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# Quantification of Crystalline and Noncrystalline Material in Ground Kaolinite by X-ray Powder Diffraction, Infrared, Solid-State Nuclear Magnetic Resonance, and Chemical-Dissolution Analyses<sup>1</sup>

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**Abstract:** The capabilities of X-ray powder diffraction (XRD), infrared absorption (IR), solid-state magic-angle-spinning nuclear magnetic resonance (MAS-NMR), and chemical dissolution methods were assessed for estimating the amount of noncrystalline material in a ground kaolinite. The Georgia kaolinite was ground in a mechanical mortar for various lengths of time to produce a set of ground samples containing different amounts of the resulting noncrystalline material. In the XRD method, the intensities of characteristic reflections at 7.2 and 4.47 Å did not respond proportionally to the amount of crystalline kaolinite. Although a transmission-type X-ray diffraction method using the *hk* reflection gave a slightly better estimate than the reflection-type X-ray diffraction method using the basal reflection, both methods gave overestimated values for the amount of noncrystalline material. This overestimation may have been caused by a masking effect due to coaggregation. Using the characteristic IR absorption band at 3700 cm<sup>-1</sup> underestimated the amount of the noncrystalline material, if the proportion of this material <50%.

Extraction with NaOH gave estimations 15 to 20% greater than extraction with alkaline Tiron, except for the sample ground for 24 hr, for which both extractions indicated the presence of about 50% noncrystalline material. X-ray powder diffraction data of the residues after these extractions indicated that they consisted of crystalline kaolinite. <sup>29</sup>Si NMR spectra of samples ground for ≥30 hr suggested that SiO<sub>4</sub> tetrahedra were considerably distorted. <sup>27</sup>Al NMR spectra showed a signal for tetrahedral Al for the sample ground for 10 hr, which increased with an increase in grinding time. Plots of the Al(IV)/[Al(IV) + Al(VI)] ratios vs. time were similar to those of chemical extraction curves. Inasmuch as extraction with hot 0.5 M NaOH is a rather harsh treatment, the composition of the noncrystalline material must have been similar to that of the crystalline kaolinite. The chemical dissolution using alkaline Tiron appeared to be superior to other methods, such as XRD, IR, and NaOH extraction, for estimating the amount of noncrystalline material in kaolinite.

**Key Words:** Chemical dissolution • Dry grinding • Infrared spectroscopy • Kaolinite • Noncrystalline • Nuclear magnetic resonance • X-ray powder diffraction

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