



# **FibRe**

## **Design for Circularity: Lignocellulose-based Thermoplastics**

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**Abstract booklet  
Poster presentations**



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**Cellulose derived Diels-Alder covalent adaptable networks: a route towards recyclable thermosets**

Polymer networks, also known as thermosets, are known for their excellent properties such as high strength, thermal and chemical stability. This makes thermosets ideal for demanding applications where lightness and great mechanical performance is required (i.e. structural adhesives, automotive, coatings, sports equipment, protective gear, etc.). However, thermosets are associated with poor processing and recycling capabilities. As a result, their end-of-life options are limited to landfill, incineration or re-use as low value filler, preventing thermosetting materials from meeting the demand for sustainable and circular materials. In recent years, research has focused on imparting thermosets with inherent mechanical or chemical recyclability. For example, the introduction of dynamic covalent bonds in polymeric networks has formed a promising solution to the challenge of developing circular thermosets. So-called covalent adaptable networks (CANs) bear dynamic reversible bonds, such as Diels-Alder, Aza-Michael, imine and disulfides, that can form or break upon an external stimulus (i.e. heat or mechanical stress). Their incorporation into a network give rise to materials that have improved mechanical properties and thermal/chemical stability associated with thermosets, but also contain re-processing and recycling capabilities.

We focused on the use of cellulose as starting material to design and synthesize CANs. Cellulose, generally derived from plants and wood, is the most abundantly available biopolymer with attractive mechanical properties, good thermal stability, biodegradability, non-toxicity and biocompatibility, making it a good candidate for the synthesis of high-performance materials such as CANs. Moreover, the characteristic large number of hydroxyl group in the anhydroglucose units in the cellulose backbone are available for a wide range of modification. We modified microcrystalline cellulose (MCC) with furan-moieties targeting varying degrees of substitution in the range of 0.5-2.5. The furan functionalized cellulose was further crosslinked with multifunctional bismaleimide monomers to obtain the cellulose derived Diels-Alder CANs.

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**Mycelium based composites and mycozymes production from agro-waste biomass valorization**

Lignocellulosic biomass is a leading resource for sustainable materials and renewable energy, addressing environmental issues from fossil fuels overuse. Fungal mycelium, an exciting alternative, grows on various organic substrates like agricultural residues. It is fully biodegradable, low in density, cost-effective, and requires minimal processing energy [1]. Wood decay fungi, mostly Basidiomycota, efficiently utilize lignocellulose by producing degrading enzymes to convert complex matrices of lignin, cellulose and hemicellulose into available sugars for their growth [2], [3]. At the same time mycelium-based composites are produced as emerging materials that utilize abundant agri-food lignocellulosic waste, such as husks or stems, by fungal growth. Fungi strengthen and bind lignocellulosic fibers through microscopic filaments forming mycelium, acting as a natural glue [4]. This produces a low-density material, which can be further processed for desired properties like higher density or specific shape. However, mycelium-based composites vary widely based on the fungal species, substrate composition and structure, and post-production treatment. These factors, linked to fungal enzymes, influence the overall performance of the lignocellulose biorefinery [5]. This project aims to use fungal technologies to upcycle abundant underutilized lignocellulose biomass, creating innovative bio-based materials and industrially relevant enzymes. The main goal is to understand and develop processes using agri-food biomasses to generate mycozymes and mycelium based-materials.

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## Enzymatic de-esterification of bark to aid thermoplasticization

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Bark is a major by-product of forestry industries, with an estimated annual production of about 359 million m<sup>3</sup> [1]. Like conventional lignocellulosic materials, bark is also rich in polysaccharides and lignin [2], which can be chemically modified into thermoplastics. However, bark also contains significant quantities of suberin [3]- a hydrophobic biopolymer of long chain fatty acids (LCFAs), which can potentially interfere with thermoplasticization efforts. To overcome this, it is essential to extract the suberins from bark- this would not only aid in improving the processivity of bark, but will also generate a valuable side-stream with multiple industrial applications [4].

