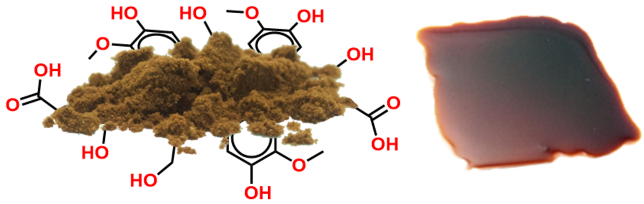
INTERNAL AFFAIRS OF A BIOPLASTIC

Investigation of the molecular structure of a lignin-based polymer

Lignin is one of the most abundant organic compounds on Earth – the one that makes plants "woody" by glueing together cellulose fibers. In paper production, where cellulose is extracted from wood in the so-called Kraft process, lignin occurs as a by-product en masse. It was long regarded as waste and, at best, incinerated as a fuel. However, this may be about to change. In the search of new, sustainable materials, lignin has turned out to be a promising raw material for the production of bioplastics – such as thermosets, materials that irreversibly harden upon heating. Unlike oil-based compounds, lignin compounds are very heterogenic, however. Researchers therefore have to develop procedures for making thermosets with specific properties. Such research requires the knowledge of the materials' molecular structure and how it relates to its macroscopic properties.



Chemical structure and physical appearance of lignin (left) and a thermoset (right) that can be made from it.
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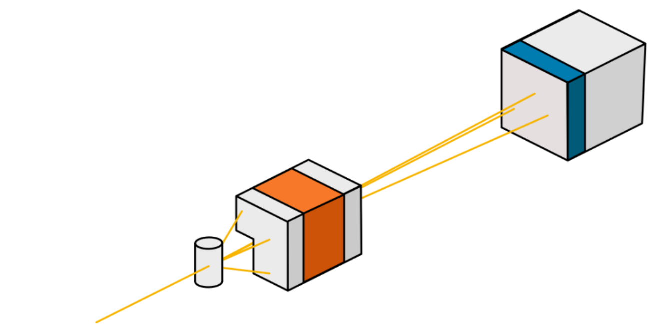
EXPERIMENTAL SETUP

Setup	PETRA III, DESY (Germany), P03 beamline
Camera	LAMBDA 4.5M Si detector
Resolution	4,718,592 pixels
Acquisition frequency	10 Hz
Photon energy	11.7 keV

In collaboration between the KTH Royal Institute of Technology (Sweden) and DESY (Germany), an international team of researchers investigated different lignin-based thermosets in detail. First, they chemically separated the raw material – Kraft lignin – into different fractions. From this, three thermosets were produced using three different cross-linkers – chemical agents that connect the macromolecules to form a three-dimensional network. To determine the internal molecular structure of the thermosets, the researchers set up an experiment at the P03 beamline at PETRA III (DESY). They used a combination of small- and wide angle X-ray scattering techniques (SAXS and WAXS). The WAXS signal was recorded with a LAMBDA 4.5M camera.

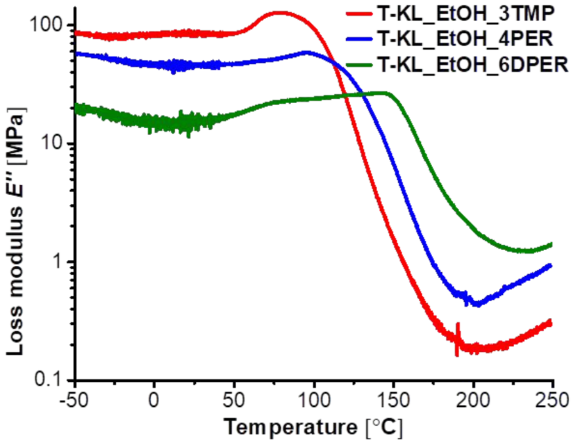
Stephan V. Roth, who is one of the authors of the study, explains the advantages of the chosen detector: **"The small pixel size of 55 µm enables high resolution in q-space. Single photon counting capability enables a very low noise level and straightforward background corrections. This is needed for analyzing the WAXS peaks from low-Z materials, such as lignin, and was essential for the success."**



Setup of combined WAXS/SAXS experiments with two different detectors. The L-shaped LAMBDA 4.5M camera with 55 µm pixels was used to detect the WAXS peaks.

RESULTS

With their X-ray analysis, the researchers characterized the molecular structure, or molecular morphology, of the three thermosets. At the nanoscale, the lignin components are either arranged in a T-like structure or in a sandwich structure. In the present case, the sandwich structure was found to be predominant – similarly for all three thermosets. However, their properties upon heating, which were studied with an array of additional techniques, differed. Thus the distribution of the different cross-linkers must be responsible for the differences in behavior, not the morphology. With their collaborative study, the team showed that despite the heterogeneity of lignin, it is possible to produce thermosets with tunable properties.



Loss modulus, a property describing the elastic properties of a polymer, of the three different thermosets upon heating ("thermal curing").

REFERENCE

- Iuliana Ribca, Marcus E. Jawerth, Calvin J. Brett, Martin Lawoko, Matthias Schwartzkopf, Andrei Chumakov, Stephan V. Roth, and Mats Johansson (2021): Exploring the Effects of Different Cross-Linkers on Lignin-Based Thermoset Properties and Morphologies. ACS Sustainable Chemistry & Engineering 9 (4), 1692-1702. https://doi.org/10.1021/acssuschemeng.0c07580

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