INDUSTRIAL PILOT PROJECT

In situ monitoring of cellulose dissolution using a combination of synchrotron SAXS and WAXS

THE INDUSTRIAL CHALLENGE

The environmental impact of textile fibers is a growing concern and there is a great need to replace cotton and synthetic fibers with a more sustainable option. Tree to Textile (TTT) has developed a new cellulose-based textile fiber, produced via a sustainable process free from toxic chemicals. TTT is also reviewing alternative cellulosic raw materials from agricultural waste and recycled materials. When developing man-made fibers it is crucial to understand all the process steps, from dissolution to fiber spinning and post treatment. It is known that the raw material has a great impact on the dissolution behavior of the spin dope. However, the dissolution is a very quick process (within minutes) and is thus particularly challenging to monitor with conventional characterization methods.

WHY USING A LARGE SCALE FACILITY

X-ray scattering is a powerful tool for analyzing ordered structures in materials. Thus, X-ray scattering has the potential to follow the disintegration of ordered polymer structures into randomly molecularly dispersed polymers in the cellulose dissolution process. The high flux X-ray at synchrotron radiation facilities allows us to capture the disintegration of cellulose in high temporal resolution during dissolution close to the real processing condition, while lab-based X-ray equipment is much slower owing to the lower flux. A simultaneous *in situ* small angle X-ray scattering (SAXS) and wide angle X-ray scattering (WAXS) experimental setup allows us to probe the nanoscale gel networks and atomic arrangement, respectively, on the same material at the same time.

HOW THE WORK WAS DONE

Dissolution of cellulose was monitored in a stationary system, which was set up with the help of the beamline scientist, Dr. Xiao Sun. Cellulose from different sources was mixed with solvent in capillaries. The SAXS/WAXS experiments were conducted at the P62 beamline of Petra III, Hamburg, using an integrated Linkam stage to cool the capillary with a 2 °C/min temperature cycle from +25 to -30 °C and up to +25 °C again. The precise control of the temperature is crucial for this particular dissolution process. To minimize radiation damage, an absorber was placed in between the beam and the sample. This reduces the total flux but still allows good signal-to-noise of scattering data with 1s exposure.

THE RESULTS AND EXPECTED IMPACT

The SAXS measurements, which gives information about ordered structures within the material in the nm range, indicated initial swelling of the different pulps as well as the final state of the cellulose solution, i.e. presence or absence of cellulose aggregates. With WAXS, which measures structural units in the Å range such as interplanar distances in crystallites, it was possible to follow the kinetics of disintegration of the cellulose crystallites for different pulps and solvents by extracting the Bragg peaks via curve fitting. Some samples showed no crystalline signal after the cooling cycle which indicated that cellulose was dissolved. However, simultaneous SAXS measurements could reveal that aggregations were present, thus showing that some cellulose was not completely dissolved or had formed a gel network. Consequently, the combination of synchrotron SAXS/WAXS is a powerful tool to acquire information on the progression of cellulose dissolution.

Figure. Mounting of a capillary in the Linkam stage.

"It has been incredible to come here and see how such a large facility actually works. I feel that I have gained a better understanding of what types of experiments that are possible, what questions could be answered, and I will take that with me in my further work"
/Åsa Östlund, TreeToTextile

Contacts: Åsa Östlund – Tree to Textile, asa.ostlund@treetotextile.com
Shun Yu – RISE Research Institutes of Sweden, shun.yu@ri.se

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