

Supplementary Information

Preparation and characterization of cotton fiber fragments from model textile waste via mechanical milling and enzyme degradation

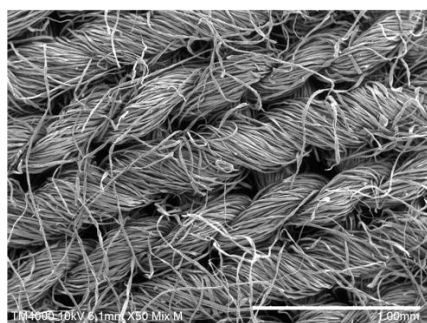
Siyan Wang¹, Jeannie Egan², Sonja Salmon^{3,*}

^{1,2,3} Department of Textile Engineering, Chemistry and Science, Wilson College of Textiles, North Carolina State University, Raleigh, NC 27695-8301, USA

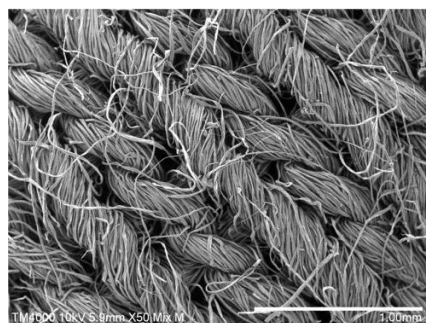
¹ swang76@ncsu.edu

² jmegan@ncsu.edu

^{3,*} Correspondence: sisalmon@ncsu.edu



(a) Red cotton fabric



(b) Blue cotton fabric



(c) Red cotton fabric



(d) Blue cotton fabric

Figure S1. SEM (a, b) and stereoscope images (c, d) of red and blue cotton fabrics. Scale bar 1.00 mm.



Figure S2. Optical microscopy images of undyed cotton in the original, milled and enzyme treated states. Scale bar 500 μm.

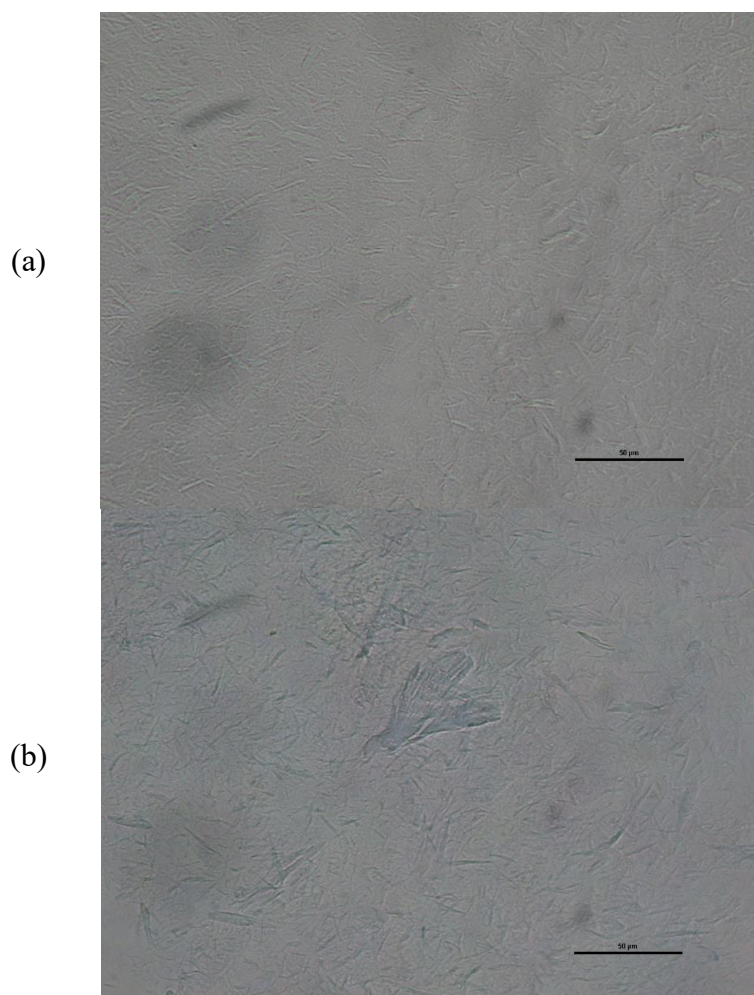




Figure S3. Optical microscopy images of (a) undyed, (b) blue and (c) red cotton fiber fragments resulting from 116-hour treatment in the 2mag mixer with a cellulase dose of 16 FPU/g fabric. Scale bar 50 μm . The less degraded narrow dark red segment in the fiber fragment near the top of (c) and the dark, approximately 50 μm long cylindrical fragment near the center-right of (c), with cracks parallel to its length, are evidence of zones in cotton fibers called “reversals”.



Figure S4. Red cotton dissolved in CED solvent overnight.

The small purple particles attached on the flask wall were undissolved red cotton fibers, indicating that this sample resisted complete dissolution in CED solvent.

