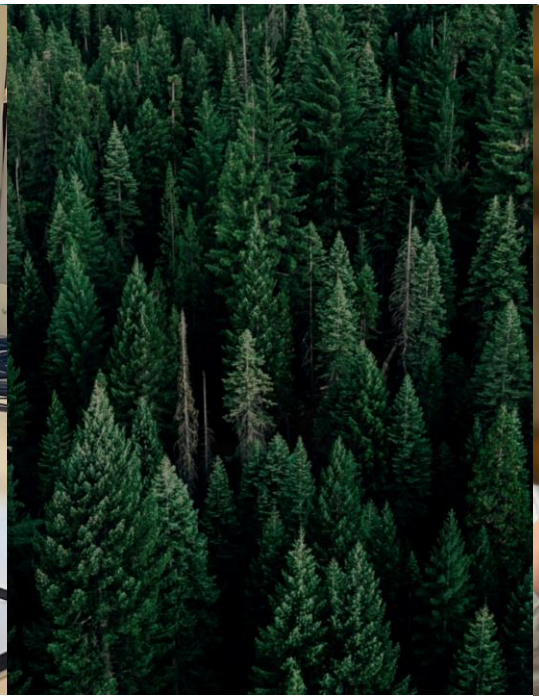
WWSC

WALLENBERG WOOD SCIENCE CENTER

Wood nanotechnology – new materials from trees


Annual report 2022

KTH VETENSKAP OCH KONST CHALMERS LINKÖPINGS UNIVERSITET TREESEARCH Knut och Alice Wallenbergs Stiftelse

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Wallenberg Wood Science Center

Wallenberg Wood Science Center (WWSC) was first launched in 2009 as a collaboration between KTH Royal Institute of Technology (KTH) and Chalmers University of Technology (Chalmers), eventually also with participating researchers from Stockholm University (SU), Luleå Technical University (LTU) and Umeå University (UmU). The second phase of WWSC was launched in January 2019 and also brought about that the Laboratory of Organic Electronics (LOE) at Linköping University (LiU) became a center member. As of the end of 2022, more than 60 PhDs have completed their doctoral training within the framework of the center. According to the Web of Science (2023-06-14), the affiliations “Wallenberg Wood Science Center”, “Wallenberg Wood Science Centre”, or “WWSC” have gathered >35,000 citations. The center’s h-index is 86.

The funding for 2022 was 72 MSEK, the major share being a generous donation from the Knut and Alice Wallenberg (KAW) foundation (40 MSEK), the participating universities (22 MSEK) and the industry via Treesearch (10 MSEK). The research goals are set in a long-term perspective, and the center agreement signed by the funding organizations is for 10 years, ending in 2028. Currently, (December 2022), the center engages ~70 faculty members/researchers, ~25 postdocs and ~50 PhD students.

The research in the center has a focus on new materials from trees. The aim of WWSC is to create knowledge and build competence that has the potential to form the basis for an innovative future value creation from forest raw materials by developing methods and processes that provide molecular and structural control. The scientific activities have two main objectives; the first is on fundamental understanding of wood tissue, wood fibers, cellulose, hemicelluloses, lignin and related components, including bio-based polymers. This includes extraction, disintegration, purification processes and their mechanisms, characterization of biomolecules, nanocelluloses, fibers, colloids etc., as well as novel modification routes and biopolymer synthesis. The second objective is new material concepts, where the wood material components (fibrils, fibers, wood veneer, lignin polymers, wood-based colloids etc.) are combined with other constituents and assembled into materials and devices. Research activities in WWSC span broadly from refining of wood and wood components, via extraction/fractionation of biopolymers and other constituents in wood to the utilization of wood polymers and other constituents in advanced nanotechnological devices with potential use in energy applications or electronics.

To capture and nurture the full breadth of the activities in WWSC, the center is organized into five highly integrated programs which are led by an experienced researcher in each field (program responsible in brackets).
Program 1: Wood components – extraction, characterization and properties (Prof Lisbeth Olsson, Chalmers)
Program 2: Biobased polymers and modelling (Prof Eva Malmström, KTH)
Program 3: Fibers and fiber nanotechnology (Prof Lars Wågberg, KTH)
Program 4: Composites for energy and electronics (Prof Mats Fahlman, LiU)
Program 5: Biocomposites and wood materials (Prof Lars Berglund, KTH)

WWSC 2022

2022 was the year when the pandemic finally ceased, and everyday operations reverted to what they used to be prior to Covid-19. Travelling picked up, research exchanges reinitiated, virtual conferences were replaced by physical meetings, and the WWSC Academy Schools and WWSC Workshops could finally be organized as physical meetings. Center activities were brimming like never before, ~70 researchers, ~30 postdocs and ~50 PhD-students were all contributing to the ongoing research efforts.

The workshop in June was organized in the picturesque setting of Marstrand in the archipelago outside Gothenburg. The majority of speakers were invited guests from the USA and Europe and were either physically present or participated in ZOOM. One session was devoted to a workshop in Research and Innovation which was led by Sandor Albrecht from the Wallenberg Launch Pad-Program (WALP) supported by KAW, which turned out to be successful since at least one more company (Proligreen.com) was spun out as a direct consequence of this workshop. Professor Ingo Burgert, one of the members of the WWSC Scientific Advisory Board (SAB) participated and gave a very much appreciated lecture.

In November, the Winter Workshop was held at the Vildmarkshotellet in Kolmården and gathered more than 130 participants. The meeting hosted a combination of internal and external speakers. Innovation and Entrepreneurship were on the agenda also for this meeting, now with a more direct focus on WWSC-related aspects provided by Magnus Wikström at MaWi development. Both professor Harry Brumer (Uni British Columbia) and professor Robert Pelton (McMaster University) from WWSC SAB contributed with very much appreciated lectures.

2022 marked the onset of a new era since ForMAX, the beamline at MAX IV specifically designed for fiber-based materials, was taken into operation. ForMAX brings about new possibilities to assess the nanoscale structure of lignocellulosic materials using scattering techniques such as SAXS/WAXS and tomography. It will be very interesting to follow future developments in this area and to learn more about the nanoscale structure of wood-based materials that can pave the way for completely new materials from wood.

It is also impossible to summarize 2022 without highlighting all the excellent researchers, including PhD students and postdocs, associated with WWSC. Their dedication and hard work are reflected in the large number of high-quality papers that have been published during the year, numerous conference publications, by the ongoing work and not least by the prizes, grants and other achievements that have been won in fierce competition. I want to extend my sincere appreciation to all of you for your search for fundamental understanding and novel findings that make it possible to develop extraordinary materials from wood-based resources.

In the following pages, please find a mini overview of the research focus in each of the WWSC programs.

WWSC Program 1: Wood components – extraction, characterization and properties

Active PIs

Chalmers: Prof Lisbeth Olsson – Program 1 responsible, Assoc prof Johan Larsbrink, Prof Anette Larsson, Prof Lars Evenäs, Prof Hans Theliander, Assoc prof Merima Hasani, Prof Eva Olsson, Prof Gunnar Westman
KTH: Prof Monica Ek, Assoc prof Martin Lawoko, Assoc prof Francisco Vilaplana, Assoc prof Olena Sevastyanova, Assoc prof Lauren McKee
LiU: Prof Xavier Crispin, Dr Viktor Gueskine

Overview of the activities within the program

The scientific work in Program 1 aims at providing a deep insight into structural and processing characteristics of wood and bark components, the main focus being on complexity of their molecular, nano- and macroscale interactions pertaining to the recalcitrance during decoupling and valorization in general. Moreover, their structural changes upon processing, whether pre-treatments, separations or fractionation and functionalization, are poorly described and pose additional challenge on design of biorefining concepts.

One part of the program focuses on decomposition and separation. For studying fouling of microcrystalline cellulose in membrane filtration (polyethersulfone membranes), we combined fluid dynamic gauging (FDG) and molecular dynamics (MD) (1.1.1a). Furthermore, the filter cake formation in electro-assisted filtration of MFC (important in dewatering) and its dependence of filtration conditions was studied (1.1.1 b). Mass transfer challenges in wood decomposition was approached using earlier built diffusion cells, combined with QCM-D measurements. The study mapped the influence of ionic strength and salts on the diffusion of lignin through cellulose fibers (1.1.2a). Certain cell wall cellulose motifs are insoluble in water-alkaline systems and new dissolution strategies are under development (1.1.3a). As a basis, methodology for studies of crystallinity and ultrastructure were developed and applied to recalcitrant fractions of the biomass.

Another part of the program targets lignin applications. To be able to use lignin targeted for different applications, a deeper knowledge on the chemical, structural and material properties of different lignin preparations needs to be acquired. Colloidal lignin particles were produced from hardwood and softwood kraft lignins (1.2.1). These particles had a size of 150-200 nm and their morphological and cytotoxic properties were assessed aiming at investigating the applicability in cosmetic and pharmaceutical sectors. Another possible application of lignin is based on its functionality in UV-protective coatings. In 1.2.1b, the influence of natural or chemically modified lignin from different sources on the UV-protective properties of polymer coatings were investigated. It could be concluded that methacrylated lignin is a promising environmentally friendly bioadditive for UV protection of polymer coatings. To address some shortcomings in Zn-ion aqueous batteries, lignin-carbon cathodes were evaluated. Good performance in hybrid electrolyte suitable for mono- and divalent ion batteries like Li-ion, Zn-ion, Ca-ion with lignin-based cathode materials was demonstrated (1.2.2a).

A novel biorefinery process aiming at extracting high quality lignin exhibiting well-characterized structures was established. Analytics combined with chemometrics demonstrated that the chemical properties can be tailored (1.4.1a). To gain fundamental understanding of variation

in lignin structure and composition, lignin was extracted from spruce (softwood) and birch (hardwood) (1.4.1b). Structural differences were found, and they were mapped towards their influence on the extractability of lignin.

Enzymes can play an important role in cleaving specific chemical bonds in biomass streams or in modifying biomass derived molecules and polymers. Project 1.3.1a focused on discovery and characterization of the functionality of glucuronoyl esterases (GE-cleaving bonds between hemicellulose and lignin). The study spread light on the diversity of GE action. Galactose oxidases (GalOx) are enzymes able to convert the O6 hydroxyl groups of galactose moieties to aldehydes. Bacterial enzymes from the Auxiliary Activities family 5 (AA5) where GalOxs are found were characterized (1.3.3). Enzymes that differed significantly in protein sequence from previously studied enzymes were targeted. Surprisingly, in screening 40 different substrates, the selected enzymes were primarily active on smaller primary alcohols.

LPMO (lytic polysaccharide monoxygenases) is a group of enzymes that oxidatively cleave cellulose and hemicellulose. We have detailed the functionality of LPMOs on xylan and showed that the substitution groups on the xylan backbones affect the activity of LPMOs (1.3.1b). Removal of some xylan substitutions led to the identification on previously unknown xylanolytic capacities of LPMOs. In addition, we could also demonstrate that LPMOs increase the hydrolysability of mildly steam pretreated spruce by acting on both cellulose and xylan. Several hemicelluloses were purified, and their chemical composition were determined. Details on the interaction with cellulose were studied using molecular dynamic simulations (1.4.2). Such information helped in explaining LPMO action on xylans. Furthermore, applying novel methodology (fractionation and NMR analysis) allowed studies of xylan structure and its solubility.

Valorization of suberin was investigated by applying cutinases that can break the bond between the valuable fatty acids in suberin and phenolics (1.3.2) and the release of fatty acids were demonstrated. Carbohydrate binding modules (CBMs) were explored to crosslink polysaccharides (1.3.1c). Twelve different CBMs was shown to induce gel formation and it was in addition demonstrated that gel properties were CBM dependent.

Using DNP solid NMR, the sensitivity of NMR measurement increased significantly. By developing new methodology, studies of heterogeneity in biomass could be performed (1.1.2b). Structural insights of biomass were reached by developing and applying SEM, FIB-SEM and TEM (1.3.2b). This approach led to insights of the biomass nanostructure. An establishment of a Terahertz characterization platform (1.5.2), complementing earlier analytical capacities and the technique enables studies of weak hydrogen bonds. The technique was applied to distinguish cellulose I and II as well as for studies of weak hydrogen bonds on the surface of nanocellulose. A new project (1.1.2 a) aims at building a data library of lignocellulosic derivates and combined with AI methodology allow prediction of thermoplastic properties.

Two WWSC spin-off companies; Proligreen AB and Glycolink AB were established.

WWSC Program 2: Bio-based polymers and modelling

Active PIs

KTH: Prof Eva Malmström – Program 2 responsible, Prof Monica Ek, Prof István Furó, Prof Minna Hakkarainen, Prof Mats Johansson, Assoc Prof Martin Lawoko, Assoc Prof Lauren McKee, Prof Karin Odelius, Dr Linda Fogelström, Assoc Prof Per-Olof Syrén, Prof Francisco Vilaplana, Assoc Prof Jakob Wohlert
Chalmers: Prof Anette Larsson
Linköping University: Prof Mats Fahlman

Overview of the activities within the program

Program 2 is focusing on all biopolymers which can be isolated from wood except (nano)cellulose which is covered by Program 3, as well as on bio-based polymers attainable by polymerization of low molar mass extractables or degradation/fractionation products by sustainable methods, all in collaboration with Program 1.