In this work, we have explored a special class of esterases called **suberinases** for their action on releasing LCFAs. We have identified two acidophilic fungi- *Penicillium* sp4. ASM 115 (P-115) and *Talaromyces* sp. AS 616-3 (T-616), with extracellular esterase activity on model LCFA substrates. The crude enzymes were acidophilic and had broad temperature tolerance. The esterase activity was also >10 fold higher on medium (C8, C12) and long chain (C16, C18) substrates, compared to a short chain substrate (C4), indicating their potential for de-esterifying suberin from bark.

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**Effect of moisture on processing and finished parts of dialcohol cellulose affecting design of process equipment**

While thermoplastic behavior of cellulose is challenging, we know that dialcohol cellulose exhibit thermoplastic behavior in terms of softening, increased ductility, and melt processability. However, it is somewhat difficult to process dry dialcohol cellulose, resulting in high process forces and high temperatures causing discoloration of the material. It has been shown that the melt processability of dialcohol cellulose can be improved by addition of water, making it possible to process 100 % partly modified fibers at temperatures below 140°C, without causing discoloration of the material. Water is thus acting as a plasticizer for dialcohol cellulose.

The suggested structure for the heterogeneously modified dialcohol cellulose is a core-shell-structure with amorphous dialcohol cellulose as a shell around a core of more crystalline cellulose [1].

The project aims at investigating combined effects of degree of modification and effects of amount of moisture on the melt processability of dialcohol cellulose and connect processing properties with structural properties of the dialcohol cellulose.

The project also aims at investigating how processing should be done in larger scale and what type of equipment is suitable in terms of screw configuration, equipment set up and connect this with material properties, process parameters and recyclability of the material.

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## **Pattern of substitution affects the extractability of xylan from *Eucalyptus Globulus***

*Eucalyptus* species are today the most important source of paper pulp. However, improving the sustainability of the bio-based processes requires utilization of all the wood fractions for applications of higher value. To that end, detailed structural characterization of the hemicelluloses and elucidation of the structure-recalcitrance relationship is needed. The main hemicelluloses in *eucalyptus* are acetylated glucuronoxylan and glucomannan. The xylan is particularly interesting due to its unusual structure where some of the 4-O-methyl-glucuronic acid substitutions contain a terminal galactopyranosyl residue contrary to other characterized angiosperms [1]. Here our aim is to understand how the galactosyl, MeGlcA, and acetyl groups affect the xylan recalcitrance, and how they are distributed along the xylan backbone.

Following a previously developed method for birch [2], we have extracted *eucalyptus* xylan and mannan by sequential subcritical water method, which preserved all the key molecular features: acetylation, MeGlcA and galactose. Additional alkaline extraction steps ensured nearly complete removal of xylan. The extracts were characterized for the monosaccharide composition, molecular weight, degree of acetylation and the amount of lignin. Enzyme assisted oligosaccharide mass profiling and MS/MS was employed to unravel the distribution of acetylation, MeGlcA and galactose on the xylan. Together with the calculated mass balance we have evaluated how the molecular architecture of the *eucalyptus* hemicelluloses is related to their extractability and how it compares to that of other hardwoods. To that end i) MeGlcA with odd pattern has tighter spacing than MeGlcA with even pattern ii) Gal-MeGlcA substitutions are clustered on the xylan backbone iii) even pattern of acetylation is confirmed showing similarity to previously characterized hardwoods. Comparison hemicellulose structures from different woods allows us to evaluate which structural aspects are fundamental for the formation and function of the woody tissue, and which are more specific responses to the habitat. Differences between and within the polysaccharide structures may also have important implications to the macroscopic fiber properties and the separation of the wood polymers for industrial use.

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### **Lignocellulose biomass sodium periodate modification: an outcome from the possibilities uses for extraction.**

Lignocellulose is a complex biomaterial composed of plant cell walls. It is extremely abundant on the earth's surface, offers substantial potential for sustainable development in bio-based product manufacturing, and is an excellent alternative for petrochemistry replacement [1].

