure from 300 to 925 K. Rietveld refinements revealed that most of the zeolitic H_2O is lost by ~390 K, accompanied by a decrease in the *a* and *c* unit-cell parameters. Above ~600 K the sepiolite structure folds as one-half of the crystallographically bound H_2O is lost. Rietveld refinements of the "anhydrous" sepiolite structure reveal that, in general, unit-cell parameters *a* and *b* and volume steadily decrease with increasing temperature; there is an obvious change in slope at ~820 K suggesting a phase transformation coinciding with the loss of the remaining bound H_2O molecule.

Keywords: Sepiolite, Rietveld, synchrotron, powder diffraction