Program 2 aims at:

- gaining fundamental understanding on why biopolymers are sensitive to humidity or water which may hamper their potential applicability in various material applications,

- designing new sustainable materials from bio-based raw materials using as benign chemistry as possible and green chemistry principles,

- elucidating the fate of cellulose- (biopolymer-) based materials at the end-of-life.

The rationale for the design of novel materials and the potential material applications are explored in collaborations with Programs 3–5. As Program 2 has developed, it is now obvious that several of the projects are aiming to develop a material that has a potential to substitute a fossil-based counterpart, or that can function as an additive to improve the properties of an already existing material. However, an overarching common denominator is the ambition to gain fundamental understanding from the nano (molecular) level to macroscopic properties.

The effects of water in cellulosic biomaterial are ubiquitous but the molecular origins of those effects remain obscure. A central question is the effect of hydration on both micro- and macrostructure, as well as on segmental dynamics. Wohlert, Furó and co-workers have used molecular dynamics computer simulation of amorphous xylan models to study local structure and dynamics as a function of moisture content. Such fundamental understanding will support the interpretation of experimental results and may also provide guidance for how to design novel materials containing xylan or related compounds.

The interest in lignin has skyrocketed over the last decades as it is a large source of biogenic aromatic carbon which may find substantial use in a broad range of applications. Since long, WWSC has developed expertise in how to refine wood fibres to retrieve lignin and has developed characterization methods, modification pathways and curing chemistries to make lignin suitable for transformation into thermosetting applications, mainly in thin films. Research has been conducted to understand the structure-property relationship between technical lignin structure and the final thermoset performance. Benign methods for further functionalization of the lignin skeleton, such as allylation using diallyl carbonate, have been demonstrated. The

effect of using microwaves for both fractionation and functionalization has been investigated. Interestingly, the addition of epoxidized lignin to a Novolac-resin was found to improve the adhesion strength to a metal substrate as well as the corrosion resistance. It has been demonstrated that a thermoplastic material with a high lignin-content can been obtained by scalable reactive extrusion methods. Furthermore, it has been demonstrated that solvent fractionated LignoBoost Kraft lignin can be successfully used in a binary cathode interface layer (CIL). The biobased CIL holds an advantage over commercial materials by a superior performance (stability and sustainability).

It has been demonstrated that hemicelluloses, in combination with another biopolymer, can be used in wood adhesives to give rise to bond lines of sufficient strength and water resistance. However, the fundamental understanding has still been limited. By using a hemicellulose model (locust bean gum) it was possible to investigate how the adhesive performance is dependent on the molecular weight; it can be concluded that there is an optimum molecular weight; where bond performance is satisfactory, and the viscosity is not too high/low.

Functional bis-vanillin monomers were explored for thermoplastics and covalent adaptable networks utilizing polyimine condensation with a series of bis and tris amines. A fatty acid-based amine and a rigid short tris amine have been developed and are currently further characterized.

The action of a class of polyesterases (PETases) on degradation polyesters was demonstrated on wood-derived polyesters. Interestingly, it was found that the PETase enzymes could depolymerize polyester-imines containing Schiff bases, which may hold significant promise in the search for sustainable polymers that have low, or even no, capacity to accumulate in nature or in the marine environment.

Building on prior research findings, triblock copolymers comprising polyesters and polycarbonates were designed to have a soft middle segment so as to provide desired mechanical properties and to ensure a high thermodynamic equilibrium during chemical recycling to monomer (CRM). The soft segment was found to have a significant impact on both the mechanical properties and the CRM.

Since the start of WWSC 2.0, well-defined cationically charged core-shell nanoparticles obtained through reversible addition-fragmentations chain transfer (RAFT)-mediated polymerisation-induced self-assembly (PISA) in water have been designed and synthesized targeting surface engineering in bio(nano)composites. The synthetic procedure by which the NPs is made is robust, allowing for a fantastic freedom in choice of building blocks and thereby allows for the synthesis of a plethora of compounds with tailorable properties. The well-defined character of the synthesized nanoparticles also makes them useful as well-defined colloids.

It has been found that cellulose oxalate is formed when nanocellulose is derived from Norway Spruce using oxalic acid. It forms a Pickering emulsion which may have a potential to be used in foodstuffs and cosmetics.

WWSC Program 3: Fibres and fibre nanotechnology

Active PIs

KTH: Prof Lars Wågberg – Program 3 responsible, Adj prof Tomas Larsson, Assoc prof Torbjörn Pettersson, Prof Daniel Söderberg, Prof Michael Malkoch, Prof Fredrik Lundell
Chalmers: Assoc prof Merima Hasani, Prof Gunnar Westman
LiU: Igor Zozoulenko, Prof Xavier Crispin
Stockholm University: Prof Aji Mathew, Prof Lennart Bergström, Assist prof, Mika Sipponen

Overview of the activities within the program

The continued focus areas in Program 3 have been the liberation and chemical modification of the fibrils in the fibre wall of cellulose-rich fibres and the development of filaments and fibril-based materials with a controlled structural organisation of the fibrils. In addition, we have had a start-up this year of a new project headed by Mika Sipponen, SU, on lignin nanoparticles and their composites as functional materials which was possible since one of Professor Lennart Bergströms projects with Dr Pierre Munier was successfully finished during 2021.
The liberation of fibrils within the fibre wall using new approaches has been continued during the year and by exposing the fibres to extremely high pressures pulses with short duration it has been shown that the fibre walls are significantly affected and that both the crystallinity and the size of the crystallite of the cellulose are decreased. The studies of the interactions between the fibrils in the fibre wall during water removal and drying has also been continued. Results show that the hornification of cellulose-rich materials is a kinetically locked state and that this seemingly irreversible process is diffusion controlled. This diffusion can either be diffusion of water molecules out from the contact zone or a diffusive mixing of molecules from adjacent cellulose surfaces. The work with chemical modification of fibres and the fibrils within the fibre wall has also continued in several projects. In one of the projects ZnO nanowires have been grown on macroscopic fibres before the preparation of a paper from these modified fibres. The so produced paper has photocatalytic properties and by for example dipping the paper in water and exposing it to UV-light it is possible to reduce dissolved oxygen in the water to H_2O_2. In another project a new family of functional and dendritic-linear-dendritic block-copolymers with excellent hydrolytic stability has been developed. These polyelectrolytes have then been mixed with cellulose fibrils to form stable hydrogels and aerogels which have excellent antibacterial properties. In another project the fibril surfaces have been fitted with methacrylic acid groups that can be reacted with functional monomers or oligomers to create inter-fibre composites with excellent mechanical and water holding capacities. In the latest development, bifunctional PEG-based copolymers have been used to form gels with the methacrylic acid-functionalized CNFs. The so-formed gels have a 100 % shape recovery after drying and rewetting which implicates that the irreversible cellulose interaction during drying can be avoided with this treatment. The work with a covalent tailoring of CNCs for different end-use applications has also continued and a new tool-box for different end-use purposes has been developed. A series of CNC derivatives were developed and the modification that led to the formation of chiral nematic water dispersions also resulted in the development of double-curved surfaces upon drying. The addition of ionic or hydrophobic substituents to the CNCs also allowed for the development of triboelectric nano-generators from films made from the CNCs. The work with the development of methylmorpholine-based solvents for the dissolution of cellulose in aqueous, alkaline media has also progressed during the year. Both microcrystalline cellulose and macroscopic cellulose-rich fibres have been dissolved at room temperature using these new solvents. The use of CO_2(g) as a possible coagulant has also been studied.

The work with theoretical modelling has also continued during the year. In Norrköping the development of models for nanocellulose and modified nanocelluloses has continued and the influence of both carboxyl groups and/or the introduction of dialcohol cellulose on the interaction between these materials and different solvents have been evaluated. These models have also been used in a number of co-operations throughout program 3 and outside program 3 where the macroscopic properties of nanocellulose materials could be linked to the molecular interactions that could be simulated with the developed models. As such, this work with modelling has functioned as a natural “glue” between different branches of WWSC 2.0. At KTH the work using coarse grain modelling of anisotropic, dilute fibrillar dispersions has continued. Based on these results it has been possible to develop a new scaling law for the rotational diffusion of semiflexible nanofibers in entangled systems which is extremely relevant for the earlier studies in the program where the colloidal stabilization effect of these fibrillar dispersions was established, even at very low fibril concentrations.

In the second part of program 3 the focus has been on controlling the fundamental interactions between nanocellulose materials and other nanoparticles and with polymers/polyelectrolytes using a bottom-up engineering procedure. The work on fundamental colloidal chemical interactions between nanocellulose materials have been continued during the year. The factors controlling the formation of Volume Spanning Arrested States (VASs) have been identified for different types of CNF and it has been possible to determine how the CNFs can be organized by using different methods. It has for example been shown that the VASs can be formed at such low concentrations as 0.2 g/l and we have also been able to form Layer-by-Layer supported microchannels within these VASs which allows for the transportation of liquids/solvents within these extremely water-rich hydrogels. By controlling the CNF interactions in flow-focusing using specially designed dendritic polyampholytes together with the CNFs it has been possible to tailor both the stiffness and the toughness of the formed filaments. This indeed shows that the flow-focusing-assisted self-assembly in combination with tailored polymers/polyelectrolytes is a very promising future technology. High resolution X-Ray scattering techniques in combination with rheological measurements have also been used to study the orientation of CNCs, montmorillonite (MMT) platelets and combinations of the two under different shear conditions. The results show a clear synergistic orientation of the two components that could be followed both by X-Ray scattering and rheological investigations. The self-assembly of these two components will have a huge impact on the macroscopic materials made from CNC and MMT. The bottom-up engineering using CNFs has also been taken one step further by combining TEMPO-oxidized CNFs (TCNFs) and chitin nanofibers as reinforcing elements in PLA-based composites. These composites were then the extruded and 3D-printed into functional filters for water purification.

In a newly initiated project, freshly prepared dispersions of lignin nanoparticles were used to prepare photonic crystals. By using a centrifugation-assisted size classification it was shown possible to prepare close-packed lignin structures with different photonic properties from the initially polydisperse nanoparticle dispersion.

Many of the projects within program 3 have also used high resolution X-Ray scattering techniques to study structure, dynamic structure formations and to resolve changes in crystalline ordering during treatments of cellulose-rich materials. This is a very satisfactory development especially now when the FORMAX beamline in Lund is ready for use.

WWSC Program 4: Composites for energy and electronics

Active PIs

LiU: Prof Mats Fahlman – Program 4 responsible, Assoc prof Simone Fabiano, Prof Magnus Jonsson, Assoc prof Renee Kroon, Prof Daniel Simon,
Chalmers: Prof Per Lundgren, Prof Aleksander Matic, Prof Christian Müller, Assoc prof Tiina Nypelö, Prof Hans Theliander

Overview of the activities within the program

Combining forest-based fibers and bulk systems with functional compounds enable a wide range of energy, electronic, photonic and electrical applications. In Program 4, we try to explore and advance along this pathway by introducing, e.g., p-type conducting polymers, n-type conducting polymers, carbon(ized) materials, silicon dioxide microparticles and metallic materials in order to obtain various active properties on the material level. In addition, we explore a wide range of engineering techniques and production protocols to manufacture fibers, scaffolds and substrates then targeting a specific device or system needed for dedicated applications. The results of WWSC Program 4 in 2022 are summarized below.

Building on the recent development in WWSC of processable n-type organic conducting ink, several composite materials have been fabricated and deployed in a variety of applications. A cellulose/conjugated polymer-based n-type yarn for thermoelectric textiles was developed in a joint project involving Chalmers and LiU as a power source for wearable electronic devices. A regenerated cellulose yarn was spray-coated with a conducting polymer-based ink composed of BBL and PEI (see Fig. 1). A prototype in-plane thermoelectric textile, produced with the developed n-type yarns and p-type yarns, the latter composed of PEDOT:PSS coated regenerated cellulose, displayed a stable device performance in air for at least 4 days. In another project, 3D-printable BBL- and cellulose-based hydrogels were developed as electrode materials for cell-growth and bio-interfacing scaffolds.

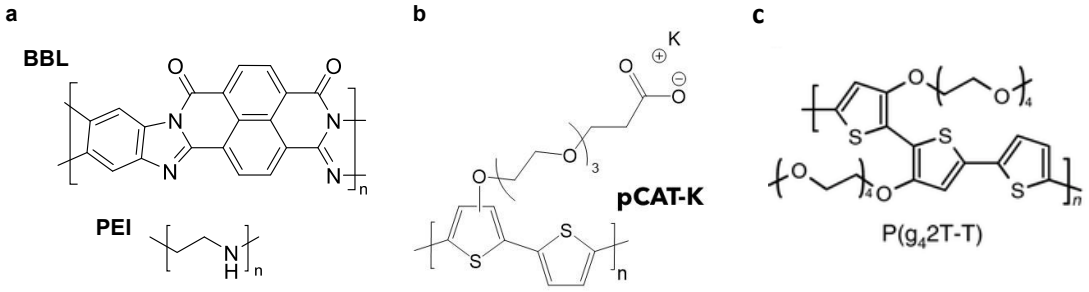

Figure 1. Chemical structures of a) BBL and PEI; b) PCAT-K; c) P($g_4$2T-T).