Sodium periodate, a powerful oxidizing agent, has shown promising results in its recycling, making it a versatile tool for modifying cellulose and hemicellulose structures generating aldehydes. These results open up exciting possibilities for environmental and technological applications [2].

While previous research primarily focused on post-extraction oxidation for structure modification, to our knowledge, no work has utilized oxidation modification directly on biomass. In that sense our research aims to investigate periodate oxidation as a method of structure modification to increase the solubility of carbohydrates and prompt the extraction of polysaccharides from biomass [3]

The results revealed that periodate oxidation partially oxidizes carbohydrates to aldehydes in birch wood, enabling their extraction from the matrix for further functionalization. According to it, it was possible to verify a decrement in glucose and xylose with increasing periodate addition during oxidative extraction, titration confirms an elevation in aldehyde formation, and extracts molecular weight align with literature findings.

This pioneering investigation marks the initial steps towards periodate-mediated extraction from biomass raw materials. It offers a new and inspiring pathway towards sustainability and technological innovation.

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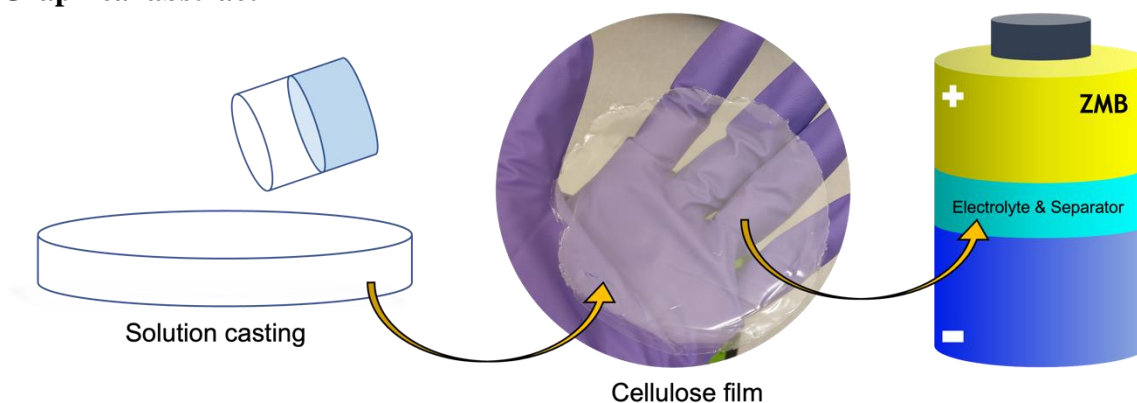
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## **Cellulose-based materials as a separator for zinc metal batteries (ZMB)**

A separator is an essential component in a battery cell, which ensures ions flowing between negative and positive electrodes while also acting as an insulator for electrons. However, the commonly employed glass fibre separator for aqueous zinc metal batteries (ZMB) is too thick (~150  $\mu\text{m}$ ) and fragile. Therefore, the development of thin separator with appropriate mechanical properties is desired to advance the technology. In this work, cellulose-based materials are prepared via solution casting method. The objective of this research is to investigate the effect of different cellulose materials composition on the structure, morphology, mechanical, and electrochemical properties of the separators. The research will employ battery testing techniques to evaluate the performance of cellulose separator for ZMB.

