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Hydrogen bonded silanols in the 10 Å phase: Evidence from NMR spectroscopy

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Abstract

Using ^{29}Si, ^{1}H, and ^{2}H magic-angle spinning (MAS) and ^{29}Si{^{1}H} heteronuclear correlation (HetCor) nuclear magnetic resonance (NMR) spectroscopy the tetrahedral sheets of the 10 Å phase are shown to contain Q^2-type Si bonded to silanol groups that donate hydrogen bonds to interlayer H_2O. ^{29}Si NMR spectra of 10 Å phase samples synthesized from oxide and from crystalline talc starting materials contain a peak near –87 ppm for Q^2 Si, in addition to the main peak at –98 ppm for the talc-like Q^3 of the tetrahedral sheet. The ^{1}H MAS NMR spectra of the 10 Å phase contain two distinct peaks, at chemical shifts of +7.8 and +3.2 ppm, in addition to a narrow peak near +0.9 ppm from the talc-like hydroxyl groups. ^{29}Si{^{1}H} HetCor data indicate that the +7.8 ppm ^{1}H resonance corresponds to silanol groups and that at +3.2 ppm arises from interlayer H_2O. Comparison of the observed data with correlations of ^{1}H NMR chemical shift and ^{2}H quadrupolar coupling indicates that the silanol groups donate moderate hydrogen bonds to interlayer H_2O, $d(O\cdots O) \approx 2.8$ Å, whereas most interlayer H_2O donate only very weak or no hydrogen bonds at ambient conditions. Our results suggest that formation of the 10 Å phase involves formation of vacancies, which allow favorable hydrogen bond interaction between interlayer H_2O and the normally hydrophobic talc-like 2:1 layers.

Keywords: NMR, 10 Å phase, talc, silanol, hydrogen bond