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Requested Dates: Wednesday October 19 to Monday October 24
Requested Equipment:
6 mm probe platformed to HFD, with 500-1000Hz spinning speeds (manual control).
Liquid nitrogen in the small dewar for removing caps and inserts.

Sample Information:
kaolinite:D2O mixtures in FEP inserts inserted into a 6 mm rotor. 5-10 samples with varying kaolinite:D2O ratios prepared beforehand in the wetlab. No health hazards involved.

Research Proposal:
(1) Prepare kaolinite:D2O mixtures sealed in FEP inserts in the wetlab before coming to the NMR lab.
(2) Shim the 6 mm probe on D2O to get 2-3 Hz linewidth, and repeat shimming at the start of each day (takes about 30-60 minutes)
(3) Calibrate 2H and residual 1H powers and pulse widths on D2O (1-2 hours)
(4) Measure static and spinning (ca. 500 Hz MAS) 2H and residual 1H NMR spectra of the pure D2O and the kaolinite:D2O mixtures to obtain chemical shift and linewidth information. 4 static and 4 spinning spectra alternating static-spinning-static-spinning etc... (1 day)
(5) Measure spinning 2H and residual 1H T1 (inversion recovery) amd T2 (Hahn echo) of D2O and the kaolinite:D2O mixtures (1 day)
(6) Use remaining time to perform similar experiments on MFT:D2O mixtures that are also prepared before coming to the NMR lab (2-3 days)

Previous Work:
Four kaolinite:D2O samples, in approximate ratios of 1:3, 1:2, 2:1 and 3:1 were prepared. We found that the 2H chemical shift depends on the amount of kaolinite, with more kaolinite giving a larger low frequency shift (4.8 ppm for pure D2O, and approaching 4.5 ppm for 3kaolinite:1D2O. We believe this implies fast exchange between surface and bulk D2O, with a weighted average shift. We also found that the kaolinite shortens both 2H T1 and 2H T2, compared to pure D2O, and we believe this is a result of the kaolinite slowing the motion on the D2O molecules at the surface, resulting in more efficient relaxation. For the next step, we need to make more accurate measures of the weight of kaolinite (i.e. 5 mg, 10 mg, 15 mg, 20 mg), instead of the previously used "eyeballing" method, then repeating the same experiments. We have also performed similar measurements on MFT:D2O mixtures (in "eyeball" ratios), we need more accurate sample weights before we can present our data.

Relevant Literature References:
(1) Computer simulation of water molecules at kaolinite and silica surfaces MR Warne, NL Allan and T Cosgrove, Phys Chem Chem Phys 2, 3663-3668 (2000).
(2) Static and dynamic structure of water in hydrated kaolinites II. The dynamic structure. M Lipsicas, C Straley, PM Costanzo and RF Giese J. Colloid. Interfac. Sci. 107(1), 221-230 (1985).