Evolution of the Porous Structure and Surface Area of Palygorskite Under Vacuum Thermal Treatment

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Abstract: The modification of the external surface area and the two types of microporosity of palygorskite (structural and interfiber porosity) were examined as a function of the temperature of a vacuum thermal treatment to 500 ° C. The methods used included: controlled-transformation-rate thermal analysis, N_2 and Ar low-temperature adsorption microcalorimetry, conventional and continuous gas-adsorption volumetry (for N_2 and Ar) at 77 K and CO_2 at 273 and 293 K, water vapor adsorption gravimetry, and immersion microcalorimetry in water. At temperatures < 100 ° C only 18% of the structural microporosity was available to N_2, 13% to Ar, and 100% to CO_2 at 273 K. In both experiments the channels filled at very low relative pressures. At temperatures between 70 ° and 130 ° C the structure folded, and the mineral transformed to anhydrous palygorskite, which showed no structural microporosity. The interfiber microporosity was found to be independent of the temperature treatment, and the external surface area decreased slightly from 65 to 54 m^2/g. The water adsorption isotherms showed that the folding of the structure was reversible up to final outgassing temperatures > 225 ° C.

Key Words: Adsorption • Microcalorimetry • Microporosity • Palygorskite • Surface area • Thermal treatment

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