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SANS experiments on wood during pretreatment in ionic liquid/water mixtures

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Cellulose makes up for most of the material in the lignocellulosic's cell wall, and it could provide an abundant source for fuels, materials and chemicals. Mild and selective conversion processes would be desirable for decentralized value-generation from the synthesis power of nature. However, the utilization is still difficult due to the composition and the structure of the biomass'cell wall. Cellulose shows a dense, crystalline structure and the access to these macromolecules is further restricted by lignin and hemicellulose. An efficient conversion hence requires the application of a pretreatment to gain access to cellulosic macromolecules for subsequent conversion processes.

Mechanistic understanding of the pretreatment can likely be gained at the molecular level. However, the cellulose in the cell wall exists in fibrils made of several cellulose chains, which are hold together via intermolecular hydrogen bonds. This regular arrangement forms crystalline structures that are a major obstacle in enzymatic hydrolysis [1]. Hence, molecular analysis needs to be extended by structural analysis to monitor the mechanistic steps of pretreatment.

Ionic liquids proved to be good solvents for the cellulose and the hydrophobic lignin [2], and the high concentrations of ions at elevated temperatures around 100℃ give rise to chemical reactions that constitute the desired pretreatment and improve the enzymatic hydrolysis [3]. Due to the abundance of water in such processes, we systematically studied the effect of water on this pretreatment. Using small angle neutron scattering (SANS), the tissue after the pretreatment was compared to the native wood and a first time-resolved setup was established for this pretreatment.

At higher water contents, the crystallinity is enhanced, and the cell structure is rather preserved but cellulose fibrils show coalescence similar to what has been observed after pretreatment with dilute mixtures. In concentreated EMIMAc, the crystallinity of the cellulose is decayed, and the cell structure of the wood is rather destroyed [4]. The results indicate extraction and reorganization on the scale of macrofibrils, which is supported by other analytical methods supporting the results. A latest kinetic SANS study completes the whole picture drawn here.

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