Hydrophobic Development and Mechanical Properties of Cellulose Substrates Supercritically Impregnated with Food-Grade Waxes

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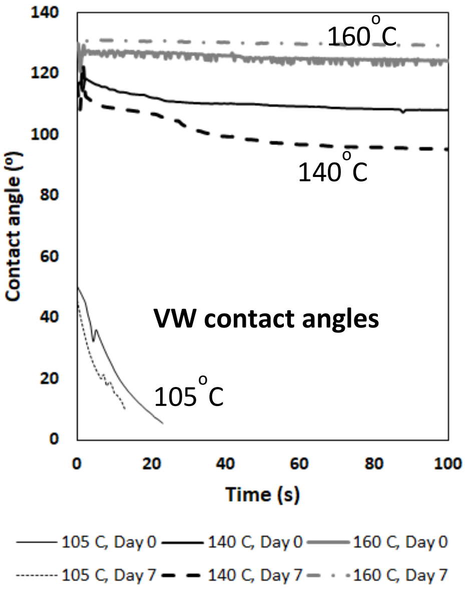
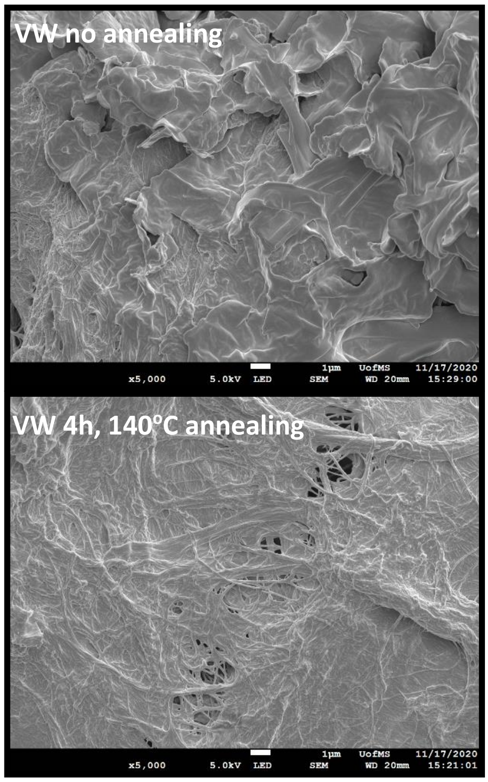
Supercritical impregnation (SCI) is a relatively common process that introduces dissolved solute from the supercritical fluid into porous substrates to improve their functionality. Common in wood or textiles applications, in this study, food-grade hydrophobic waxes were introduced into paper substrates to render them water repellent, and potentially improve their overall strength. This research finds applications in food packaging or other similar developments.



The waxes used for this study were beeswax (BW), carnauba wax (CW) and vegetable wax (VW), each dissolved into supercritical carbon dioxide (20°C, 200 bar, i.e. supercritical in pressure only). Hydrophobicity of the paper was improved dramatically when the treated paper samples were subsequently annealed for four hours at 140°C, with resulting contact angle (CA) measurements recorded between 110 – 120°C. Higher annealing temperatures resulted in higher CA. This dependence upon annealing temperature was explained by the development of a dual micro-/nano-scale roughness observed on the impregnated fibers, which was largely absent with samples that did not undergo annealing. CA graphs and SEM images are shown for VW studies. FTIR analysis revealed hydrogen bonding between the waxes and cellulose, which varied depending on the wax. Lower intensities of the FTIR peaks with annealed samples tended to confirm a phase transition of the waxes, which also helps explain the different roughness patterns observed on the fiber surfaces.



The mechanical strength of these prepared samples was assessed via DMA analysis, and a reduced tan delta signal in the temperature range leading up to the secondary alpha transition temperature for paper (118°C) was observed with BW/CW impregnated samples, tentatively confirming additional hydrogen bonding between the wax mixture and cellulose. In the temperature degradation range for paper, those impregnated and annealed without wax to generate a background sample had a sharp reduction in storage modulus occurring up to 40°C lower than equivalent samples with wax-impregnated papers. This dramatic improvement in the temperature range of degradation was thought to be due to the additional strength created by hydrogen-bonds between the waxes and cellulose.