

## SUPPLEMENTARY MATERIALS:

# A Spectroscopic Study of Solid-Phase Chitosan/Cyclodextrin-Based Electrospun Fibers

Chen Xue and Lee D. Wilson \*

Department of Chemistry, University of Saskatchewan, 110 Science Place, Saskatoon, SK, S7N 5C9, Canada; chx257@mail.usask.ca

\* Correspondence: lee.wilson@usask.ca; Fax: +1-306-966-4730; Tel: +1-306-966-2961

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## Experimental Methods

### *<sup>13</sup>C solid state NMR Spectroscopy*

<sup>13</sup>C solid state NMR spectra were obtained with a Bruker AVANCE III HD spectrometer equipped with a 4 mm DOTY CP-MAS (cross-polarization with magic angle spinning; cp-mas) solids probe operating at 125.77 MHz (<sup>1</sup>H spectral frequency at 500.23 MHz). The <sup>13</sup>C CP-TOSS (Cross-polarization with total suppression of spinning sidebands) spectra were obtained at a spinning speed of 6 to 7.5 kHz, a <sup>1</sup>H 90° pulse of 3.5 μs, variable contact time (0.75 to 3 ms) with a ramp pulse on the <sup>1</sup>H channel, and 1000-4000 accumulated scans with a recycle delay of 2 s for all samples.

