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# Mechanisms of Palygorskite and Sepiolite Alteration as Deduced from Solid-State $^{27}\text{Al}$ and $^{29}\text{Si}$ Nuclear Magnetic Resonance Spectroscopy

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**Abstract:** The mechanisms of palygorskite and sepiolite alteration to smectite under mild hydrothermal conditions were investigated by solid-state  $^{27}\text{Al}$  and  $^{29}\text{Si}$  magic-angle spinning-nuclear magnetic resonance (MAS-NMR) spectroscopy, X-ray powder diffraction (XRD) and transmission electron microscopy (TEM). Palygorskite altered to smectite in the presence of NaOH at 150°C.  $^{27}\text{Al}$  MAS-NMR spectroscopy showed that the Al coordination changed from chiefly octahedral in palygorskite to chiefly tetrahedral in the smectite product.  $^{29}\text{Si}$  MAS-NMR spectroscopy showed that the nearest neighbor environment of Si also changed when palygorskite altered to smectite. The XRD data showed that the synthetic smectite is trioctahedral in nature with tetrahedral charge. The TEM results revealed that the needle-like morphology of palygorskite was preserved in the product smectite. The MAS-NMR results in conjunction with the above XRD and TEM studies suggest that the mechanism of palygorskite alteration was a dissolution and recrystallization process rather than a solid-state reorganization to form 2:1 layer silicate units from the preexisting chain structure. Sepiolite altered to smectite in the presence of 2 N salt solutions at 300°C. The trioctahedral nature of the product smectite as detected by XRD and the foil-like morphology of product smectite as shown by TEM suggest that the mechanism of sepiolite transformation to smectite was also a dissolution and recrystallization process. The tetrahedral Al coordination detected by  $^{27}\text{Al}$  MAS-NMR in the smectite altered from sepiolite corroborated the XRD and TEM results.

**Key Words:** Al coordination • Hydrothermal transformation • Nuclear magnetic resonance • Palygorskite • Sepiolite • Smectite • X-ray powder diffraction

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