Another research direction has been the use of polar side chain functionalized organic semiconductors (p($g_4$2T-T) and PCAT-K) in cellulose composites/coatings. CNF:P($g_4$2T-T) nanocomposites were prepared and provided an excellent electrochemical response but also displayed a reversible reduction of its elastic modulus from E >> 100 MPa to about 10 MPa, making them promising materials for wearable electronics and bioelectronics. The PCAT-K polymer was designed with the aim of creating recyclable electroactive cellulose coatings,

where it was demonstrated that the PCAT-K could be easily fixated onto and recovered from cellulose substrates via simple acid-base chemistry.

Cellulose-based materials have been explored for several different energy-related applications, such as the use of cellulose separators as well as sulphated cellulose nanocrystals and sodium carboxymethyl cellulose additives to improve electrochemical performance in Zn metal batteries, and electrospun lignin carbon fibers as supercapacitor electrodes. Several projects involving LiU and KTH were devoted to developing energy-regulating surfaces using cellulose-conducting polymer combinations, creating materials with opposite behaviour in terms of their radiative cooling and solar heating capabilities (see Fig. 2). This enabled the design of a cellulose solar heater with suppressed radiative cooling thanks to a coating that reflects infrared light (low cooling) while it transmits visible light (to allow solar heating). Such materials were used to create a thermal gradient and drive a lateral ionic thermoelectric device by being expose to the sky, both during daytime and night-time. Another exciting example is the development of a cellulose materials capable of self-adaptive solar heating and evaporative drying.

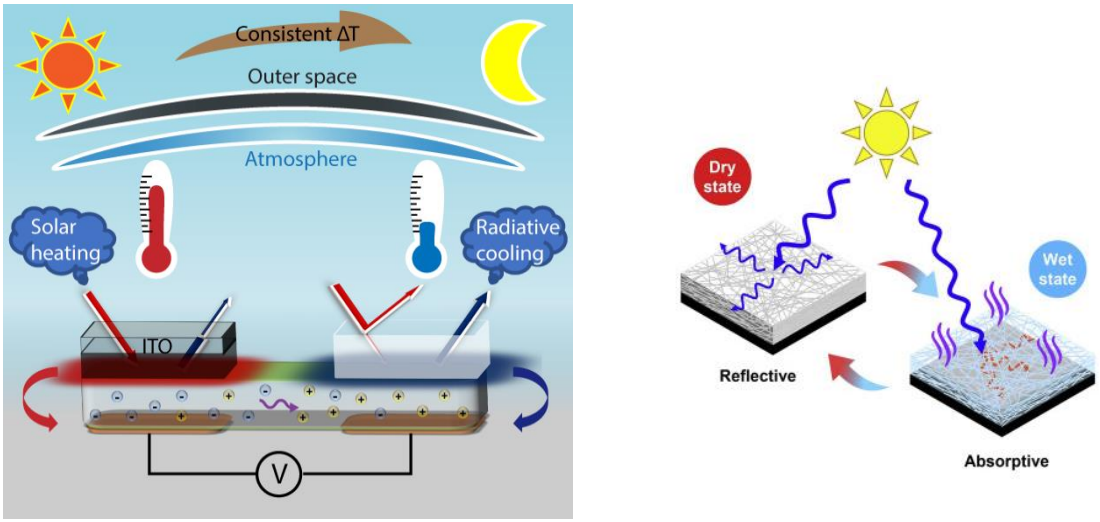

Figure 2. Left panel: schematic picture of a lateral ionic thermoelectric device driven by solar heating and radiative cooling; Right panel: Janus cellulose material where material is white in its dry state, which suppresses solar heating. Upon wetting, the material becomes black and starts to absorb solar light, which promotes water evaporation such that the material dries and becomes white and reflecting again.

WWSC Program 5: Biocomposites and wood materials

Active PIs

KTH: Prof Lars Berglund – Program 5 responsible, Prof Qi Zhou, Assist prof Yuanyuan Li, Prof Mikael Hedenqvist, Assoc prof Richard Olsson
Chalmers: Prof Roland Kádár, Assoc prof Tiina Nypelö
LiU: Assoc prof Klas Tybrandt, Assoc prof Isak Engquist, Assoc prof Eleni Stavrinidou
Luleå Technical University: Prof Kristiina Oksman
Umeå University: Prof Jyri-Pekka Mikkola

Overview of activities within the program

This program is focused on a variety of biocomposites, where wood or cellulose is combined with a polymer or inorganic additives. The mechanical function of cellulosics is extended by the added components. An essential research activity is the strive for nanostructural control, so that structure and property combinations can be tailored. The major cellulosic substrates are wood by itself or nanocellulose in the form of cellulose nanofibrils (CNF) or cellulose nanocrystals (CNC). The program is covering three areas:

5.1 Functional biocomposites and wood materials
5.2 Melt-processing and rheology
5.3 Biocomposites and nanocellulosics

Nanocellulose has dominated the international lignocellulose research field for a long time. An evolving trend in WWSC is the inclusion of nanocellulosic aspects of pulp fibers and wood substrates. By using pulp fibers and wood veneer substrates, it becomes possible to exploit the favorable nanocellulose organization in the wood cell wall as well as the hierarchical structure of wood (tubular cells for liquid transport combined with oriented cellulose in the cell wall). Sustainable development issues are important in Program 5, including tailored structures for energy harvesting, interesting chemistry with specific functionalities and scientific research related to applications.

The Functional biocomposites part (5.1) includes soft robotics (Tybrandt, LiU), electronic plants (Stavrinidou, LiU) and other aspects of organic electronics. Zhou, KTH and Engquist, LiU have close collaboration with coordinated PhD-thesis projects. The materials problem is to combine electrical and ionic conductivity in functionalized wood substrates. Device-related applications include electrodes and transistors. The ion conductivity of wood itself is improved by sulfonation of the pre-existing cell wall lignin. Li, KTH is working on energy-harvesting functions and has prepared a new aerogel structure where nanocellulose is present in the wood cell lumen space. This results in high specific surface area and excellent thermal insulation properties.

In 5.2, Melt processing and rheology, Kadar at Chalmers and Oksman at LTU focus on melt-processed biocomposites. These activities have been extended by additional KAW-funding for biocomposites collaboration between Chalmers, LTU and KTH. Wood fibers are the main reinforcement component and substantial progress has been reported in terms of rheological understanding (Chalmers) and recycling aspects for sustainable development (LTU).

In 5.3, Biocomposites and nanocellulosics are investigated. Three PhDs have been examined during the year, two on transparent wood biocomposites and one on enzymatic modification (LPMO:s, Zhou, KTH) of pulp fibers for the purpose of nanocellulose oxidation. Nypelö and coworkers at Chalmers are also investigating LPMO oxidation, where their substrates are cellulose nanocrystals. Hedenqvist, KTH is interested in barrier properties controlled by gas

solubility in the barrier film and the kinetics of the diffusion process. His coatings are inspired by plant cuticles. Olsson, KTH is working on inorganic hybrids between nanocellulose and metal-oxides, with controlled inorganic morphologies. Nanocomposites based on 2D-platelets is an example where progress in nanostructural control has been driven by high-resolution techniques using XRD from synchrotron sources. Mikkola at UmU is investigating new materials from cellulose and chitin, where ionic liquids are important in the modification and component preparation stages.

List of projects

5.1 Functional biocomposites and wood materials

5.1.1 Soft Cellulose-based robotics K Tybrandt, LiU

5.1.2 Wood template electronics I Engquist, LiU

5.1.3 Distributed electronic functions in wood, from soil to xylem E Stavrinidou, LiU

5.1.4 Functionalized wood templates Q Zhou, KTH

5.1.5 Wood substrate aerogels and electrodes Y Li, KTH

5.1.6 Photonic materials – from wood cellulose to nanocrystals Q Zhou, KTH

5.1.7 Wood mineralization towards sustainable energy conversion Y Li, KTH

5.2 Melt-processing and rheology

5.2.2 Advanced Rheological Characterization R Kadar, Chalmers

5.2.3 Extrusion process of biocomposites K Oksman, LTU

5.3 Biocomposites and nanocellulosics

5.3.1 2D-reinforced nanocellulose composites L Berglund, KTH

5.3.2 Mimicking natural cuticle barrier materials M Hedenqvist, KTH

5.3.3 Inorganic-organic nanocellulose hybrids R Olsson, KTH

5.3.4 Wood biocomposites for mechanical, optical and functional properties L Berglund, KTH

5.3.5 Assembly of hierarchical materials from biopolymers and particles T Nypelö, Chalmers

5.3.6 Lytic Polysaccharide monooxygenases (LPMOs) for adv. nanocellulose materials Q Zhou, KTH

5.3.7 Composites from boreal forests and chitin, JP Mikkola, UmU

WWSC Academy

Persons involved

Paul Gatenholm, professor, Chalmers

Objectives and PhD student training concept

The WWSC Academy is a very important part of the WWSC Program and aims to contribute to fostering a new generation of scientists who will transform the world towards a circular bio-economy based on forest resources.

- The main goal is to provide graduate level, fundamental education within wood materials and science for all WWSC PhD students (32 students graduated in 2018, 30+ students graduated in 2014/15 and 50+students are enrolled since 2019). The WWSC Academy is not replacing the graduate education carried out at the respective Universities; it is a complement.
- All WWSC PhD students have to attend the series of Summer and Winter Graduate Schools designed to provide the fundamentals of Wood Science and Technology but also knowledge of the Forest and the Forest Industry. All schools are combined with site visits.
- All PhD students have to present their research as pitch- and poster presentations twice a year during the WWSC annual workshops. The best presentations receive awards.

2022 activities

During 2022 we organized two Graduate Schools. The Winter School 2022 was held at Selma Spa in Sunne and the topic was: IPR, innovation process, from idea to product and market, knowledge implementation. The school also included site visits to Stora Enso Skoghall and Paper Province, as well as a few examples of journeys of WWSC Academy Alumni after graduation. In total, 42 PhD students participated; 36 from WWSC Academy and 6 from Treesearch. The PhD students came from; KTH (23), Chalmers (10), LiU (5), SU (1), LTU (1), LNU (2).

The topic of the Summer School 2022 was organized in collaboration with Åbo Akademi, and held at Käringsund, on Eckerö, Åland, and the topic was Wood biopolymer science. The school provided insights on fundamental properties and relationships between biopolymers in wood, and included guest lecturers from University of Helsinki, BOKU, University of Guadalajara and Stockholm University. In total, 67 PhD students participated; 36 from WWSC Academy, 10 from Treesearch, and 20 from Finnish Universities. The PhD students came from; KTH (22), Chalmers (14), LiU (5), SU (2), LTU (2), UmU (1), SLU (1) ÅA (17) and U Helsinki (3).

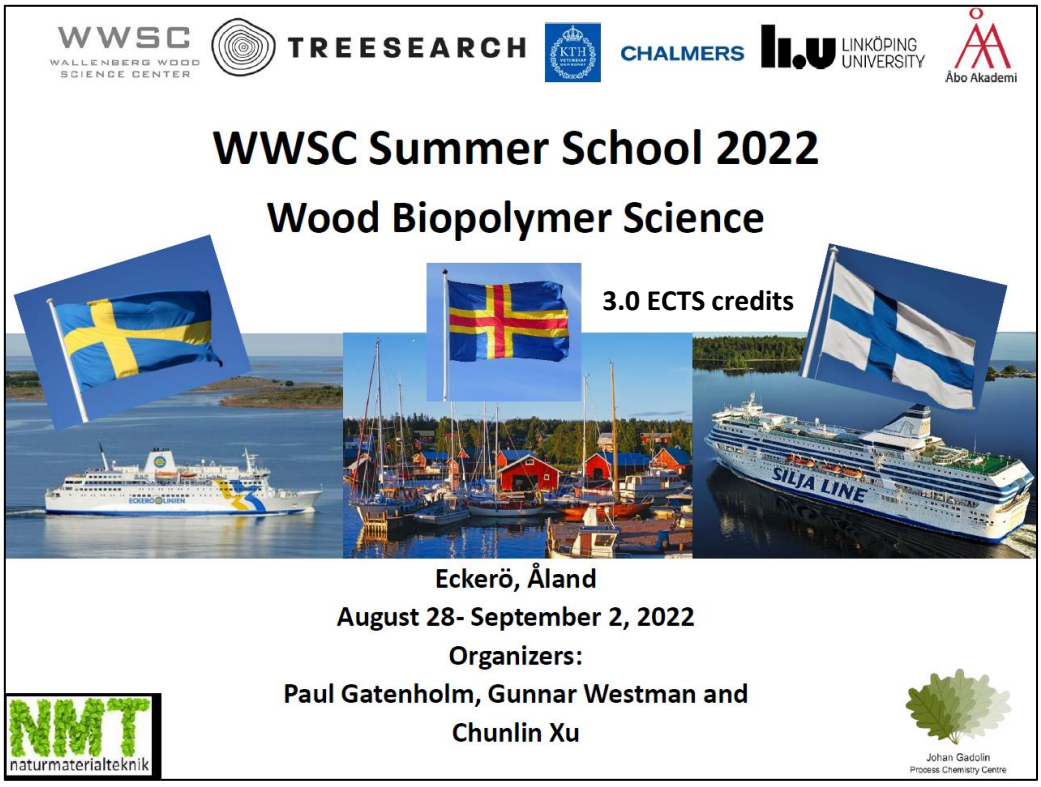

Figure 1. In 2022 WWSC Academy organized two Graduate School. The Winter School gave an introduction to IPR and the innovation process and the Summer School provided insights into wood biopolymer science.

