Mechanism of Synthesis of 10-Å Hydrated Kaolinite

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Abstract: The synthesis of 10-Å hydrated kaolinite was accomplished by: (1) direct reaction of HF with a dimethyl sulfoxide (DMSO)-kaolinite intercalate and water washing; (2) methanol washing of a DMSO-kaolinite intercalate followed by reaction with any alkali fluoride salt and water washing; and (3) room-temperature (RT) water-washing of a methanol-washed DMSO-kaolinite intercalate. In all syntheses the optimum yield required a kaolinite in which DMSO was bound strongly to the interlayer surface. In the first synthesis, water inclusion between clay layers appeared to be facilitated by the reduction of cohesive interlayer forces brought about by replacement of surface and edge OH^- by F^-. The fluorination reaction was accomplished either by direct reaction of HF or by HF produced through the hydrolysis of NH_4F at 60 ° C. In the second synthesis, intercalated DMSO was replaced by methanol. F^- solvated readily in methanol but not in DMSO. Consequently, F^- produced through hydrolysis of the alkali fluoride salt entered the interlayer space and contributed to the fluorination reaction. Furthermore, the diffusion of methanol out of the interlayer space during the RT-washing step was slowed by F^- solvation which aided the exchange of methanol for water. High yields of 10-Å kaolinite hydrate were obtained irrespective of choice of alkali fluoride salt. The third synthesis was dependent on matching the diffusion of methanol out of the interlayer space with diffusion of water into this space. At room temperature the diffusion rates were close enough to maintain the clay in the expanded state throughout the hydration process, and high yields of 10-Å kaolinite hydrate were obtained. At 60 ° C the diffusion rates were too dissimilar, and very low yields of hydrate were obtained.

Key Words: Dimethyl sulfoxide • Fluoride salt • Hydrate • Kaolinite • Methanol • Synthesis • X-ray powder diffraction

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