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To: Jim Shell, G-PLUS Facilitator
Re: Final Project for G-PLUS Project

Since our previous report we have accomplished additional Nuclear Magnetic Resonance (NMR) experiments on the sodium (^{23}Na) nuclei in the provided fiberglass samples (CertainTeed). The spectra were obtained using the Hahn spin-echo pulse sequence during magic angle spinning (MAS) in a 200 MHz magnet, spun at about 28 kHz. A 0.1M NaCl solution was used as a standard. Magic angle spinning was performed on these samples in order to narrow the line width of the spectra in case multiple peaks were present that may have been hidden within the broad spectrum obtained previously without spinning.

Shown below in *Figure 1* are the ^{23}Na-NMR spectra for all the fiberglass samples. Please note that the letters represent the plant from which each sample came; the NMR spectra on the samples **without binder** are shown on the left, and the results for the samples **with binder** on the right. Although there are peaks shown at the two ends (9000 ppm and -9000 ppm) of the spectral window, these artifacts result from an unavoidable imperfection in the NMR detector. In addition to these artifacts, peaks present at around -4900 ppm and -4000 ppm are observed and expected due to spinning sidebands (a result from performing MAS) and are not relevant to this investigation. The relevant signals for the binder and no binder samples are centered at around -4440 ppm.

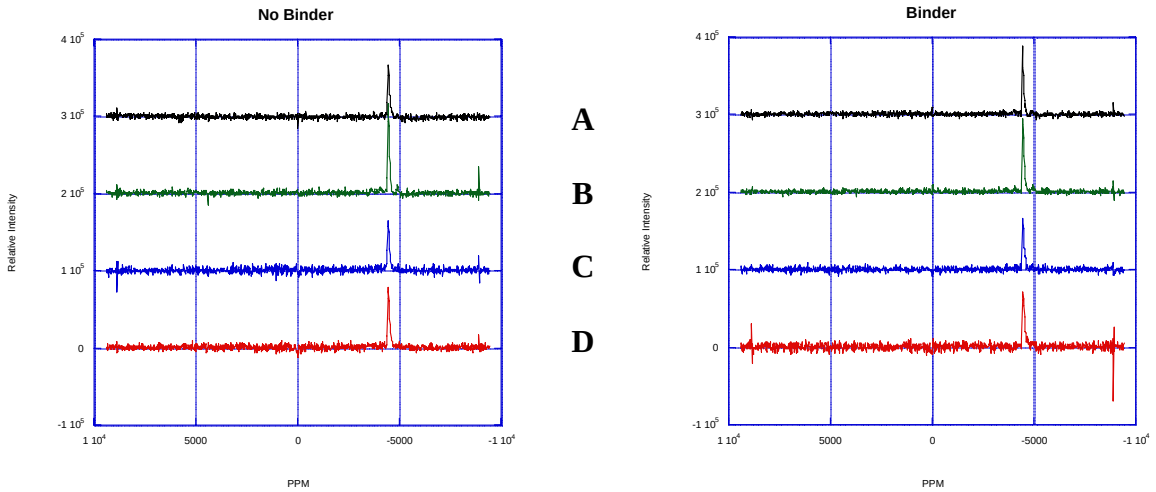

Figure 1: NMR spectra of the fiberglass samples with, and without binder performed with MAS.

Shown below in *Figure 2* are the same ^{23}Na-NMR spectra as in *Figure 1*, but ranging from -3000ppm to -6000ppm. From the spectra, it can be seen that the lineshapes of the samples with and without binder are very similar. Note that the intensities of these peaks are all relative to one another.

