Simplified, Complete CsCl-Hydrazine-Dimethylsulfoxide Intercalation of Kaolinite

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Abstract: The coarse (2−0.2 μm) and fine (<0.2 μm) size fractions of several soil and reference kaolinite samples were completely intercalated and expanded to 11.2 Å using a simplified CsCl-hydrazine-dimethylsulfoxide (DMSO) treatment. Rapid equilibration of the clay in hot (80 ° C) hydrazine monohydrate and hot (100 ° C) DMSO, and the use of ceramic tile mounts, limited the sample pretreatment time to only 15 min. The fine size fraction of kaolinite may be X-rayed immediately after pretreatment, though analysis of the sample 2 or 12 hr after pretreatment produced more intense, sharp basal reflections. This difference may be due to a better ordering of the DMSO-kaolinite complex with time and to a drying of the excess DMSO. The coarse size fraction of kaolinite did not entirely expand to 11.2 Å when analyzed immediately after pretreatment. A 9.6-Å peak was also present and possibly represents a mixed layering of expanded and nonexpanded kaolinite layers. The larger crystals seem to require additional time for the reaction to become complete as evidenced by the presence of an intense, sharp 11.2-Å peak and absence of the 9.6-Å peak when the coarse clay was analyzed 2 or 12 hr after sample pretreatment. Kaolinite particles <50 μm did not react completely even when they were analyzed 24 hr after pretreatment. Therefore, this technique should be limited to <2-μm particles.

Key Words: Dimethylsulfoxide • Expansion • Intercalation • Kaolinite • X-ray powder diffraction

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