### **Graphical abstract**



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## **Morphological and structural properties of water-swollen cellulose fibres of different degrees of modification**

The use of conventional, oil-based plastics and our end-of-life management take a toll on our planet. Yet, plastics play a significant role in facilitating life as we know it, and, indeed, in many applications, conventional plastics may be the best and safest material to use. Hence, finding wood-based plastics with the same desirable applicability using cellulose fibers is a hot research topic. Native cellulose has a high glass transition temperature, typically beyond temperatures where it starts to thermally degrade, posing a major drawback in processing in the same tools used for conventional thermoplastic polymers. However, one way to make cellulose fibers suitable for thermoplastic processing is to partially chemically modify the cellulose in them to dialcohol cellulose (DALC) while keeping their fibrous morphology. A recent study [1] demonstrates that dialcohol cellulose fibers can be processed by melt processing. The other interesting feature of DALC fibers is that they are hydrophilic. Hence, they swell and exhibit significant moisture sorption [2]. The presence of sorbed water inside the fiber wall aids in better thermal softening and processability. This quality of modified cellulose fibers could be essentially taken advantage of, by using water as a plasticizer and no other polymer matrix as a plasticizer for processing [1]. This entails the necessity to understand: (i) The morphology before processing of modified fibers, and (ii) The effect of water content on the structure of modified cellulose fibers. The structural and morphological details of the modified fibers are studied using X-ray scattering and optical microscopy in the present work.

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## **A study on orientation of wood fiber in extruded biocomposites**

A general procedure for combining material functions and numerical modelling to predict the orientation of highly-filled wood polymer composites (WPCs) in a single screw extrusion and validation thereof is elaborated in this study. Capillary rheometry was used to determine the shear viscosity and wall slip functions as well as the melt density of the biocomposites. The numerical model consisted of a model film die where the melt flow was simulated using Finite Element Method in the generalized Newtonian constitute equation framework. Fiber orientation was modeled using the Folgar-Tucker approach and included fiber-fiber interaction during the process. Reference extrusion tests were performed on a single screw extruded on the biocomposites. The extrusion setup included two melt pressure transducers that were used to determine the die inlet initial conditions (end of the extruder / die inlet) and provide feedback on the wall slip boundary conditions (pressure discharge along the die). Overall, the pressure error between experiments and simulations was less than 6.5% for all screw speeds investigated. Extrudates were produced and the wood fiber orientation was estimated based on SEM micrographs and image analysis and compared with the simulations of fiber orientation. We show that the general procedure outlined can be calibrated to predict the overall orientation distribution of wood fiber biocomposites during single screw extrusion.

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## Azetidinium salt Functionalized Pulp

For the tailored modification of Bleached Kraft Pulp (BKP) and formation of novel properties for packaging and superabsorbent materials, pulp was modified with itaconic acid followed by azetidinium salt conjugation. The modified cellulose fibres were prepared using itaconic acid and its anhydride, effectively grafting carboxylic acid groups onto the fibre surface, before further reaction with the azetidinium salts. As itaconic acid is predicted to become a valuable bio-based chemical for the future, exploration of its applications is favourable for the formation of new materials. Through the modification with itaconic acid itself, the fibres already gain novel properties, however further modification with azetidinium salts gain even more control and other possibilities for applications.

The reaction of itaconic acid or anhydride with pulp fibres occurs through a condensation reaction via the carboxylic acid and hydroxyl groups in the acid and fibre surface, respectively. Depending on the water content, either acid or anhydride may be used, keeping in mind that anhydrides are more reactive, however also are easily hydrolyzed in water. The reactions are performed in a heterogenous mixture of pulp in water with the aid of coupling reagents, through gas-phase reactions with the anhydride, or through high consistency mixing of anhydride with pulp, all gaining itaconic-BKP with increased fibre charge. The conjugation of the Azetidinium salts to the itaconic-BKP was performed in heterogenous mixtures of the fibres in water (Figure 1).

Azetidinium salts were prepared by a two-step process from dialkylamines and epichlorohydrin, producing 3-Chloro-2-hydroxypropyl-N,N-dialkylamines which thereafter were ring-closed into the azetidinium salts. For the verification of successful grafting of itaconic acid/anhydride and azetidinium salts, ATR-FTIR was used, alongside titrations for the assessment of degree of substitution and fibre charge.