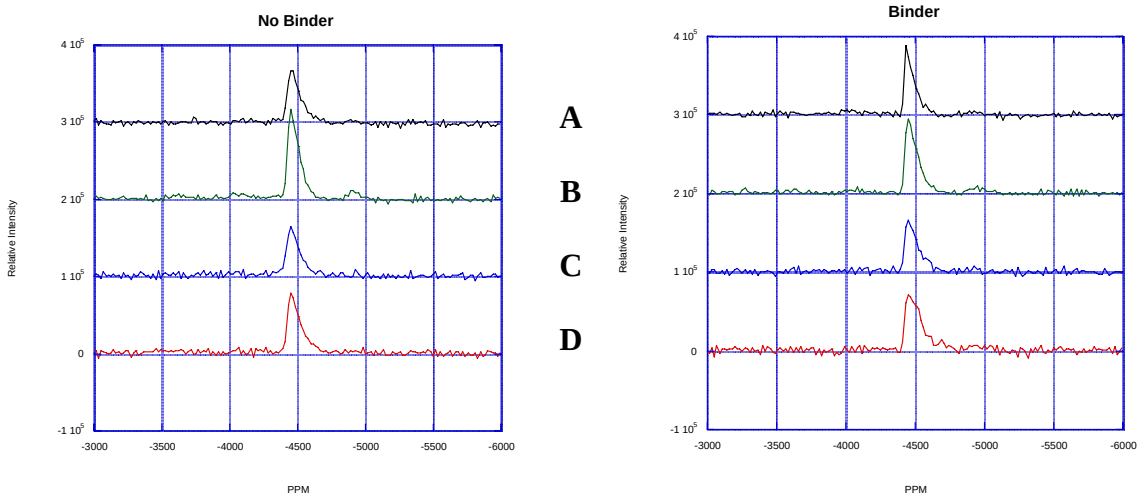

Figure 2: NMR spectra of the fiberglass samples with, and without binder performed with MAS, with an expanded scale relative to *Figure 1*.

These samples were fit to the sum of two Gaussian line shapes. Representative fits for the spectra derived from fiberglass at site B are shown below. *Table 1* lists the details of these fits, containing the $^{1}/_{2}$ width at half-height and center-of-mass (the "shift") of each fitted distribution.

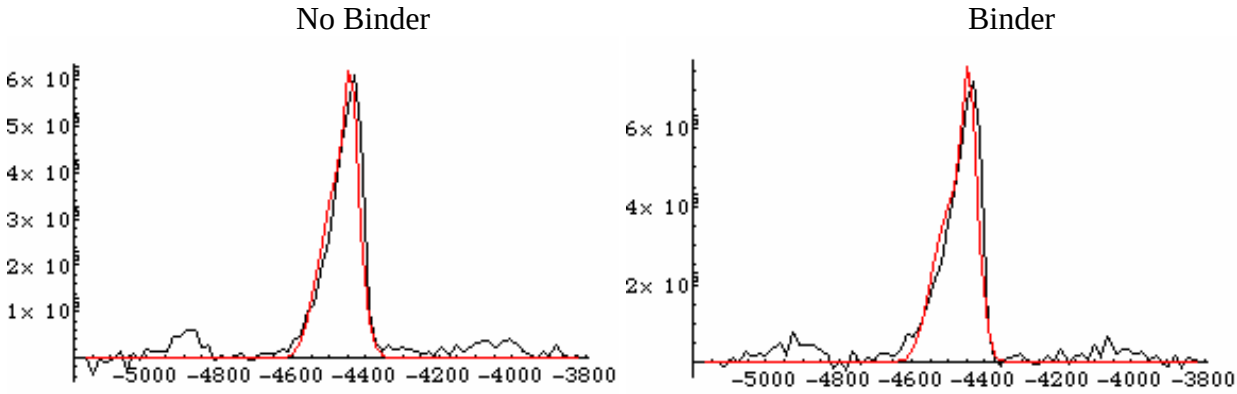

Figure 3: NMR spectra and fitting curves for sample B, with the red curve representing the fit and the black curve representing the actual spectra.

