Characterization of Tubular Chrysotile by Thermoporometry, Nitrogen Sorption, Drifts, and TEM*

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Abstract: The maximum crystal radius R_n of ice in hollow wet chrysotile tubes is established by thermoporometry to be between 2.8 and 3.2 nm, and the internal pore volume V_n of the tubes to be between 0.008 and 0.02 ml/g. The hollow tubes of chrysotile and, for comparative reasons, small plates of talc, are hydrothermally synthesized at temperatures between 563 and 600 K and at pressures between 75 and 120 hPa. Size and shape of the pores can be varied by changing the Mg/Si molar ratios in steps of 3/1.5 and 3/2 for chrysotile and 3/3.6 and 3/4 for talc. The tubular morphology of the aggregates dried at 393 K is investigated by 1) transmission electron microscopy (TEM), 2) nitrogen adsorption and desorption at 77 K, and 3) diffuse reflectance infrared fourier transformed spectroscopy (DRIFTS). The radius within the hollow tubes, R_i, is between 2.5 and 4.0 nm as measured by TEM, and between 2.8 and 3.2 nm as determined by nitrogen adsorption and desorption. The measured radii agree well with the value calculated from crystallographic data, which is smaller than 5.3 nm. Within the dried aggregates the tubes are clustered in regular patterns, in which each tube is surrounded by six other tubes. The external radius, R_o, between the clustered tubes is from 1.6 to 2.9 nm as observed by TEM, and from 1.8 to 2.3 nm by N_2 adsorption and desorption. The external radius is not measured by thermoporometry. Where thermoporometry only measures the average pore size and pore volume within the tubes, TEM and N_2 adsorption and desorption additionally provide the corresponding values between the tubes. A third pore radius, 5 to 20 nm between the clusters of chrysotile tubes, is established with N_2 adsorption and desorption.

Key Words: Chrysotile asbestos • Infrared spectroscopy • N_2 adsorption and desorption • Thermoporometry • Transmission electron microscopy • X-ray diffraction

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