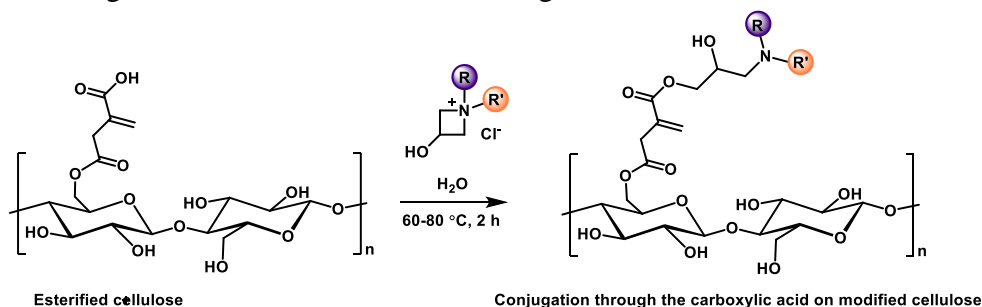


Figure 1. Conjugation of Azetidinium salts to itaconic-BKP



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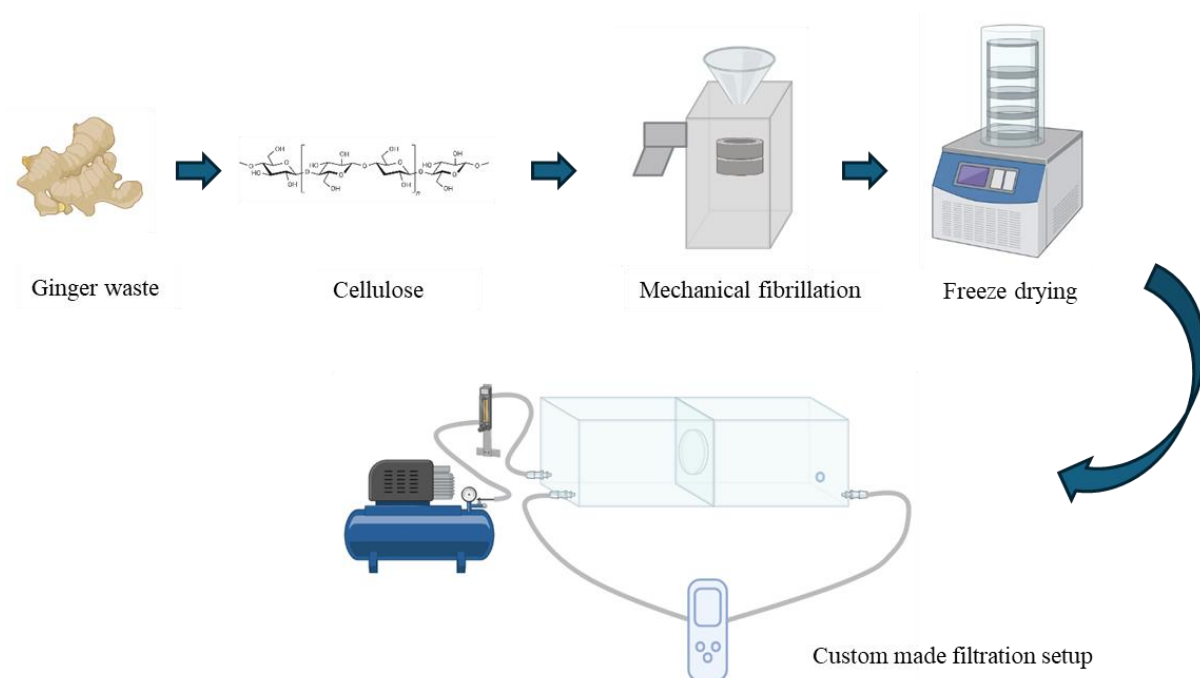
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**Development of cellulose base aerogels for air filtration using juice industry solid residues**

