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Modification of the hydroxyl surface of potassium acetate intercalated halloysite between 25 and 300 °C

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ABSTRACT

Changes in the hydroxyl surfaces of potassium acetate-intercalated halloysite were studied over ambient to predehydroxylation temperature range using a combination of X-ray diffraction and Raman spectroscopy. XRD shows that the halloysite is completely expanded to 13.80 Å. Upon heating the intercalation complex to 50 °C under nitrogen, two expanded phases are observed with *d*-spacings of 11.47 and 8.95 Å. Upon thermal treatment, the 11.47 Å phase is stable in both the heating and cooling cycles. The ~9.0 Å phase undergoes an expansion at 100 °C to 9.2 Å. Upon exposure to air, the intercalated kaolinite returns to a 13.80 Å phase.

The completely intercalated halloysite showed a band at 3602 cm^{-1} attributed to the inner surface hydroxyl hydrogen bonded to the acetate ion together with bands at 3620 and 3695 cm^{-1} assigned to the inner hydroxyl and the inner surface hydroxyls, which do not react with the acetate. Mild heating of the intercalation complex to 50 °C caused a rearrangement of the surface structure with Raman bands being observed at 3606 and 3597 cm^{-1}. Further thermal treatment at 100 °C caused these bands to shift to 3615 and 3601 cm^{-1}. At the predehydroxylation temperature for potassium acetate intercalated halloysite (300 °C) two bands were observed at 3602 and 3612 cm^{-1}. Above this temperature no hydroxyls are spectroscopically evident. Upon cooling to room temperature, the Raman spectra of the hydroxyl surfaces are altered with two bands observed at 3604 and 3596 cm^{-1}.