Graduated WWSC PhD students during 2022

Name	University	WWSC Project – thesis title
Céline Montanari	KTH	Transparent Wood Biocomposites for Sustainable Development
Hui Chen	KTH	Light Scattering Effects in Transparent Wood Biocomposites
Salla Koskela	KTH	Lytic polysaccharide monooxygenases for green production of cellulose nanomaterials
Hanieh Mianehrow	KTH	Two-dimensional Nanocomposites Based on Cellulose Nanofibrils and Graphene Oxide
Jonas Garemark	KTH	Integrated Cellulosic Wood Aerogel Structures

WWSC Workshops

WWSC
WALLENBERG WOOD SCIENCE CENTER

WWSC Summer Workshop 2022

Marstrands Havshotell, Marstrand
June 20-22, 2022

Monday June 20

10.15 *Bus from Gothenburg C to Marstrand Havshotell*
12.00 *Lunch*
13:00 **Welcome address:** Eva Malmström, Center Director
13:30 **Research and Innovation – Overview of the Wallenberg Launchpad:** Sandor Albrecht, KAW WALP
14:50 *Break (possibility to check in to hotel room)*
15:20 **Relating cell wall nanostructure to mechanical properties: experiments and modelling:** Daniel Cosgrove, Penn State University, USA *(via Zoom)*
16:00 **WWSC Academy Pitch session**
17:00 **Poster session 1**
18:00 **Poster session 2**
19:00 *Dinner*

Tuesday June 21

09:00 **Spatial distribution of functional groups in cellulose derivatives by DNP-enhanced solid-state NMR spectroscopy:** Staffan Schantz, AstraZeneca
09:40 **Functional wood materials: keeping up with sustainability challenges:** Ingo Burgert, ETH Zürich, Switzerland
10:20 *Break*
10:50 **Biomedical devices based on cellulose nanocomposites:** Mary Donahue, LiU
11:20 **Solution-processed optoelectronic devices: on cellulose-based substrates in the future:** Feng Gao, LiU
12:10 *Lunch*
13.00 *Free time to visit Marstrand*
16:00 **Synchrotron X-ray Technologies to Assist Our Understanding of Nanocellulose Science and Applications:** Benjamin Hsiao, Stony Brook University, USA (via Zoom)
16:40 **Bacteria and fungi: advantages and disadvantages for lignin Valorization:** Davinia Salvachúa, NREL, USA (via Zoom)
17.20 *End of day 2*
18.00 *Dinner at Sillsalteriet (ca 20 min walk, Malepertsgatan 7)*

Wednesday June 22

09:00 **Fundamental interactions in cellulose**: Jakob Wohlert, KTH
09:40 *Break (WWSC LG meeting)*
10:40 **Building up data-driven foundations for customizable trees:** Stefania Giacomello, KTH
11:20 **Forest feedstocks with improved or new properties:** Hannele Tuominen, SLU (via Zoom)
12:00 **Concluding remarks**
12:10 *Lunch*
13:30 *Bus leaving Marstrands Havshotell, arriving at Gothenburg C ca 14.30 – 15.00*

KTH CHALMERS LINKÖPINGS UNIVERSITET

WWSC
WALLENBERG WOOD SCIENCE CENTER

WWSC Winter Workshop 2022

Vildmarkshotellet, Kolmården
December 7-9, 2022

Wednesday December 7

Buses from Stockholm C, Katrineholm C, and Norrköping Resecentrum to Kolmården

12.00 *Lunch*
13:10 **Welcome address:** Eva Malmström, Center Director
13:25 **Nanocellulose Composite Materials for Advanced Wound Care:** Daniel Aili, LiU
14:10 **About xylan and water:** Tiina Nypelö, Chalmers
14:40 *Break (possibility to check in to hotel room)*
15:10 **Scaling Principles for Understanding and Exploiting Adhesion:** Alfred Crosby, University of Massachusetts Amherst, USA *(via Zoom)*
16:00 **WWSC Academy Pitch session**
17:00 **Poster session 1**
18:00 **Poster session 2**
19:00 *Dinner*

Thursday December 8

09:00 **Molecular simulation studies of aggregated microfibrils:** Antti Pajanen, VTT, Finland
09:30 **Taming the invisible with cellulose:** Magnus Jonsson, LiU
10:00 *Break*
10:45 **MAX IV and ForMAX as a versatile tool for WWSC:** Daniel Söderberg, KTH
11:15 **WWSC Bibliometrics – benchmarking research impact:** Christian Müller, Chalmers
12:00 *Lunch*
13.00 **Discovery, engineering, and applications of copper-containing CAZymes (and cousins):** Harry Brumer, University of British Columbia, Canada
13:45 **Cellulose in aqueous alkaline hydroxides:** Merima Hasani, Chalmers
14:15 **3D-printing of cellulose composites for OECTs applications:** Matteo Massetti, LiU
14:35 *Break*
15:05 **Towards a Practical Route to Polymer-Grafted Bleached Market Pulp:** Bob Pelton, McMaster University, Canada
15:50 **Work session – Innovation and Entrepreneurship within WWSC:** Magnus Wikström, MaWi Development
19:15 *Dinner*

Friday December 9

09:00 **Chemical and enzymatic modification of wood for functional materials:** Qi Zhou, KTH
09:45 *Break (WWSC LG meeting and latest check-out from hotel room)*
10:50 **Circular Materials from the Forest – a Chemistry Perspective:** Peter Olsén, KTH
11:20 **How do we design for function and recycling:** Karin Odelius, KTH
11:50 **Concluding remarks**
12:10 *Lunch*
13:30 *Buses leaving Vildmarkshotellet for Stockholm C, Katrineholm C, and Norrköping Resecentrum*

KTH CHALMERS LINKÖPINGS UNIVERSITET

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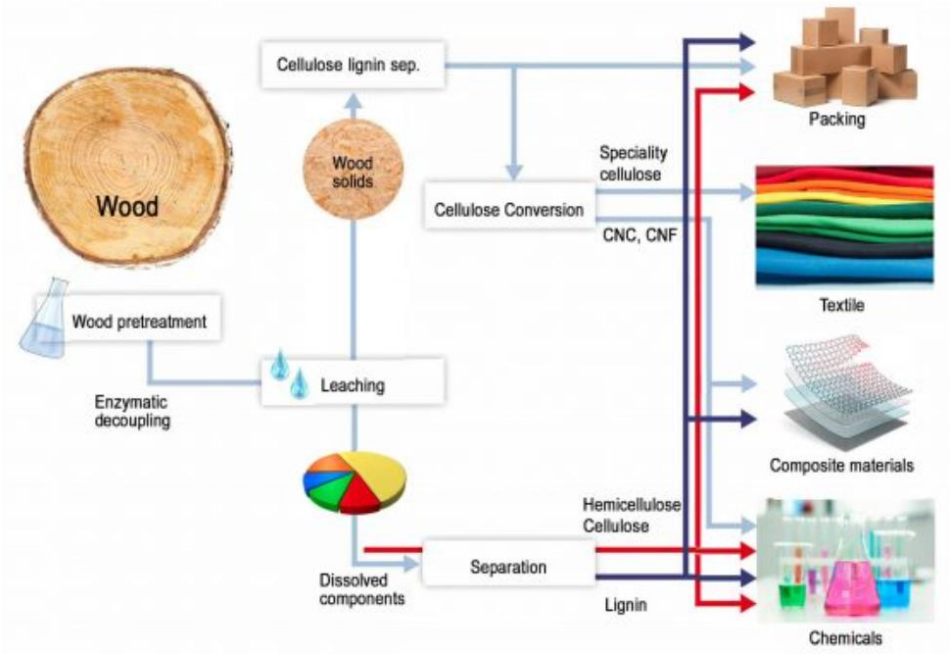
Subprojects Program 1

Wood components – extraction, characterization and properties



1.1.1a Fractionation of wood components using membrane filtration

Persons involved

Kenneth Arandia (PhD student), Nabin Kumar Karna (postdoc), Anette Larsson (co-supervisor), Hans Theliander (main supervisor), Chalmers, Tuve Mattsson, Aalborg University

Activities and results

This year we explored the combination of fluid dynamic gauging (FDG) and molecular dynamics (MD) simulations to study the fouling behaviour of microcrystalline cellulose (MCC) particles on polyethersulfone membranes. The aim was to provide a better mechanistic understanding of the fouling behaviour of MCC through experimental and computational methods.

Cross-flow microfiltration experiments of a dilute MCC suspension showed a significant decline in pure water flux after each membrane cleaning and flushing procedure (see Fig. 1), indicating that highly resilient layers were formed close to the membrane surface. The formation of resilient cellulose layers was also obtained through MD simulations of the free energy profiles, where a deep energy minimum was predicted at close interparticle separations of the cellulose–cellulose (Fig. 2) and cellulose–PES systems. This energy minimum suggests high binding energy at close interparticle distances, implying the need for a specific force to remove the layer or redisperse the cellulose particles. These results highlight how we can gain insights into the fouling behaviour of an organic model material using both FDG and MD simulations.

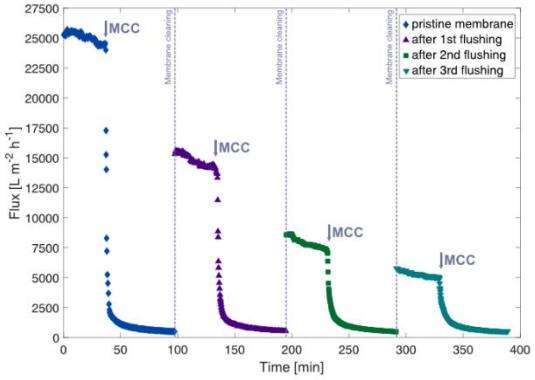
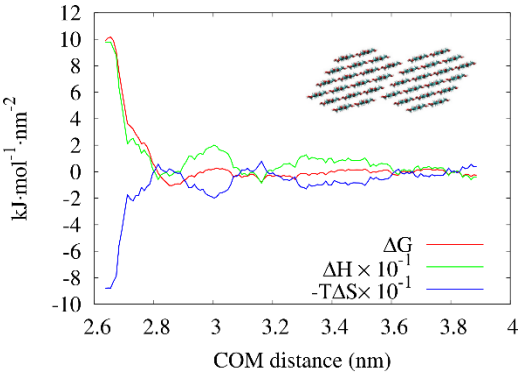


Fig. 1. Flux vs. filtration time for the cross-flow MF of MCC suspensions. Dashed lines indicate membrane cleaning and flushing procedure.



a. 110-110 surface

Fig. 2. Example of MD simulations: cellulose-cellulose interactions, showing the entropic and enthalpic contributions to the free energy (110-110 surface).

Scientific output

[1] Manuscript submitted.

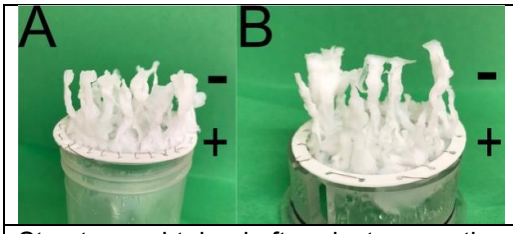
1.1.1b Dewatering of nano celluloses fibrills

Persons involved

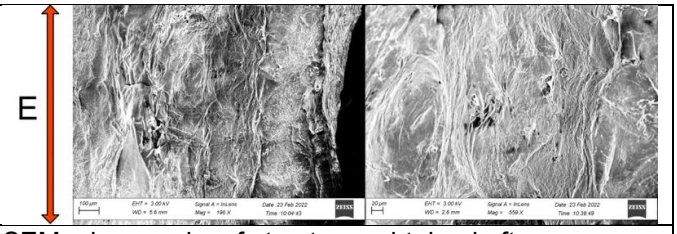
Nabin Karna (postdoc), Anna Lidén (Hjort) (PhD student), Hans Theliander (main supervisor), Chalmers

Activities and results

This year we have been working with a bench-scale dead-end filter press, modified to allow for electroassisted filtration, to study dewatering of a suspension of MFC produced via 2,2,6,6-tetramethylpiperidinyl-1-oxyl (TEMPO)-mediated oxidation (ζ potential of −35.1 mV and a carboxylate content of 1.07 mmol/g TEMPO-MFC). A filter cake was produced with a channeled/pillared structure related to the design of the anode mesh, indicating that the cellulose microfibrils were aligned in the direction of the electric field. This was investigated further, qualitatively and quantitively, using scanning electron microscopy and wide-angle X-ray scattering, which showed a preferred orientation on a microscopic level but only a partial orientation on a molecular level (fc between 0.49 and 0.57). The influence of the density of the anode mesh, in terms of the structure/permeability of the filter cake and dewatering rate, was also evaluated using two different anode mesh densities (5 Å~ 5 and 10 Å~ 10 mm). It was not, however, found to have any major impact on the dewatering rate.