Air pollution and airborne particulate matter create substantial environmental problems and risks to human health. Petroleum-based air filters are produced using non-renewable raw materials, require harsh chemicals during processing, and become non-biodegradable wastes after use. Therefore, sustainable filter materials prepared using environmentally benign methods are in high demand. Fibrillated cellulose-based aerogels can be used as eco-friendly and effective air filter materials. Preparing fibrillated cellulose from waste materials provides additional economic benefits to the process. In this work, lignocellulosic solid residues from the juice industry were used to purify the cellulose fraction, and the extracted cellulose was mechanically fibrillated using a super mass collider. Aerogels were prepared by freeze-drying fibrillated cellulose at different dry-weight concentrations. The prepared fibrillated aerogels were tested for pressure drop, and particulate matter removal using a custom-made setup. The initial results (visual) showed that the produced filters could retain particulate matter (quantification of these results is in progress). The pressure drops of the aerogel filters made using 0.5% cellulose suspension are more similar to those of commercial face masks and almost equal in air flow rates of 1 and 4 l/min. The overall findings of this study highlight the promising prospects of using cost-effective and eco-friendly materials for air filtration, highlighting their potential effectiveness.





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## **Effects of Ring Opening and Chemical Modification on the Properties of Dry and Moist Cellulose - Predictions with Molecular Dynamics Simulations**

Thermoplastic properties in cellulose-based materials can be achieved by ring-opening the glucose units in the glucan polymer chain or by introducing new functional groups. To evaluate the material properties relevant for processability and usability, we employed molecular dynamics simulations to study amorphous cellulose and eight cellulose derivatives. These derivatives were examined in both dry and wet states, with and without ring openings, and functionalised with aldehyde, hydroxylamine, and carboxyl groups. The systems were analysed for their density, glass transition temperature, thermal expansivity, hydrogen bond characteristics, and other relevant properties.

All ring-opened systems exhibited higher molecular mobility, indicated by decreased glass transition temperatures, reduced modulus and strength, and increased water- and self-diffusivity. Consequently, these modifications enhanced the thermoplasticity and processability of the materials compared to unmodified cellulose. Specifically, dialcohol cellulose and hydroxylamine-functionalised cellulose were particularly noteworthy due to their high molecular mobility at elevated temperatures suitable for processing, while still maintaining high stiffness and strength at room temperature.

As expected, increasing water content also improved properties important for processability. Additionally, smaller side groups and ring openings demonstrated that both steric effects and electrostatics significantly influence polymer processability.

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## **Lignin-poly(lactic acid) biopolymer blends for advanced applications – Effect of impact modifier**

Utilizing materials from renewable sources, such as biomass, reduces the environmental impact of conventional polymers and decreases reliance on finite fossil fuels. Lignin, an abundant and highly branched polyphenolic macromolecule, is naturally found in various plants. Its composition, which includes coniferyl, coumaryl, and sinapyl alcohols, varies depending on the botanical origin, harvesting season, and extraction method [1]. Lignin acts as a matrix for cellulose fibres in vascular plants, contributing to their strength and stiffness. Typically, lignin is combusted in pulping mills for energy and is considered a waste byproduct of the pulping industry. However, lignocellulosic biomass, a widely available, carbon-neutral, and renewable bioresource, offers significant potential for sustainable material development. To enhance lignin's compatibility with poly(lactic acid) (PLA), the hydroxyl groups in lignin were chemically acetylated[2] [3]. This modification, combined with the addition of Biomax Strong, an impact modifier, significantly improved the material properties of PLA-lignin blends. The inclusion of Biomax Strong increased the impact strength and thermal stability of these blends, resulting in unique mechanical properties such as reduced tensile modulus and increased ductility. These enhancements indicate that the modified polymers have the potential to perform as well as or better than conventional polymers. Ongoing collaborative research efforts aim to further optimize these materials, fostering an industry-wide transition to more sustainable practices.

