Thermal Decomposition of a Dickite-Hydrazine Intercalation Complex

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Abstract: The intercalation complex of a low-defect dickite from Tarifa, Spain, with hydrazine was studied by high-temperature X-ray diffraction (HTXRD), differential thermal analysis (DTA), and thermogravimetry (TG). The X-ray diffraction (XRD) pattern obtained at room temperature indicated that the intercalation of hydrazine and H_2O into dickite caused an increase of the basal spacing from 7.08 to 10.24 Å, which is slightly lower than the 10.4-Å spacing commonly observed after intercalation into kaolinite. Heating between 25–50 °C produced a structural rearrangement of the complex, which decreased the basal spacing from 10.24 to 9.4 Å, and the resulting 9.4-Å complex was stable between 50–90 °C. Heating between 90–300 °C caused a gradual reduction in spacing, which occurred through a set of intermediate phases. These phases were interpreted to be interstratifications of intercalated and non-intercalated layers. These changes were also observed by DTA and TG. Two main endothermic reactions and two main stages of mass loss, respectively, were indicated in the DTA and the TG curves in the temperature range 25–200 °C. This behavior suggests that intercalated molecules, hydrazine and H_2O, occupied well-defined sites in the interlayer of the dickite. The intercalated molecules were lost in an ordered fashion as confirmed by the infrared analysis of the decomposition products; H_2O was lost in the first stage and ammonia was identified in the second stage. Above 300 °C, complete removal of the intercalated molecules restored the basal spacing of the dickite. However, the basal reflections were broadened, the relative intensities were changed, and changes in the dehydroxylation temperature indicated that the intercalation-desorption process induced some stacking disorder in the dickite structure.

Key Words: Dickite • DTA-TG-DTG • HTXRD • Hydrazine • Intercalation Complex • Kaolinite

Clays and Clay Minerals; October 2000 v. 48; no. 5; p. 586-592; DOI: 10.1346/CCMN.2000.0480511

Clay Minerals Society (www.clays.org)