Structures obtained after electroosmotic dewatering at 24 V/cm using a denser (a) and a wider (b) mesh. Locations of the anode (+) and cathode (-) are as marked.



SEM micrographs of structures obtained after electroosmotic dewatering at 24 V/cm (wide mesh). The direction of the electric field, E, is vertical.

Scientific output

Lidén, A., Naidjonoka, P., Karna, N. and Theliander, H., ”Structure of Filter Cakes during the Electroassisted Filtration of Microfibrillated Cellulose”, Ind Eng Chem Res., Published on line, 2022, https://doi.org/10.1021/acs.iecr.2c03216

1.1.2a Mass transfer challenges in wood decomposition

Persons involved

Roujin Ghaffari (PhD-student), Anette Larsson (main supervisor), Lars Evenäs, Alexander Idström, Chalmers, Martin Lawoko (co-supervisor), KTH, Gunnar Lidén, Henrik Almqvist, LTH

Activities and results

The scope of the current project is to study mass transfer events in wood decomposition. First, mass transfer of degraded lignin molecules through fibers in kraft cooking was targeted. Diffusion cells were used to study the effect of size of confinements, alkalinity and molecular weight of lignin on the diffusion of degraded lignin molecules through cellulose fibers. We observed that the diffusion process of degraded lignin molecules accelerates by increasing the concentration of NaOH and pore sizes, and by decreasing the M_W of lignin. The results of these studies were written into two publications (one published (Ghaffari et al. 2022) and one under revision).

The influence of ionic strength and the presence of various salts on the diffusion of lignin through cellulose fibers and adsorption of lignin to cellulose fibers is currently being studied using diffusion cells and QCM-D methods.

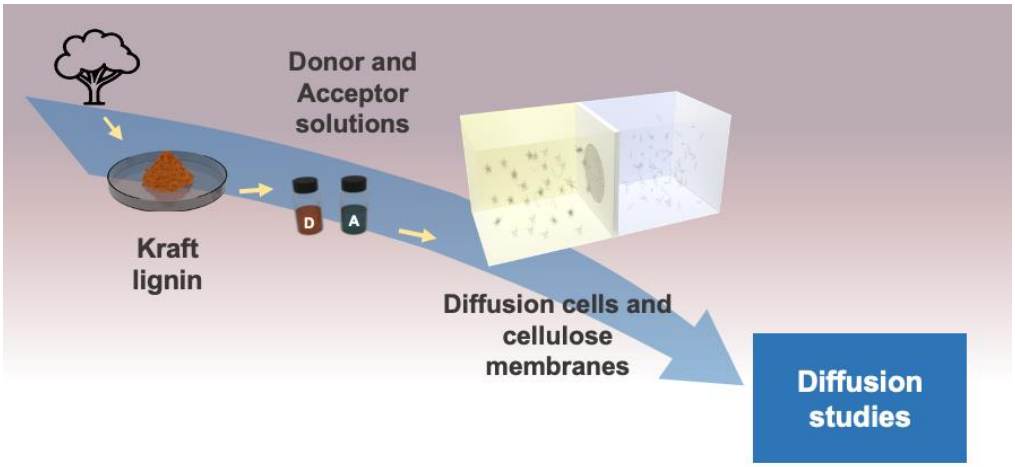


Figure 1 Schematic representation of the experimental procedure

In the continuation of this project, mass transfer events during the ionSolv process are targeted. A novel methodology to study the diffusion of ionic liquids into wood pieces and the diffusion of degraded lignin products out of the wood pieces is currently being developed and will be tested in the coming year.

Scientific output

Ghaffari R, Almqvist H, Nilsson R, et al (2022) Mass Transport of Lignin in Confined Pores. Polymers (Basel) 14:. https://doi.org/doi.org/10.3390/polym14101993

1.1.2b NMR as tool to study heterogeneity in biomass

Persons involved

Koyuru Nakayama (postdoc), Hampus Karlsson (postdoc), Lars Evenäs (supervisor), Chalmers

Activities and results

The purpose of this research project has been to measure and obtain molecular insights that can be correlated with macroscopic material properties, and processing strategies that are expected to advance production of new or improved materials based on biomass. The beginning of the year focused on the implementation of rotor-synchronized solid-state NMR methodology for the new solid-state NMR instrument. Moreover, the project on non-destructive determination of xylan oxidation with high molecular precision was finalized. During the second half of the year, we started to exploit DNP solid-state NMR to obtain detailed information, with superior sensitivity, on the molecular organization of biopolymers, the heterogeneity of modified cellulose or biocomposites, and the molecular alignment in fiber materials.

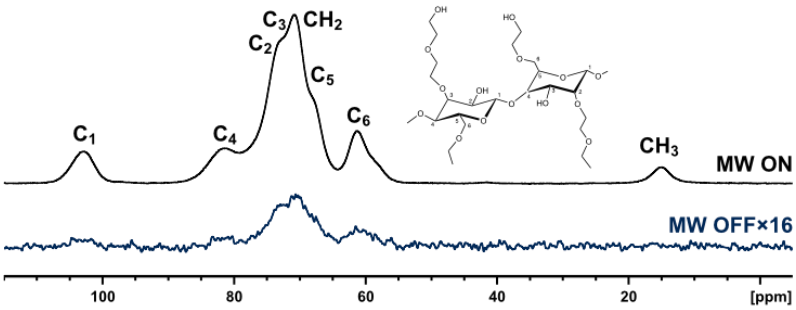


Figure: (top) DNP-enhanced ^{13}C NMR spectrum of ethyl hydrox-yethyl cellulose, (bottom) standard NMR spectrum. Signal enhancement is ~100 times.

Scientific output

A. Avella, A. Idström, R. Mincheva, K. Nakayama, Evenäs, J.M. Raquez, G. Lo Re. Reactive melt crosslinking of cellulose nanocrystals/poly(ε-caprolactone) for heat-shrinkable network. Compos. Part A-Appl. S., 2022, 163, 107166 (doi.org/10.1016/j.compositesa.2022.107166)

C. Palasingh, K. Nakayama, F. Abik, K.S.Mikkonen, L. Evenäs, A. Ström, T. Nypelö. Modification of xylan via an oxidation–reduction reaction. Carbohyd. Polym., 2022, 292, 119660 (doi.org/10.1016/j.carbpol.2022.119660)

H. Ulmefors, T. Yang Nilsson, V. Eriksson, G. Eriksson, L. Evenäs, M. Andersson Trojer. Solution-Spinning of a Collection of Micro- and Nanocarrier-Functionalized Polysaccharide Fibers. Macromol. Mater. Eng., 2022, 2200110 (doi.org/10.1002/mame.202200110)

C. Magnani, M. Fazilati, R. Kádár, A. Idström, L. Evenäs, J.M. Raquez, G. Lo Re. Green Topochemical Esterification Effects on the Supramolecular Structure of Chitin Nanocrystals: Implications for Highly Stable Pickering Emulsions. ACS Appl. Nano Mater., 2022, 5(4), 4731-4743 (doi.org/10.1021/acsanm.1c03708)

M. Haque, I. Abdurrokhman, A.Idström, Q. Li, A. Rajaras, A. Martinelli, L. Evenäs, P. Lundgren, P. Enoksson. Exploiting low-grade waste heat to produce electricity through supercapacitor containing carbon electrodes and ionic liquid electrolytes. Electrochim. Acta, 2022, 403, 139640 (doi.org/10.1016/j.electacta.2021.139640)

1.1.3a Developing dissolution strategies for the recalcitrant structures of the cell wall

Persons involved

Probal Basu (postdoc), Merima Hasani (supervisor), Chalmers

Activities and results

The main aim of the project is to elucidate cellulose motifs of the cell wall insoluble in aqueous alkaline systems and based on that develop dissolution strategies for dissolution these recalcitrant structures. The research has been conducted in two parallel tracks:

- Recovering insoluble cellulose structures from a series of aqueous hydroxide solutions and analysis of their composition, supramolecular and morphological organization.
- Developing spectroscopy and microscopy techniques capable of assessing crystallinity and ultrastructure of these fractions.

Achievements

- Crystallinity, molecular weigth, distribution of oxidized groups & composition of the insoluble cellulose structures have been assessed.
- Commonly, isoluble material comprises whole fibers rather than parts of fibers.
- The thin helical S1 layer plays likely a critical role in preventing dissolution
- Mechanical disintegration of fibers promotes dissolution likely due to disruption of the robust S1 layer

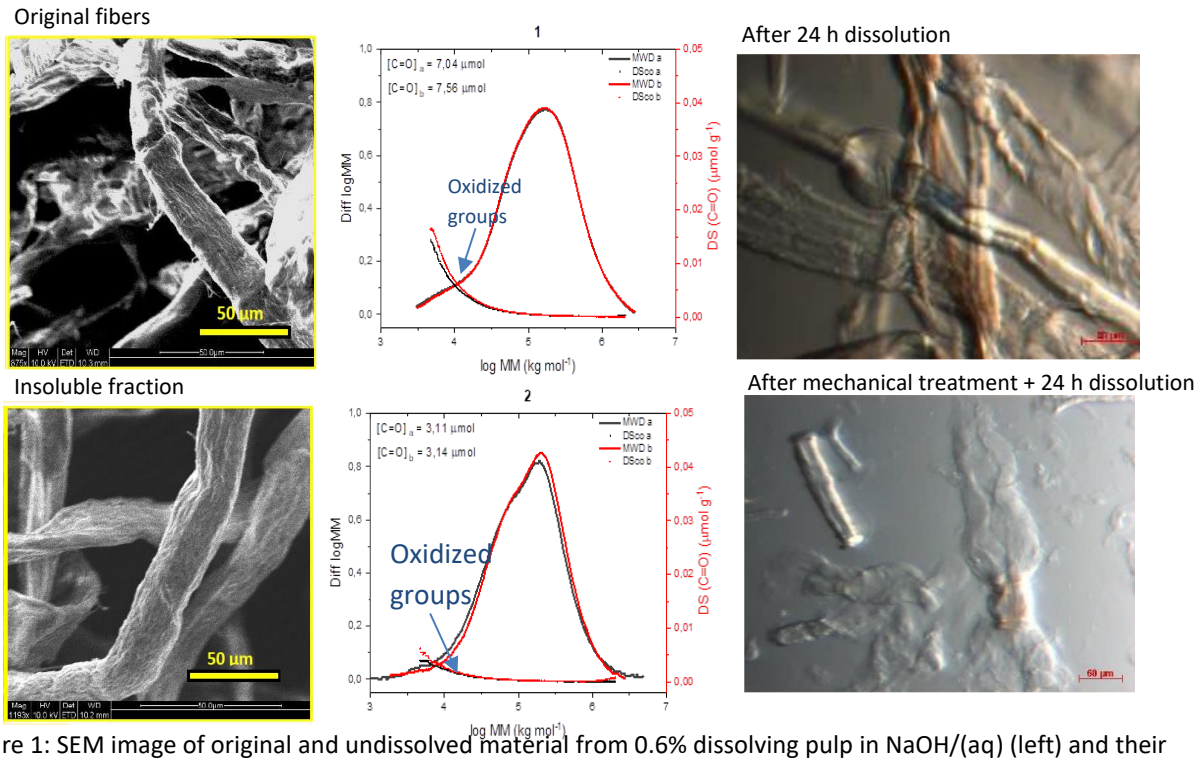


Figure 1: SEM image of original and undissolved material from 0.6% dissolving pulp in NaOH/(aq) (left) and their molecular weight distributions along with oxidized group profiles (middle): micrographs of undissolved fiber with and without mechanical pre-treatment.

Scientific output

Manuscript in preparation.