This study demonstrates that incorporating an impact modifier into PLA-lignin blends can enhance impact strength and thermal stability, reduce tensile modulus, and increase ductility. The improvements in impact strength and elongation, along with the reduction in modulus, are attributed to better stress redistribution, enhanced material compatibility, and higher energy absorption due to the impact modifier in combination with lignin. The adoption of bio-based materials in material development is driven by their sustainability, design flexibility, economic viability, and environmental benefits, playing a vital role in advancing the circular bioeconomy.

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**Recycling and bio-based aspects for EV interior design**

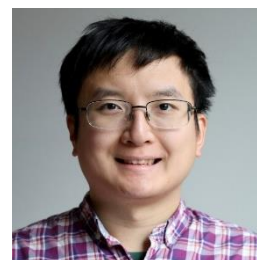
This poster presents the Mission 0 House project where Polestar and Borgstena have partnered with Chalmers University of Technology, to investigate the recyclability and feasibility of alternative feedstock-based polymers for the car interior. The research involves a comprehensive review of the recyclability of bio-based polymers and their blends, focusing, but not exclusively, on polymers like Polyamides (PA), Polycarbonate (PC), Polypropylene (PP), and Polyethylene terephthalate (PET). It explores the compatibility and recycling challenges of commercial and bio-based polymers, particularly when combined, and investigates mechanisms causing material property decline during recycling. It also explores the existing and emerging recycling methods for textile-grade polymers, their development stages, and their potential to achieve zero greenhouse gas emissions. The project also assesses the chemical processes involved and their direct and indirect greenhouse gas emissions. Additionally, the study focuses on recycling regenerated cellulose (e.g., Lyocell, viscose) as mono-materials and in blends with bio-based polymers and natural fibers. The goal is to identify challenges and propose **zero greenhouse gas pathways to efficient polymer recycling**.

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### **Enzymatic processing of steam-pretreated spruce**

Lignocellulose-based processes have the potential to replace various fossil-based processes for the production of fuels and materials. However, the processes often require high temperatures, harsh chemicals, and solvents to process the biomass, which makes them less environmentally friendly. To address this issue, enzymatic treatments could serve as an eco-friendly method for biomass processing. Carbohydrate-active enzymes (CAZymes) have been used to modify wood fibres and create novel materials. For example, glycoside hydrolases (GHs) and lytic polysaccharide monooxygenases (LPMOs) have been used to assist in the fibrillation of wood fibres for the production of nanofibers and nanopapers [1].

Our study will focus on optimising the reaction conditions and applications of LPMOs. LPMOs are a group of monooxygenases that catalyse copper-mediated oxidative cleavage of polysaccharides. It has been shown that LPMOs can stimulate the saccharification of pretreated corn stover by cellulases [2]. However, they are prone to oxidative damage and the activity tends to decrease over time [3], which can be a disadvantage in batch reactions [4].

Even though LPMOs saturated with Cu<sup>2+</sup> have been shown to have a fast reaction rate, they were also inactivated rapidly. On the contrary, unsaturated LPMOs had a more stable and prolonged reaction progress, which could be beneficial to industrial bioprocesses [5]. Therefore, our aim is to test different strategies to improve the stability of the LPMO reaction. The strategies we will focus on are stepwise enzyme addition and Cu<sup>2+</sup>-unsaturated reaction. We want to investigate whether these strategies could lead to a more sustained product formation and a higher final saccharification yield. The study will initially focus on saccharification of mildly steam-pretreated Norway spruce.

