Synthesis of a 10-Å Hydrated Kaolinite

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Note added in proof: We have recently used DMSO as the intercalating agent without adding a fluoride salt. The clay/DMSO suspension was washed three times with absolute methanol followed by three water washings. This sequence of treatments produced substantial yields of the 10-Å hydrate. It is not clear what the methanol washing did to the intercalated clay, but the treatment may have increased the level of stacking disorder and thus increased the stability of the 10-Å hydrate.

Abstract: Hydrated kaolinite (d(001) = 10 Å) can be synthesized by mild healing of a kaolinite-organic suspension, allowing time for the clay to be intercalated by the organic solvent, and then dissolving a fluoride salt in the liquid. After mild heating of the suspension, the salt and organic solvent are removed by repeated water washings. The kaolinite retains interlayer water in the form of a 10-Å kaolinite hydrate. The influence of the intercalating agent, type of salt, concentration of salt, and the time of treatment on the synthesis of 10-Å hydrate was examined for several kaolinites. The most effective salt is NH_4F; much smaller yields were obtained using KF and RbF. Not all organic molecules gave high yields of the hydrate; dimethyl sulfoxide, formamide, and hydrazine worked well but N-methyl formamide did not. The reaction between clay and salt resulted in the replacement of some hydroxyls by fluoride. This replacement was rapid; after 1 min of fluoride treatment a substantial yield of hydrate was obtained. The intercalation step separated the layers and also disordered the kaolinite, facilitating the F for OH replacement at or near crystallite edges. This replacement weakens the interlayer bonding at the edges and thereby reduces the possibility of layer collapse and attendant dehydration.

Key Words: Dimethyl sulfoxide • Fluoride • Formamide • Halloysite • Hydrazine • Hydroxyl • Intercalation • Kaolinite

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