

# CONTROLLED SYNTHESIS OF ALUMINOSILICATE NANOTUBES USING MULTIVARIATE REGRESSION ANALYSIS OF SYNTHESIS CONDITIONS

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Aluminosilicate minerals like imogolite nanotubes and allophane nanospheres coprecipitate due to the weathering of volcanic glass and ash. They impact nutrient and contaminant mobility in soil, as well as the formation of clays like halloysite depending on Si availability. It has been suggested that allophane might precede imogolite, and this detail is vital for laboratory production of these minerals since other aluminum-based minerals, like boehmite, can concurrently occur. Imogolite, in particular, has gained increased use in a wide range of applications, including catalysis, gas storage, sensors, gene delivery, and polymer nanocomposites, and so the presence of other minerals as impurity phases is non-ideal. However, few studies have investigated imogolite's synthesis while factoring in all relevant synthesis parameters to maximize nanotube yield and account for these other mineral phases. This present study investigates the complex system of physical and chemical conditions that influence the formation of imogolite and its impurity counterparts using multivariate linear regression analysis. Samples are synthesized and analyzed by powder x-ray diffraction, *in situ* and *ex situ* small-angle x-ray scattering (SAXS), and transmission electron microscopy. Regression analyses combined with linear combination fitting of powder diffraction patterns are used to model the influence of different synthesis conditions, including concentration, hydrolysis ratio and rate, and Al:Si elemental ratio on the particle size of the initial precipitate and the phase abundances of the final products. Particle size distribution analysis and Guinier analysis of the SAXS data are used to confirm whether allophane or proposed proto-imogolite precedes imogolite formation. The approach used in this study should be useful to model and understand other geochemical systems while accounting for the effect of conflicting biases.