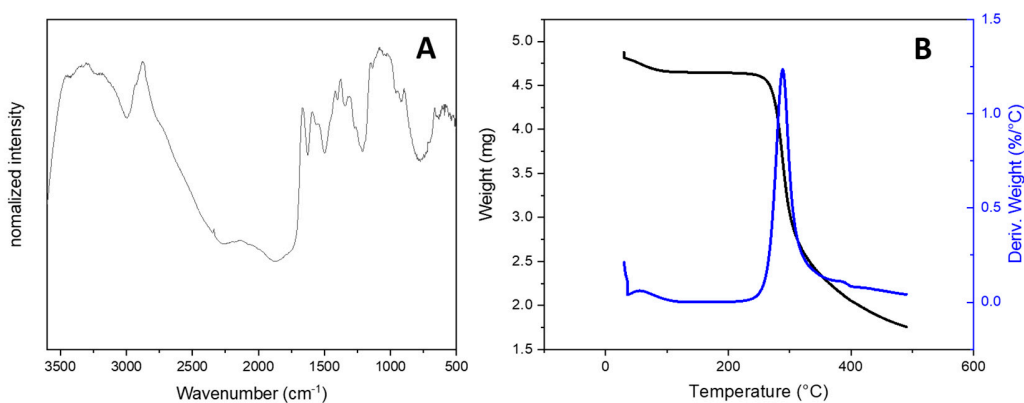
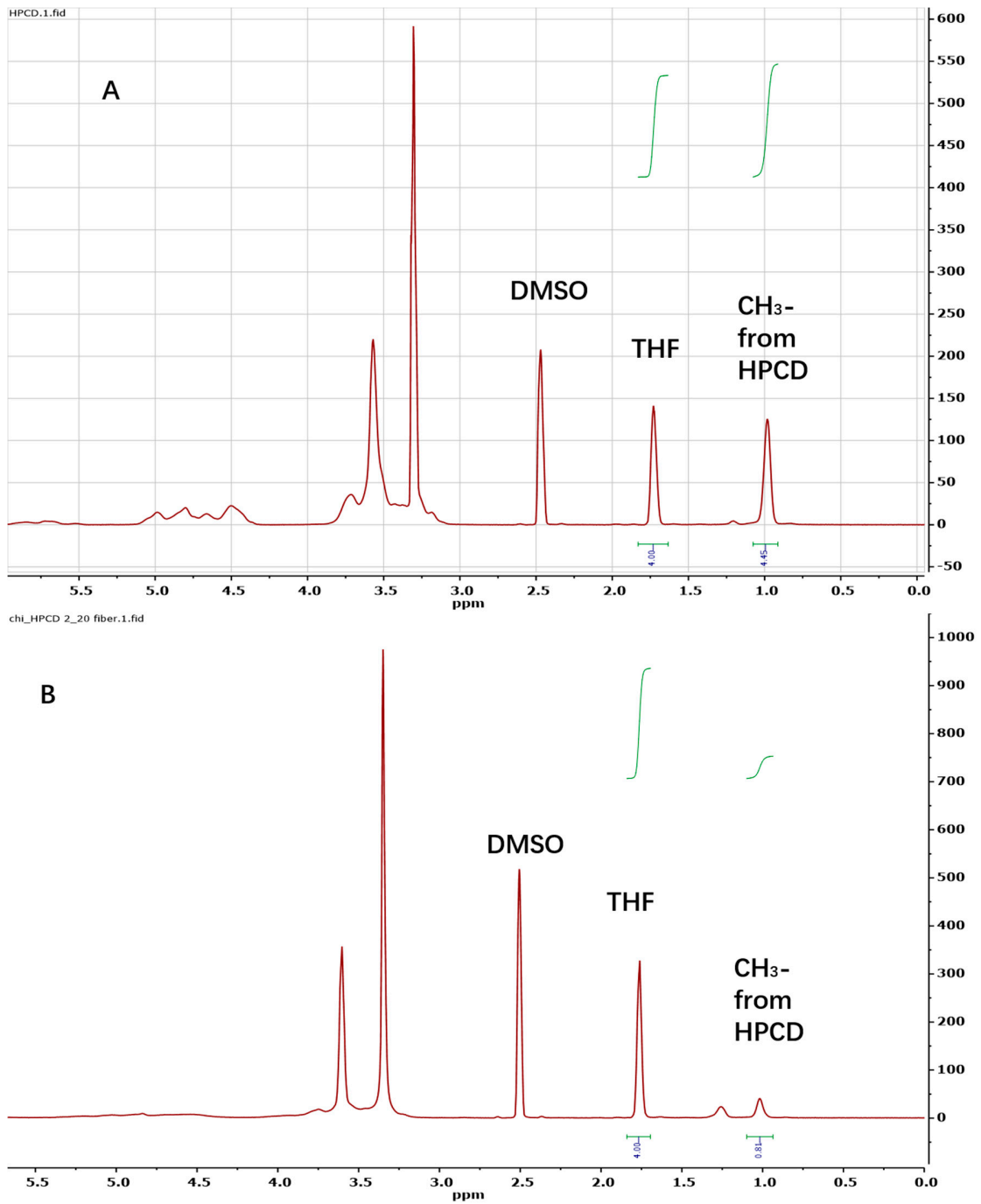
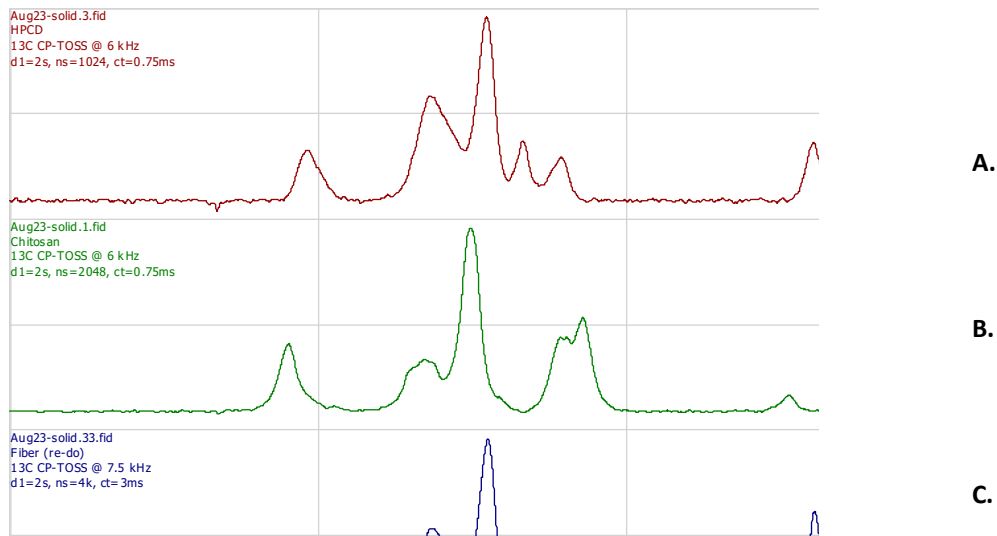


Figure S1. FT-IR spectrum (A) and DTG plot (B) of pristine chitosan.



**Figure S2.** <sup>1</sup>H NMR spectra of pure HPCD (A) and Chi:HPCD 2:20 fiber (B) prepared in 1% (w/w) THF/DMSO-*d*<sub>6</sub> solution for HPCD content determination.



**Figure S3.** Solid-state  $^{13}\text{C}$  CP-TOSS NMR spectra of HPCD (A), Chitosan (B), and Chi:HPCD (C) 2:50 fiber (from top to bottom, respectively).



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