1.2.1 Technical lignins

Persons involved

Olena Sevastyanova (WWSC PI), Huisi Li (PhD student), Oihana Gordobil, InnoRenew CoE, Slovenia, and others

Activities and results

Surface Chemistry and Bioactivity of Colloidal Particles from Industrial Kraft Lignins

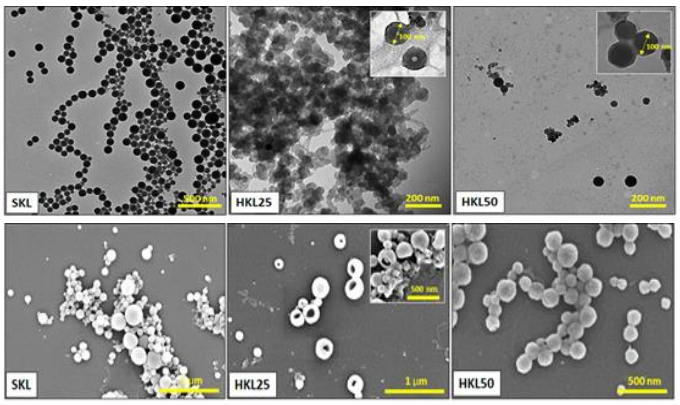


Figure 1: *TEM (top) and SEM (bottom) images of lignin colloidal particles prepared by solvent shifting method from industrially derived lignins: spruce kraft (SKL), eucalyptus kraft (HKL25 – dried at 25°C and HKL50 – dried at 50°C).*

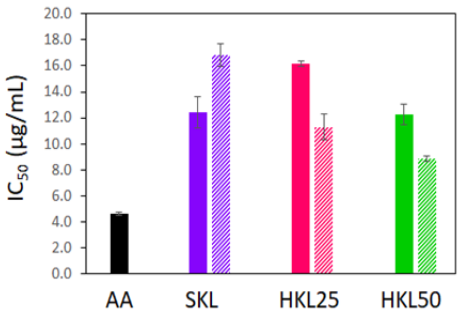


Figure 2*: Efficient concentration value (IC50) from DPPH test. (Solid bar and striped bar correspond to raw lignins and CLPs, respectively).*

Colloidal lignin particles (CLPs) in the range of 150-200 nm were produced from hardwood and softwood kraft lignins by solvent shifting method using a recyclable and non-toxic organic solvent. Softwood kraft lignin (SKL) had higher purity, higher average molecular weight and polydispersity, higher content of aliphatic hydroxyl groups, more condensed substructures and less carboxylic groups than hardwood kraft lignins (HKL25 and HKL50). Microscopic observations of CLPs revealed different appearance and morphology in terms of shape, size, inner structures, and surface roughness of produced particles. Based on the botanical origin of the lignin, clear differences in the surface chemistry of colloidal particles were noticed in the 1H NMR analysis. An opposite behaviour against DPPH radical was observed after particle size reduction of kraft lignins based on their botanical origin. Based on cytotoxicity studies of original lignins and corresponding CLPs, we can conclude that CLPs, in general, presented higher toxicity than irregular kraft lignin powders towards lung fibroblast cells. Furthermore, in both forms (powder and nanoparticles), softwood kraft lignin showed lower cytotoxic effect than hardwood kraft lignins. Additionally, the toxicity of kraft lignins and corresponding CLPs was dose-dependent; therefore, certain concentrations of these materials that are not toxic for cell systems might be used as an active ingredient in the cosmetic and in pharmaceutical sectors.

Scientific output

O. Gordobil, H. Li, A.A. Izquierdo, A. Egizabal, O. Sevastyanova, A. Sandak. Surface chemistry and bioactivity of colloidal particles from industrial kraft lignins, International Journal of Biological Macromolecules, 2022, 220, pp 1444-1453
https://doi.org/10.1016/j.ijbiomac.2022.09.111

1.2.1b Lignin-based UV protective coatings

Persons involved

Nataliia Smyk (researcher), Olena Sevastyanova (supervisor), KTH, M. Goliszek, B. Podkościelna, Maria Curie-Skłodowska University

Activities and results

The functional groups within lignin are able to effectively absorb light in the visible and UV range, making lignin a good candidate as a bio-additive in UV-protective polymer coatings , in particular. The scope of the presented project is to evaluate the influence of lignin of different origin and chemical modification on the properties of the polymer coatings. Four type of the new bio-based materials were obtained by filling of epoxy resin with LignoBoost spruce and eucalyptus lignin before and after methacrylation process. Based on FTIR investigation the possible scheme of polymerization in the presence of lignins was proposed, Figure 1.

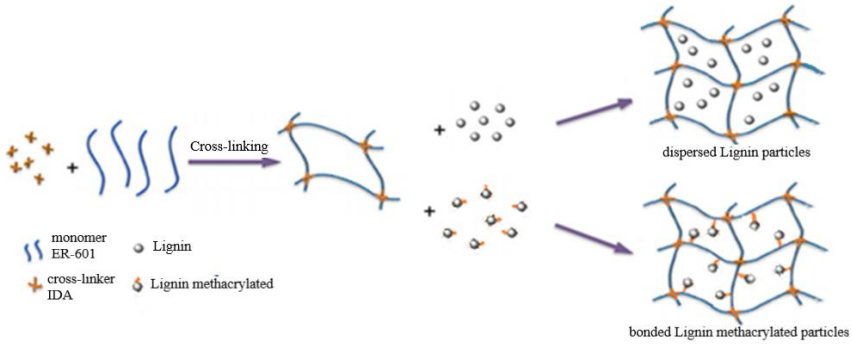


***Figure 1**: Schematic representation of the possible scheme of polymerisation.*

It was shown that methacrylated spruce and eucalyptus lignins can effectively block ultraviolet radiation across the UV spectrum (200-340 nm), Figure 2. The color properties of the coatings were investigated using the CIE L*a*b* color space. I was established that chemical modification of lignin significantly affects the color of the biocomposites. Since color is an important feature for consumers, optimizing the selection of modified lignin based on colorimetric characteristics will contribute to better commercialization of the lignin-containing coatings

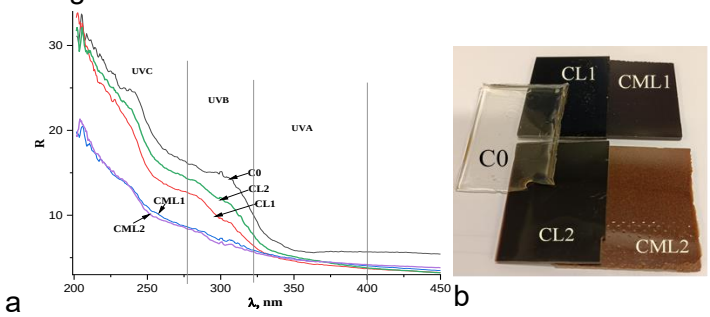


***Figure 2**: The diffusion reflectance spectra (a) and photographs (b) of the bio-composite coatings clear (C0) and filled with spruce (1) and eucalyptus (2) lignins before (L1, L2) and after modification (ML1, ML2).*

The results demonstrate that methacrylated lignin is a promising environmentally friendly bio-additive for UV protection of polymer coatings.

Scientific output

Manuscript submitted.

1.2.2a Towards Zinc-Lignin Batteries with "Water-in-Polymer Salt Electrolyte" (WIPSE)

Persons involved

Divyaratan Kumar (PhD student), Ziyauddin Khan, Ujwala Ail, Viktor Gueskine (co-supervisor), Xavier Crispin (main supervisor), LiU

Activities and results

Zn-ion aqueous batteries are among the hot candidates for low-cost and sustainable secondary batteries. However, water splitting resulting in hydrogen evolution reaction (HER) and dendritic growth upon zinc deposition are today's challenges for that technology. A new strategy to suppress HER, thus broadening the electrochemical stability window (ESW), is to use a super concentrated "water-in-salt" electrolyte (WISE), in which water is bound. WISE are usually based on TFSI salts, so we have shown that in 0.1 m $Zn(TFSI)_2$ / PAAK based WIPSE, Zn can be successfully plated and stripped in Zn//Zn symmetric cells. Lignin-Carbon as cathode also demonstrates good performance in such near-neutral pH WIPSE, though typically low pH electrolyte was required, which is advantageous for large scale energy storage devices for future applications. We therefore believe that this hybrid electrolyte can be suitable for mono- and divalent ion batteries like Li-ion, Zn-ion, Ca-ion with lignin-based cathode materials.

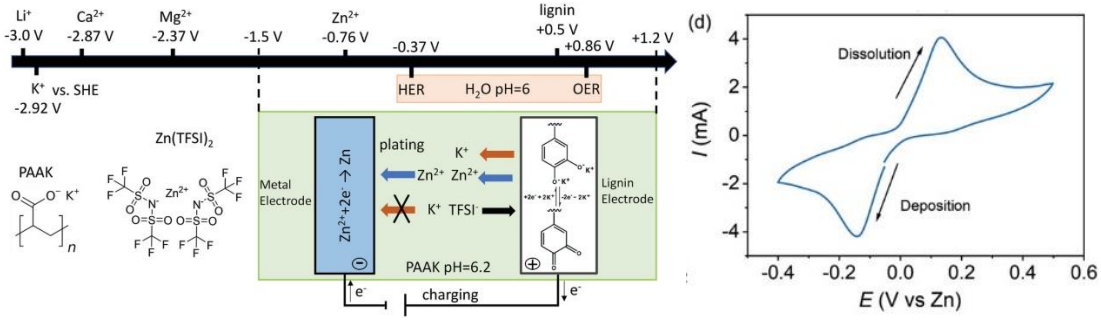


Figure 1, Scale of potential indicating the ESW of PAAK (green) as well as the Zn and lignin redox potential for pH = 6. Chemical structures of the relevant redox systems and polymer salt are illustrated, CV of Zn/PAAK-$Zn(TFSI)_2$/Zn showing stripping and plating of Zn in 0.1 m $Zn(TFSI)_2$ / PAAK at 2 mVs^{-1}

Scientific output

1. Self-Discharge in Batteries Based on Lignin and Water-in-Polymer Salt Electrolyte, D. Kumar, Z. Khan, U. Ail, J. Phopase, M. Berggren, V. Gueskine, X. Crispin, 2022, https://doi.org/10.1002/aesr.202200073
2. Zinc salt in "Water-in-Polymer Salt Electrolyte" for Zinc-Lignin Batteries: Electroactivity of the Lignin Cathode, D. Kumar, U. Ail, Z. Wu, E. M. Björk, M. Berggren, V. Gueskine, X. Crispin, Z. Khan, 2022, https://doi.org/10.1002/adsu.202200433

1.3.1b LPMO discovery and applications

Persons involved

Monika Tõlgo (PhD student), Lisbeth Olsson (main supervisor), Johan Larsbrink, Saül Llacer Navarro, Tiina Nypelö, Chalmers, Vincent Eijsink (co-supervisor), Olav Hegnar (postdoc and co-supervisor), NBMU, Francisco Vilaplana, Fabio Caputo, KTH

Activities and results

In project 1.3b we are studying novel biomass degrading enzymes called lytic polysaccharide monooxygenases (LPMOs). In Nature, biomass break-down as part of carbon cycle happens in big part via microbial enzymes. LPMOs are one class of these biomass degrading enzymes. They are oxidative enzymes which function was first elucidated in 2010 and since then they have improved biomass saccharification for production of biofuels and other biochemicals in the industry significantly. Last years, LPMOs have also found applications in materials science, for example in production of CNFs. As these enzymes are relatively new in the scientific community, a lot remains unknown about LPMOs still, both fundamentally and for their potential in applications.

During 2022, we have focused on three new sub-projects. In the first project we studied how the substitution groups on xylan backbones affect the activity of LPMOs. We showed that removing these substitution groups largely increased LPMO activity on xylans, revealing previously unknown high xylanolytic capabilities of some LPMOs. This is crucial both from applied and fundamental research perspectives. Secondly, we showed that LPMOs can aid in saccharifying recalcitrant mildly pre-treated spruce substrates, and we additionally showed the mechanism behind. Our findings bring mildly pre-treated spruce feedstocks one step closer to being utilized in wood biorefineries. Lastly, in the work largely guided by Tiina Nypelö, we showed that LPMOs can also be used to functionalize sulphated cellulose nanocrystals and demonstrated proof-of-concept crosslinking chemistry of sulphated CNCs using the carboxyl groups added my LPMOs (submitted manuscript). Our work on CNCs and LPMOs will hopefully open the use of LPMOs for CNC functionalization to produce future bio-based materials.

Scientific output

1. Tõlgo, M., Hegnar, O. A., Østby, H., Várnai, A., Vilaplana, F., Eijsink, V. G. H., & Olsson, L. (2022). Comparison of six lytic polysaccharide monooxygenases from *Thermothielavioides terrestris* shows that functional variation underlies the multiplicity of LPMO genes in filamentous fungi. Applied and Environmental Microbiology, 88(6), e00096-22. https://doi.org/10.1128/aem.00096-22
2. Enzymatic debranching is a key determinant of the xylan-degrading activity of family AA9 lytic polysaccharide monooxygenases. Oral presentation by Monika Tõlgo at the 3rd LPMO Symposium, Oslo, Norway

1.3.1a Enzyme discovery and structure-function investigation

Persons involved

Andrea Seveso (PhD student), Tom Coleman (postdoc), Scott Mazurkewich (postdoc), Johan Larsbrink (main supervisor), Chalmers, Lauren McKee (co-supervisor), KTH, Leila Lo Leggio, University of Copenhagen

Activities and results

The work focuses on discovery of new enzymes to decouple carbohydrates and lignin in the plant cell wall, and investigate how they could be used also in more applied settings to for example help purify lignin. Glucuronoyl esterases (GEs) from various bacteria have been characterized, and we recently demonstrated how the enzymes work mechanistically using detailed computational investigations (1). We are continuing studies of novel GEs that are found in large gene clusters (PULs; known to encode proteins that all target the same polysaccharide), that are presumed to target pectin. Our hypothesis is that the GE enzymes in these clusters are either pectin methylesterases (new activity for such enzymes) or that they target pectin-lignin connections that are yet to be demonstrated. Six new enzymes have been characterized so far and a manuscript is in preparation to be submitted in the spring. Additionally, we are also investigating whether the type of ester linkage between lignin and carbohydrates is a major activity determinant for GEs. Alpha- and gamma esters are known, and we have obtained new substrates to analyze this. The results show that there are large differences among GEs, where fungal enzymes and similar bacterial enzymes clearly prefer the alpha-linkage, while more dissimilar bacterial enzymes can prefer the gamma-linkage or show no preference at all. These new results reveal how microorganisms can cleave different lignin-carbohydrate bonds, and give new tools to separate major wood polymers.

