
Small-Angle X-Ray Powder Diffraction, Morphology, and Structure of Allophane and Imogolite

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Abstract: Small-angle X-ray powder diffraction analyses and high-resolution electron microscopy of allophane samples ($\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio, 1.12 to 1.68) showed that allophanes consist of nearly identical spherical particles with diameters of about 40 Å and retain their characteristic “hollow” spherical morphology at different ambient moisture and even after dehydroxylation by heating at 500° to 600° C. Unheated allophane samples gave another X-ray powder diffraction band whose maximum position varied from 12.3 to 14.5 Å depending on their $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio. The appearance of this band may denote some long-range ordering in the structure of allophane. Unlike the spherical particles of allophane, the tube unit of imogolite collapsed on dehydroxylation. This observation suggests that imogolite and allophane are different in their framework structures and that a Si- or Si(Al)-tetrahedral sheet rather than an Al-octahedral sheet constitutes the framework structure of allophane, irrespective of its $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio.

Key Words: Allophane • Dehydroxylation • Imogolite • Infrared spectroscopy • Morphology • Small angle scattering

Clays and Clay Minerals; June 1985 v. 33; no. 3; p. 237-243; DOI: [10.1346/CCMN.1985.0330310](https://doi.org/10.1346/CCMN.1985.0330310)

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