INDUSTRIAL PILOT PROJECT

Nanofibrillation of chemically modified cellulose fibres studied through in-situ synchrotron x-ray scattering

THE INDUSTRIAL CHALLENGE

BillerudKorsnäs and KTH have developed a technology where chemically modified cellulose fibres fibrillate to cellulose nano-fibrils (CNFs) at increased pH. To develop proper large-scale processes, it is important to understand the phenomenon from the microscale down to the nanoscale in terms of structure, mass transport and kinetics.

WHY USE A LARGE SCALE FACILITY?

The structure before and after fibrillation can be observed in table-top x-ray scattering instruments, but not the time-resolved changes on the millisecond or second scale. To achieve this time resolution, much more focused and intense x-rays are needed in combination with *in-situ* flow mixing, which is only available at synchrotron facilities.

HOW THE WORK WAS DONE

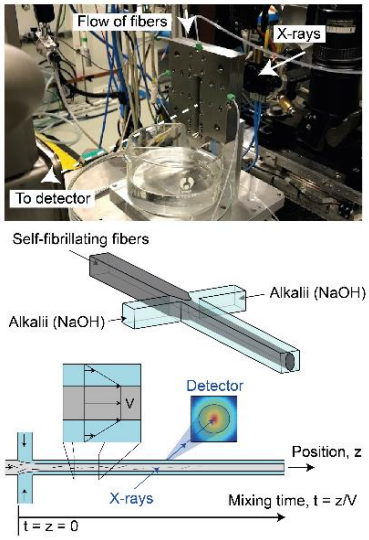


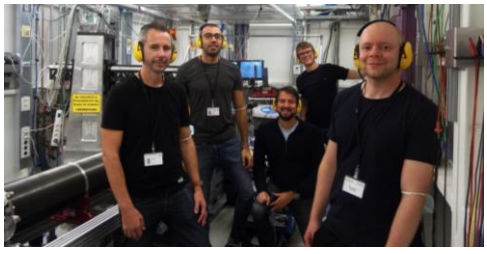
Figure 1: Setup used at the synchrotron

To study structural changes on short timescales, the chemical fibrillation was performed in a flow-focusing mixing cell (Fig 1). The hydroxide ions in the focusing flows diffuse into the core flow to initiate fibrillation. Given certain flow rates, the time instance of the fibrillation process is determined by the downstream position. By studying the scattering intensity at defined positions, i.e. different times, the structures at various stages of the process could be determined on spatial scales of 1–100 nm.

The experiments were performed at PETRA III (DESY, Hamburg) at the P03 beamline, headed by Prof. Stephan Roth. The team was also assisted by beamline scientist Dr. Matthias Schwartzkopf. The choice of going to DESY was mainly due to the beamline's excellent capabilities to simultaneously study structures ranging from atomic scale up to hundreds of nanometres using a combination of detectors for both small- and wide-angle x-ray scattering (SAXS/WAXS).

THE RESULTS AND EXPECTED IMPACT

During two days of beam time, a wide variety of experiments were performed, including variations of NaOH concentration and flow rates. The scattering patterns were compared with theoretical patterns based on hypothesised fibrillation scenarios. Unfortunately, none of these scenarios were easily distinguished in the experimental data. This was probably due to too high content of already liberated CNFs, masking the desired observations, and too high signal-to-noise ratio, *i.e.* dilute systems as ours would benefit greatly from yet lower background scattering. Nevertheless, from the perspective of being an initial experiment of this type of *in-situ* study, the beam time was a great success and the results will provide invaluable input for designing a follow-up experiment to truly reveal the fibrillation kinetics on a nanoscale.



"A great and very safe experience during intense and insightful days in Hamburg***"***
/ David Sandström, BillerudKorsnäs

BILLERUDKORSNÄS

KTH VETENSKAP OCH KONST

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Vinnova's project No: 2019-05273 **Duration:** February 2020 – December 2021

Funded by Sweden's Innovation Agency, Vinnova, in order to build competence and capacity regarding industrial utilisation of large-scale research infrastructures such as MAX IV and ESS.