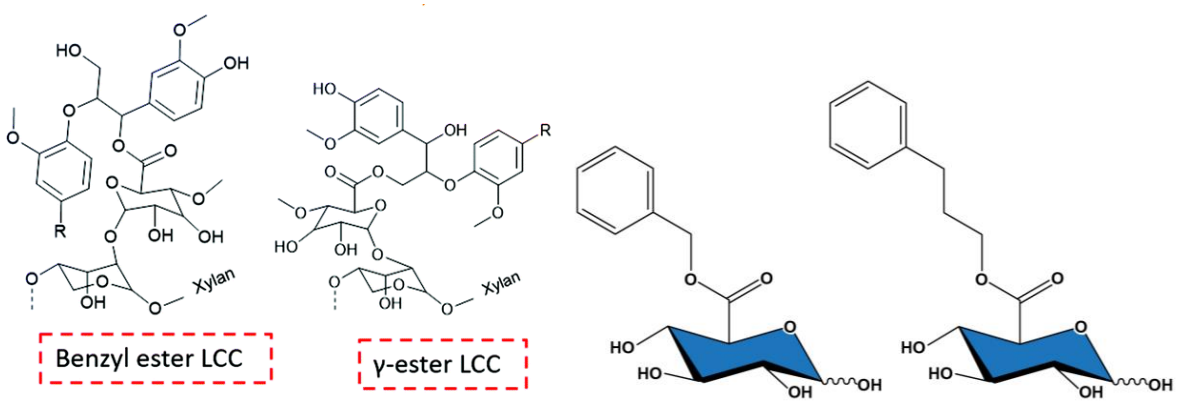


Figure 1: *proposed lignin-carbohydrate esters (left) and newly acquired substrates (right)*

Scientific output

1. Zong Z, Mazurkewich S, Pereira C, Fu H, Cai W, Shao X, Skaf MS, Larsbrink J, Lo Leggio L (2022). Mechanism and biomass association of Glucuronoyl Esterase: an α/β hydrolase with potential in biomass conversion. Nature Communications 13, 1449.
2. Manuscript submitted.

1.3.1c Enzyme discovery – enzymes in materials applications

Persons involved

Mengshu Hao (postdoc), He Li (postdoc), Lauren McKee (main supervisor), KTH
Scott Mazurkewich (postdoc), Johan Larsbrink (supervisor), Chalmers

Activities and results

The production of hydrogels using polysaccharides without chemical modification is being explored, using carbohydrate binding modules (CBMs) to cross-link polysaccharides. We are conducting a broad CBM discovery programme. We have so far characterised 12 proteins that induce gel formation, and showed that some gel properties are different with different CBMs. A manuscript describing these proteins is under review at *Nature Communications*, and this will be followed by a first publication demonstrating the new hydrogel formation mechanism. The project is supported by Carl Tryggers Foundation and Formas, as well as the WWSC. We have shown that we can use CBMs attached to active enzymes to make gels, and are exploring the applications that can be achieved with such a system. We have filed for a patent to protect the novel mechanism of hydrogel formation (now in PCT phase), and founded a company (*Glycolink AB*) to explore commercialisation of the material. *Glycolink* has been awarded innovation funding by Almi and Vinnova and recently secured an investment from KTH Holding.

Figure 1. A hydrogel forms spontaneously when a CBM protein is mixed with scleroglucan in water at room temperature. No chemical modification of the polysaccharide is required to initiate this interaction.

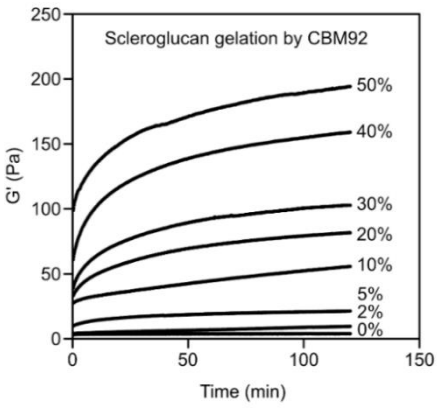


Figure 2. Rheological analysis shows that cross-linking of the polysaccharide increases as protein loading increases, verifying the protein-dependent nature of hydrogel formation.

Scientific output

Glycan processing in gut microbiomes. La Rosa SL, Ostrowski M, VP de Leon A, McKee LS, Larsbrink J, Eijsink VGH, Lowe EC, Martens EC, Pope PB. Current Opinion in Microbiology 67 (2022) 102143

One manuscript accepted for publication.

1.3.2 Enzymes for Release of Specialty Chemicals from Wood – Enzymatic Extraction of Suberin from Bark

Persons involved

Wissal Ben Ali (post doc), Vera Novy (post doc), Lisbeth Olsson (main supervisor), Chalmers

Activities and results

In the present study, we investigated the ability of cutinase to release fatty acids from suberin in tree bark and we aimed at determining the composition of suberin. For this purpose, cutinase-encoding genes from *Fusarium solani* and *Cryptococcus sp* has been successfully cloned and ex-pressed in *Pichia pastoris* cutinases were then produced in bioreactor (1,4 L) with a resulting specific activity against pNP-C4 10 mM of 0,4 U/mg for both recombinant enzymes. The fatty acids released by these recombinant enzymes from real substrate (suberin containing birch bark, spruce bark and cork) was first evaluated by a free fatty acid quantification kit.

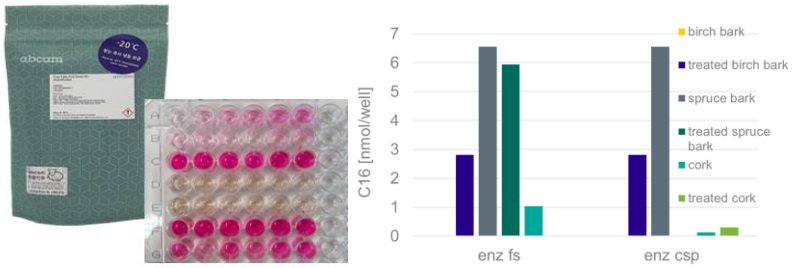


Figure 1: The fatty acids released by these recombinant enzymes from real substrate (suberin containing birch bark, spruce bark, and cork) was evaluated by a free fatty acid quantification kit

Yet, the deeper evaluation of their activity is limited by the lack of efficient high throughput methods to extract, characterize, and semi quantify the non-conventional suberin-derived fatty acids after enzymatic hydrolysis. Therefore, our on-going efforts are directed to furhter develop an efficient extraction method to maximize the release of fatty acids from the bark material and to develop a GC-MS based FAME (fatty acid methyl ester) analysis method for quantification and functional profiling of the suberin-derived fatty acids. Such methodology is the foundation to functionally characterise the different cutinase cloned and expressed in earlier phases of the project.

Scientific output

Poster presentation at the CBM conference 2022: Production of recombinant cutinase from *Fusarium solani* and *Cryptococcus sp*, and evaluation of their role in fatty acids extraction from suberin suberin.

Caputo F, Al-Rudainy B, Naidjonoka P, Wallberg O, Olsson L, Novy V. Understanding the impact of steam pretreatment severity on cellulose ultrastructure, recalcitrance, and hydrolyzability of Norway spruce. Biomass Conversion and Biorefinery. 2022; https://doi.org/10.1007/s13399-022-03405-0.

1.3.2b High resolution and in situ studies of enzymatic action and transport

Persons involved

Maria Eugenia Fortes Brollo (postdoc), Melike Mercan Yildizhan (postdoc), Eva Olsson (main supervisor), Chalmers

Activities and results

The aim of the project was to study the interaction between wood components and enzymes. Spruce wood chips were prepared by steam explosion (STEX) method of pre-treatment, under distinct temperature conditions. Different electron microscopy techniques can be used to investigate these wood specimens. Scanning electron microscopy (SEM) is used for surface evaluation on the porosity level. Focused ion beam (FIB) combined with SEM reveal the internal microstructure of wood by cross-sections cuts**,** see Fig. 1 (left). While transmission electron microscopy (TEM) addresses the nanostructure of wood composites besides the evaluation of enzymes.

Methods are developed to enable studies of enzymes on wood structures in liquids. To study the stability of liquid in the microscope, a special chip set was utilized. In this configuration there are microwells etched on to the SiN_x windows (Fig. 1 (right)). This way, the migration of the damaged species is confined within the well they were created, and the liquid in the other wells remain pristine. The liquid was significantly stable under the electron beam in STEM mode, with no apparent beam damage in the order of minutes of illumination. The low dose required to avoid beam damage necessitate using denoising algorithms to image enzymes in the liquid, which is under progress.

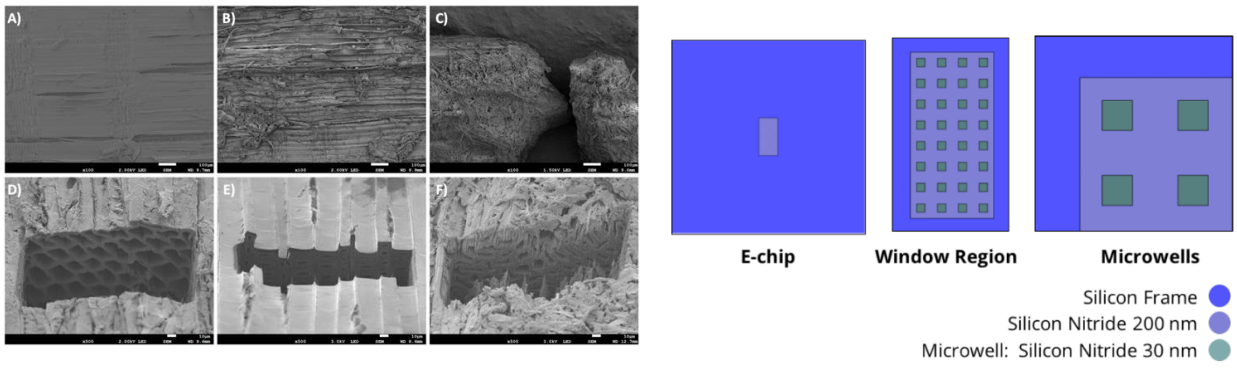


Figure 1(left): SEM images using the secondary electron signal at low magnifications of A) raw sample (spruce), B) STEX at 210°C/5min and C) STEX at 210°C/5min + 3% SO_2. SEM images of FIB cross-sections of D) raw sample, E) STEX at 210°C/5min and F) STEX at 210°C/5min + 3% SO_2. Figure 1(right): Illustration of the chips used in the liquid experiments

Scientific output

Manuscript in preparation.

1.3.3 Enzyme-catalyzed oxidation of wood polysaccharides and creation of novel composites

Persons involved

Scott Mazurkewich (postdoc),Andrea Seveso (PhD student), Johan Larsbrink (main supervisor), Chalmers

Activities and results

Galactose oxidases (GalOx) are enzymes able to convert the O6 hydroxyl groups of galactose moieties to aldehydes, and they require only oxygen and a copper atom in the active site for the reaction to occur. We have investigated bacterial enzymes from the Auxiliary Activities family 5 (AA5) where GalOxs are found, and characterized a novel enzyme which differs significantly in protein sequence from previously studied enzymes, in order to possibly discover enzymes with better activity on polysaccharides. This also represents the first bacterial enzyme characterized. We conducted initial activity screens on galactose and other sugars and demonstrated activity. Surprisingly, the enzyme was however not highly active on galactose or other carbohydrates, and inactive on polysaccharides. Instead, it is mainly active on smaller primary alcohols, and we can attribute the activity on carbohydrates on their open-chain form rather than the closed-ring form which is reported for regular GalOx enzymes. The highest activity was seen on 1,3-propanediol and furfuryl alcohol, though the biological role of this enzyme remains unclear. The enzyme is active over a broad pH range and we have demonstrated activity on close to 40 different substrates, and screened others for which no activity could be demonstrated. We have not been able to crystallize and solve the structure of the enzyme, but have generated models using AlphaFold2 to gain deeper insight into its function. The catalytic domain is coupled to another domain which was presumed to be a carbohydrate esterase, but it was not active and closer structural inspection shows that the expected catalytic amino acid residues are missing and possibly this domain has evolved to be a binding protein, though this has not yet been demonstrated.