The values from the table below indicate either two different populations of Na nuclei in the samples with and without binder, or the presence of a small second-order quadrupolar broadening from the Na central transitions. In either case, the two-component fits exhibit

approximately the same shifts, at -4430ppm and at -4450ppm. The relative intensities and half-widths of each population do not provide any further information.

Table 1: Parameters derived from curve fitting of NMR spectra.

Sample		1/2 width @ half-height 1 (Hz)	Shift 1 (ppm)	Relative Intensity 1	1/2 width @ half-height 2 (Hz)	Shift 2 (ppm)	Relative Intensity 2
A	No Binder	43.9	-4443.9	64.0	64.5	-4501.7	36.0
	Binder	30.5	-4426.6	56.8	53.9	-4473.4	43.2
B	No Binder	37.6	-4436.9	52.5	57.2	-4482.3	47.5
	Binder	39.2	-4439.8	58.2	58.7	-4494.6	41.8
C	No Binder	44.8	-4443.8	62.5	74.8	-4484.2	37.5
	Binder	44.2	-4444.2	66.2	65.3	-4511.9	33.8
D	No Binder	43.4	-4444.1	63.3	67.9	-4506.9	36.7
	Binder	40.8	-4435.2	45.4	63.8	-4501.6	54.6

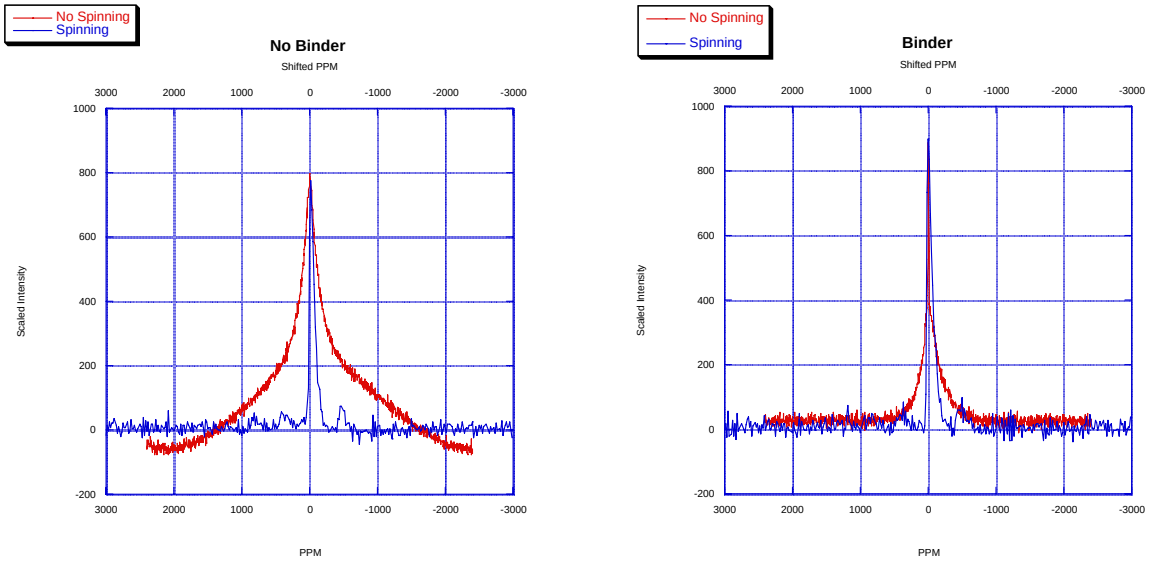

Figure 4. Comparison between the spinning and static spectra of Sample B.

From *Figures 1* and *2*, it does not appear that any other peaks are present besides the one at centered at around -4440ppm. As can be seen in *Figure 4*, spinning the samples at the magic angle does indeed suppress line broadening, but fails to expose any significant new spectral features. Our conclusion from these studies is that further analysis of the structure and composition of these fiberglass samples will require more extensive NMR techniques, as well as investigations of other nuclei. At the present time the CertainTeed partners consider such further investigations outside the scope of the G-PLUS program.