

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

JEFFREY E. POST^{1,*} AND PETER J. HEANEY²

¹Department of Mineral Sciences, Smithsonian Institution, Washington, D.C. 20560-0119, U.S.A.

²Department of Geosciences, Pennsylvania State University, University Park, Pennsylvania 16802, U.S.A.

ABSTRACT

Rietveld refinements using synchrotron powder X-ray diffraction data were used to study the crystal structure and dehydration behavior of pure monoclinic palygorskite samples from Korea and Alaska. The 300 and 100 K palygorskite structures in air compare well with previous models but provide additional details about zeolitic H₂O sites and reveal that the Al atoms are ordered into the inner M2 octahedral sites and the Mg cations into the M3 sites at the edges of the tunnels. Real-time, temperature-resolved synchrotron powder X-ray diffraction data and Rietveld refinements were used to investigate the monoclinic palygorskite structure from 300 to 1400 K (in air). Rietveld refinements showed that most of the zeolitic H₂O is lost by ~425 K, accompanied by a decrease in the unit-cell volume of 1.3%, primarily owing to a decrease in the *a* unit-cell parameter and an increase in the β angle. The structurally bound H₂O is lost in two stages, at temperature intervals of 475–540 and 580–725 K. Above ~825 K in air a portion of the Korean sample transformed to a folded structure; the Alaskan sample folded at ~575 K under vacuum. A structure model was refined for the folded structure. At ~1015 K for the sample heated in air, β -quartz diffraction peaks appeared and increased in intensity as heating continued to the maximum temperature. Cristobalite formed above ~1050 K, along with a small amount of clinoenstatite, and both phases persisted to the maximum temperature studied.

Keywords: Palygorskite, Rietveld, synchrotron, X-ray diffraction, time-resolved