
Interpretation of Solid State ^{13}C and ^{29}Si Nuclear Magnetic Resonance Spectra of Kaolinite Intercalates

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Abstract: ^{13}C and ^{29}Si nuclear magnetic resonance spectroscopy with magic-angle spinning has been used to study the short-range ordering and bonding in the structures of intercalates of kaolinite with formamide, hydrazine, dimethyl sulfoxide (DMSO), and pyridine-*N*-oxide (PNO). The ^{29}Si chemical shift indicated decreasing levels of bonding interaction between the silicate layer and the intercalate in the order: kaolinite: formamide ($\delta = -91.9$, ppm relative to tetramethylsilane), kaolinite: hydrazine (-92.0), kaolinite: DMSO (-93.1). The ^{29}Si signal of the kaolinite: PNO intercalate (-92.1) was unexpectedly deshielded, possibly due to the aromatic nature of PNO. The degree of three-dimensional ordering of the structures was inferred from the ^{29}Si signal width, with the kaolinite: DMSO intercalate displaying the greatest ordering and kaolinite:hydrazine the least. ^{13}C resonances of intercalating organic molecules were shifted downfield by as much as 3 ppm in response to increased hydrogen bonding after intercalation, and in the kaolinite: DMSO intercalate the two methyl-carbon chemical environments were non-equivalent ($\delta = 43.7$ and 42.5).

Key Words: Dimethylsulfoxide • Formamide • Hydrazine • Intercalate • Nuclear magnetic resonance • Ordering • Pyridine-*N*-oxide

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