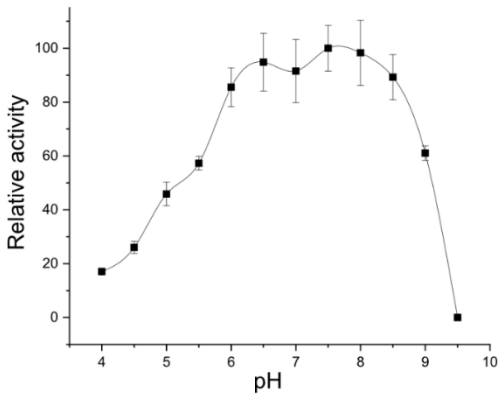


Figure 1: *pH dependency curve, using 1,3-propanediol as substrate*

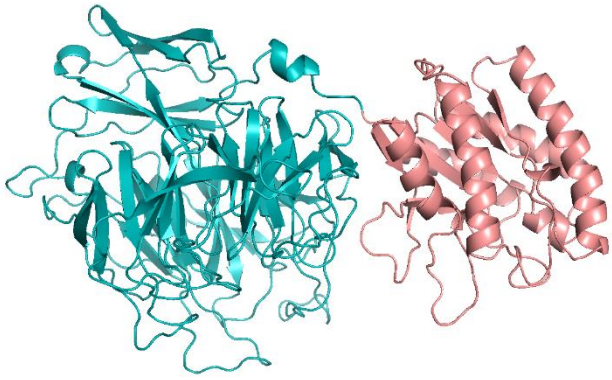


Figure 2: *structure model of the target enzyme, AA5 domain on the left, and the inactive esterase-like domain on the right*

Scientific output

Manuscript in preparation.

1.4.1a Lignin Biorefining: Extraction and Characterization of Reactive Homogeneous Lignins

Persons involved

Maria Karlsson (PhD student), Åsa Emmer, Olena Sevastyanova (Co-supervisor), Martin Lawoko (main supervisor), KTH

Activities and results

Technical lignins suffer from heterogeneity and the chemical and physical properties are poorly understood. In this project, we have developed a novel biorefinery process for the extraction of high quality lignin defined by well characterized structures, and demonstrated, by chemometrics that the chemical properties can be tailored. The process is dubbed the flexible Physical Protection (FPP) process, and has resulted in a WWSC spinoff company, Proligreen AB. The concept is shown in the Figure. To gain deeper insights into the chemical properties of lignin, a follow up study complements state of the art NMR analysis with MALDI-ToF analysis, in effect adding new knowledge on lignin structural populations and linkage sequences, which has contributed to deeper fundamental understanding on native lignin structure and its reactivity during biorefining. A manuscript on this topic is submitted.

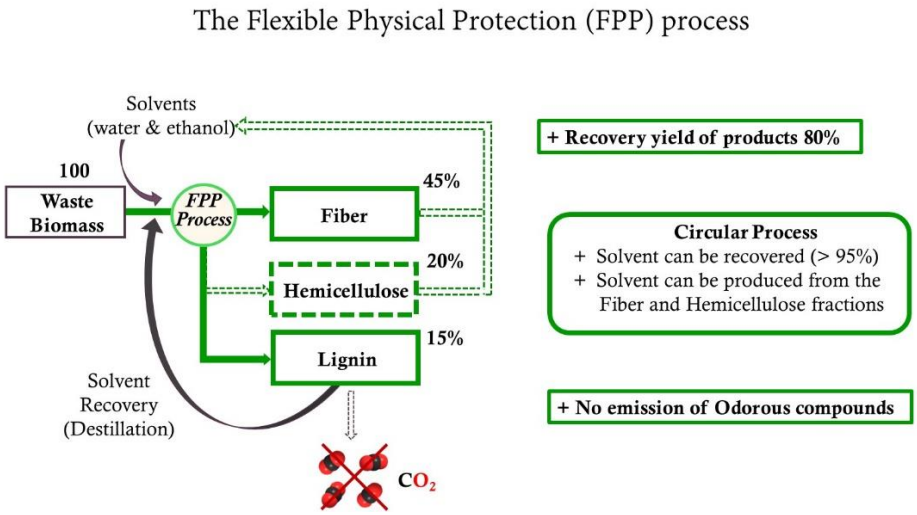


Figure: Schematic for the developed Novel Lignin Biorefinery

Scientific output

Protected lignin biorefining through cyclic extraction: gaining fundamental insights into the tuneable properties of lignin by chemometrics. M Karlsson, VL Vegunta, R Deshpande, M Lawoko. Green Chemistry 2022, 24 (3), 1211-1223

Fundamental Insights on the Physical and Chemical Properties of Organosolv Lignin from Norway Spruce Bark. B Rietzler, M Karlsson, I Kwan, M Lawoko, M Ek. Biomacromolecules 2022, 23 (8), 3349-3358

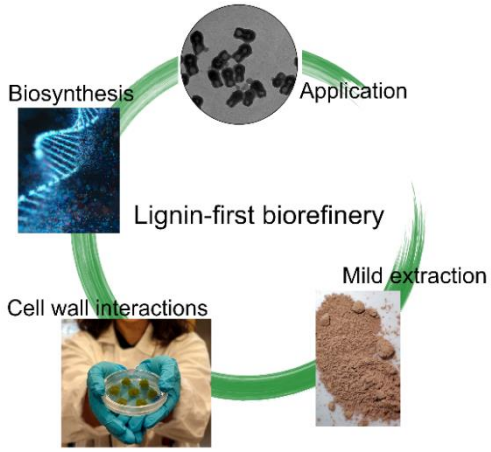
1.4.1b Native lignin structure, polymerization, and interactions with cell wall components

Persons involved

Ioanna Sapouna (PhD-student), Jonas Garemark (PhD student), Alexandros Alexakis (PhD student), Eva Malmström, Martin Lawoko, Lauren McKee (main supervisor), KTH, Roujin Ghaffari, Chalmers, Gijs van Erven, Wageningen University & Research, Anna Kärkönen, University of Helsinki

Activities and results

Lignin extracellular polymerization and its biosynthesis was studied in a spruce tissue culture (1). Chemical analysis of the product with NMR, SEC and sugar composition (HPAEC-PAD) showed that the presence of a secondary cell wall hemicellulose in the nutrient medium on which the cells grow and the medium into which they secrete monolignols affects the growth of the cells, and lignin polymerization. RNA extracted from the cells in different xylan treatments was sequenced (National Genomic Infrastructure at SciLifeLab) and compared (National Bioinformatics Infrastructure Sweden at SciLifeLab) to the reference, no-xylan samples. This comparison will elucidate the role of the hemicellulose in the monolignol biosynthetic pathway.



In another project, lignin was extracted from ball milled spruce (softwood) and birch (hardwood) with optimized, sequential protocols (2, 3). The results underlined the differences between the structure of the biopolymer in the two species, which affects its extractability and added to the understanding of the extraction process on the structure of lignin.

The lignin extracted from the above project was used for the preparation of lignin nanoparticles (LNPs) (4). The effect of the structure of lignin on the size, charge and shape of the LNPs was systematically studied with PET, DLS, FE-SEM and TEM.

Collaborations in different projects expanded further the understanding of the effect of lignin structure in different applications (5-7). For example, lignin from wood aerogels was characterized to explain the behaviour of the materials (6-7).

Scientific output

J. Garemark et al., *Advanced Functional Materials*, 2022, 2208933I.

Two manuscripts under review and two manuscripts in preparation.

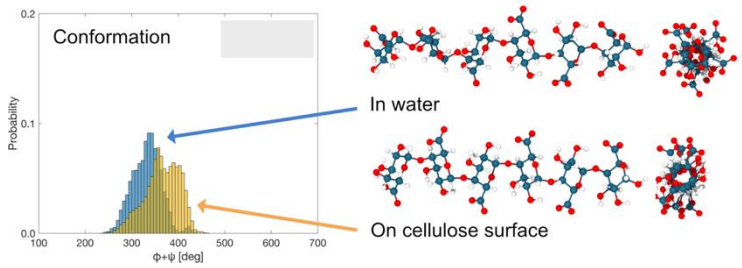
1.4.2 Wood heteropolysaccharides – molecular structure and function in the plant cell wall

Persons involved

Emilia Heinonen (PhD student), Francisco Vilaplana (main supervisor), Jakob Wohlert, Mikael E Lindström, Gunnar Henriksson (co-supervisors), KTH

Activities and results

Hemicelluloses are fundamental components in lignocellulosic biomass, contributing to wood integrity but also to their recalcitrance to fractionation. This research program will decipher the molecular structure of wood hemicelluloses and their interactions with cellulose and lignin in the cell wall, integrating analytical and computational approaches. We have previously demonstrated that xylan chains spontaneously adsorb onto hydrated cellulose surfaces in a 2-fold conformation, forming a rigid layer that functions as a transition phase towards more flexible and disordered polysaccharide/lignin domains. We have now extended this approach using molecular dynamics simulations to a wide range of cell wall polysaccharides, including mannan, glucomannan, xyloglucan, mixed-linkage β-glucan, β-galactan, homogalacturonan and rhamnogalacturonan, to understand the mechanisms that support their interactions with cellulose surfaces at the molecular level. We demonstrate that all matrix polysaccharides interact with cellulose surfaces, but only those containing a β-1,4 backbone in equatorial configuration align with cellulose chains. Particularly interesting are the conformational changes undergone by pectin homo galacturonan (see picture), where all carboxylic acid groups align away from the cellulose surfaces.



Experimentally, we have purified a wide range of wood hemicelluloses with specific molecular motifs from hardwoods (birch, aspen, eucalyptus) and softwoods (spruce). Eucalyptus xylan is particularly interesting, as it bears galactose on top of the glucuronic acid substitutions, which may have an important role modulating interactions with lignin. Our mass spectrometric approaches have revealed that the galactosyl substitutions are clustered in minor domains along the xylan backbone. We have also established a charge-based separation protocol followed by ^{1}H-qNMR and ^{1}H-PFG-NMR measurements to study the correlation of xylan structure and solubility. In collaboration with Prof. Ewa Mellerovicz at Umeå Plant Science Centre we have demonstrated that in planta modifications of the xylan and lignin pathways improve the hemicellulose extractability and saccharification of aspen wood.

Scientific output

E Heinonen, G Henriksson, ME Lindström, F Vilaplana, J Wohlert. Xylan adsorption on cellulose: preferred alignment and local surface immobilizing effect. 2022 Carb Polym 285, 119221.

One manuscript submitted.

1.5.2 Creation of a Terahertz characterization platform - Insight in the dynamics and soft structures of lignocellulosic materials

Persons involved

Gunnar Westman, Serguei Cherednichenko (main supervisors), Amit Kumar Sonker, Dan Kuylenstierna, Aleksandar Matic, (co-supervisors), Thi Ngoc Do Thanh, (postdoc), Jelka Feldhausen (master student), Chalmers, Hannah Tideland, (master student), GU

Activities and results

The use of THz-spectroscopy for characterization of cellulose is a new technique so not much data is available in the literature nor are instruments commercially available for characterization. With THz spectroscopy it should be, at least theoretically, possible to detect weak hydrogen bonds within the cellulose structure. A spectrophotometer that works in the terahertz region, 0.1-15 THz, of the electromagnetic spectrum has been built, THe-SpeCell. Hydroxyl bonds has been determined with THe-SpeCell and successful distinguish between cellulose I and cellulose II samples has been done. The system also shows reproducible information on the weak hydrogen bonds on the surface of nanocellulose. Thickness of the samples has turned out to be one of the major parameters for high reproducibility.

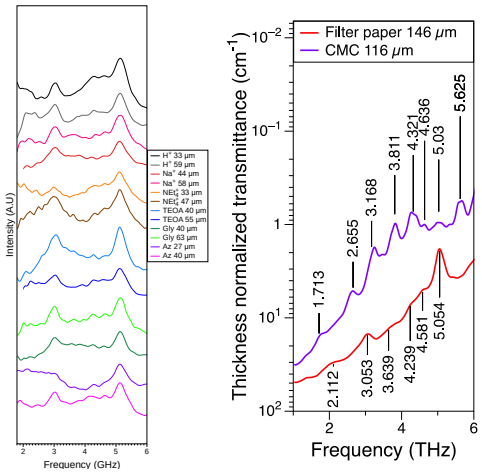


Figure: Left: Normalized intensity spectra of weak hydrogen bonding region of CNCs and additives to improve flexibility in films of two different thicknesses. Right: reference materials CMC and Whatman no. 5 filter paper. Peak at 5.054 (red curve) of Filter paper defined to Iβ-cellulose.

In figure to the left: it can be seen that signal intensities and frequencies depend on the additive used. Especially around 2.5 THz, that is attributed to translational, rotational and torsional vibration modes of the hydroxyl groups.

Scientific output

The project is a short term internal strategic project within WWSC.

TERAHERTZ SPECTROSCOPY OF CELLULOSE NANOCRYSTALS- Method development for investigating intermolecular forces in polycrystalline macromolecules. Hannah Tideland (Master Thesis)

One manuscript submitted.