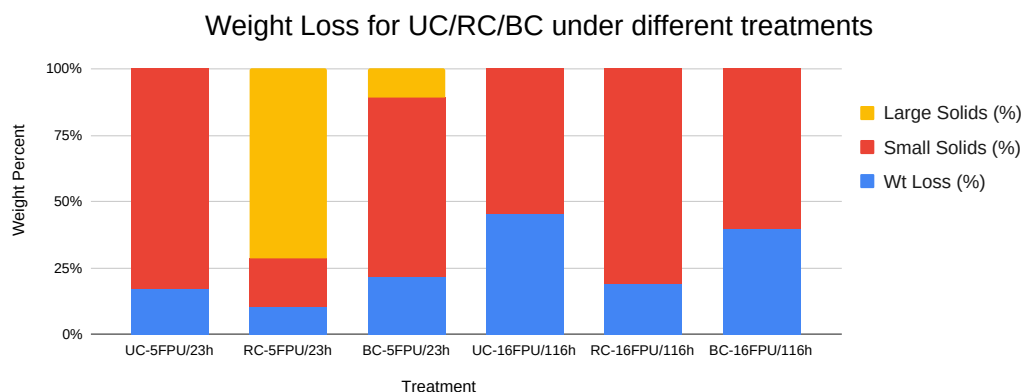


Figure S5. Weight loss graph for the different CFFs. Samples were: undyed cotton (UC), red cotton (RC), and blue cotton (BC), each treated with a lower (5FPU) or higher (16FPU) enzyme dose per gram dry fabric, and each treated for 23 hours (23h) or 116 hours (116h). Note that duplicate samples treated for 23 hours were treated using 10 mM sodium acetate buffer, whereas single samples treated for 116 hours were treated using 100 mM sodium acetate buffer, both at pH 5.0.

At higher enzyme dose and longer incubation time treatment, all three fabric types were completely converted to a slurry (no large solids left). After long reaction time with high enzyme dose, more undyed cotton was converted to soluble products (as indicated by the higher % weight loss) than reactive dyed cotton, indicating that the presence of reactive dyes interferes with enzyme catalyzed cotton degradation. The red dyed cotton exhibited the lowest weight loss, and also was the most difficult to convert completely from large solids to a slurry. Therefore, the presence of bi-functional reactive red dye presented the largest obstacles for enzymatic cotton degradation.

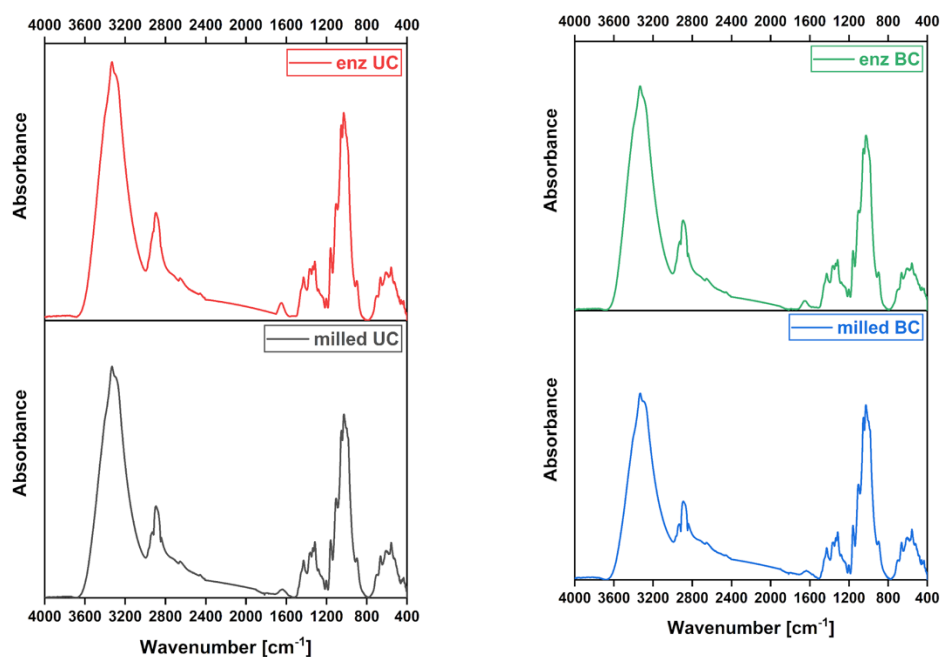


Figure S6. FTIR spectra of four CFFs (clockwise from top left): crushed Enz UC, crushed Enz BC, milled BC, and milled UC.

The pronounced OH stretching peak ($3500\text{--}3000\text{ cm}^{-1}$) observed for crushed enzyme treated cotton is attributed to water absorption and implies increased hydrophilicity of the sample after grinding. Sample grinding was performed manually, using a mortar and pestle. The grinding converted compressed flake-like samples to a loose powder form.

After crushing in a mortar and pestle, the appearance of Enz CFFs was a powder-like material (blue cotton), and a coarse flake "ground pepper"-like material (undyed cotton).