

Experimental evidence of the formation of intermediate phases during transition of kaolinite into metakaolinite

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ABSTRACT

The phase composition of partially dehydroxylated specimens of two Clay Mineral Society standards, named KGa-1 and KGa-2 samples, were studied by powder X-ray diffraction patterns (XRD), infrared (IR) spectroscopy, and thermogravimetric (TG) data. Each specimen was preheated for 6 h in isothermal conditions, and the heating temperatures were selected to cover the entire range of dehydroxylation for both kaolinites (380–550 °C for KGa-2 and 400–600 °C for KGa-1). Products preheated at 550 and 600 °C are named metakaolinite, as their XRD patterns showed no traces of kaolinite reflections. The contribution of metakaolinite to the experimental XRD pattern and IR spectrum of each preheated specimen (including 3550–3750 and 500–1300 cm⁻¹ ranges) was determined by the simulation of the experimental pattern with a sum of the XRD patterns or IR spectra corresponding to the untreated sample and metakaolinite. This procedure showed that the contents of metakaolinite determined independently by diffraction and spectroscopic methods for each of the preheated specimen have almost identical values. Although the modeled and experimental XRD and IR spectra generally match, they contain particular ranges where the intensity differences are particularly significant. The misfits suggest the presence of a third phase forming in partially dehydroxylated kaolinite. To reveal the diffraction and spectroscopic features of the kaolinite and possible intermediate phase (or phases), the contribution of metakaolinite was subtracted from the experimental XRD pattern and diffuse reflectance infrared Fourier transform (DRIFT) spectrum of each preheated specimen. Analysis of the metakaolinite-free XRD patterns and DRIFT spectra supports the idea that each preheated specimen, along with metakaolinite and initial kaolinite, contains a kaolinite-like component termed the intermediate phase. To determine the contents and specific diffraction features of the intermediate phases, the contribution of initial, untreated kaolinite was subtracted from the metakaolinite-free XRD patterns for each preheated specimen. In both samples, the content of the intermediate phase, C_{inter} , decreases with the growth of metakaolinite from 23–25% at the beginning of dehydroxylation to 2% when the XRD-determined content of metakaolinite is 98%. The analysis of the diffraction features of the intermediate phases formed during partial dehydroxylation of the KGa-1 and KGa-2 samples indicates that they represent a new type of defective kaolinite-like structure, in which, along with the one-dimensional periodicity along the c^* axis and the layer displacement vectors typical for natural kaolinite samples, the octahedral sheets of the individual layers of the structure are partially dehydroxylated.

Keywords: Kaolinite, metakaolinite, dehydroxylation, X-ray diffraction, infrared spectroscopy, thermogravimetry