Application of Electron Paramagnetic Resonance and Mössbauer Spectroscopy in the Investigation of Kaolinite-Group Minerals

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Abstract: Samples of nacrite, dickite, kaolinite, and halloysite were investigated using X-band electron paramagnetic resonance (EPR) and Mössbauer spectroscopy. Fe³⁺ gave rise to EPR signals at $g \simeq 4$ which differed with the individual polytype. Only nacrite had no resonance in this region of the spectrum, but it had one at $g \simeq 2$. Dickite had a quadruple line, kaolinite a triple line, and halloysite a single line in this region. The EPR spectra of these minerals are apparently dependent also on the orientation of adjacent layers in the structures. Other resonances were attributed to (1) clusters of ferric ions giving rise to broad resonance near $g \simeq 2$, (2) trapped holes, and (3) free radicals linked with organic matter. The Mössbauer spectroscopic results suggest that iron occurs in the ferric state (except in nacrite where Fe²⁺ is present also) as an ionic substitution (IS) in octahedral layers. This suggestion follows from the difference is the IS values between octahedral and tetrahedral symmetry sites occupied by Fe³⁺ equal to ~ 0.4 mm/sec. Linewidths depend mainly on the way the layers stack; for monoclinic modifications represented by nacrite and dickite, the linewidths are narrow ($\Gamma = 0.45$ mm/sec and 0.56 mm/sec, respectively); pseudomonoclinic halloysites also gave narrow linewidths ($\Gamma = 0.39$ mm/sec and 0.48 mm/sec). The widest line was observed for triclinic kaolinite ($\Gamma = 0.62$ mm/sec and 0.71 mm/sec).

Key Words: Dickite • Electron paramagnetic resonance • Halloysite • Iron • Kaolinite • Mössbauer spectroscopy • Nacrite

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