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## **Synthetic Polymer-Free Fibreboards Derived from Oil Palm Waste**

Empty fruit bunches (EFB) of oil palm are the abundant lignocellulosic residues from the palm oil mill process, which are commonly discarded as mulch on land or incinerated. In this work, we show a simple method mimicking paper-making process to upcycle EFB fibres into high-value boards without the need for formaldehyde binders. This was achieved by utilising cellulosic fibres from wood pulp as binders. The concept utilises the cellulose network from pulp fibres to hold the otherwise loose EFB fibres together to produce robust and rigid EFB boards. Mechanical refinement was performed using a re-circulating colloid mill to improve the binding performance of the pulp fibres. The results indicate that performing longer refining times to the pulp fibres leads to the production of denser EFB boards with enhanced mechanical properties, air resistance, and particle retention efficiency. This is because the refining stage applied to the pulp fibres induced fibrillation and the addition of fines, enhancing the contact area between the pulp fibres used as binders and the EFB fibres. Furthermore, EFB boards containing 30 wt% of pulp fibres refined for 30 minutes possessed better flexural properties ( $E_F = \sim 2.9$  GPa;  $\sigma_F = \sim 22$  MPa), compared to commercial particle board (PB) and medium density fibreboard (MDF) for furniture application. This work also shows that EFB boards can be moulded in special shapes either in never-dried state or after being formed and dried into panels, with a steaming step introduced to re-wet the boards. This capability presents good potential for furniture and packaging applications that require products with complex shapes. A lifecycle analysis model (LCA) using ReCiPe 2016 method was also developed to measure the environmental impact of upcycling EFB using this technique. The results showed that the EFB boards possessed lower environmental impact on global warming potential and all end-point categories (human health, ecosystem and resources) compared to the commercial MDF. This can be attributed to the absence of synthetic adhesives used as the binder and the avoidance of *business-as-usual* scenarios from EFB.

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## **Wettability of fatty acid esterified cellulose surfaces from molecular simulations**

Surface functionalization treatments can be used to reduce the moisture sensitivity of cellulosic materials, which is particularly relevant for textile and packaging applications. One such approach is to use enzymatic methods to link plant-based lipids to cellulose surfaces. Structure-function relationship studies have shown evidence of long-chain fatty acids (C10–C18) introducing hydrophobic character with water contact angles  $> 90^\circ$  [1,2]. Here, we investigate the hydrophobicity introduced to cellulose with short to long-chain fatty acid esterification using molecular dynamics simulations [3]. We develop software tools for building models of functionalized cellulose microfibrils and surfaces, and use the models to study how fatty acid grafting affects the surface structure, the organization of interfacial water and water-cellulose work of adhesion.

Our simulations show distinct structural effects due to the fatty acid carbon tails aggregating near the substitution sites on the hydrophilic cellulose surfaces. The degree of substitution and carbon tail length both influence the structure of the interfacial water, and medium to long chain fatty acids tend to form a patchy surface with variable water accessibility. Analysis of hydrogen bond density shows the formation of a depletion layer near the grafted surfaces. The water contact angle, as determined through the cellulose-water work of adhesion, shows a linear response to the graft chain length for C4–C12 side chains. After this, the contact area and work of adhesion between water and cellulose remain unchanged, reaching contact angle saturation for long-chain fatty acids. Such computational approach has potential use in the development and optimization of surface modification treatments for lignocellulosic materials.

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## Air-dried rigid bio-foam prepared by the Pickering emulsion approach

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Despite increasing environmental consciousness, producing foam from renewable lignocellulosic materials remains challenging due to issues such as low strength, difficulties in controlling porosity, uneconomical freeze-drying techniques, and poor water resistance. The Pickering emulsion principle is a unique method of making biobased foams that allows the tuning of foam's porosity and mechanical characteristics by controlling the interfacial interaction between the foam components. Although many reports have demonstrated the preparation of dry foam by harnessing cellulose through a Pickering emulsion template, these approaches rely on freeze-drying<sup>1, 2</sup> or oven-drying processes<sup>3, 4</sup> to obtain bio-based dry foams. This study aims to reduce energy consumption during the drying process of foam by designing a pickering emulsion template that allows the fabrication of dry foams at room temperature. The result showed that a stable emulsion of cellulose, lignin, polyvinyl alcohol, and alkene solvent with a smaller droplet diameter (3 – 8  $\mu\text{m}$ ) compared to the previous study ( $\sim 12.5 \mu\text{m}$ )<sup>5</sup> was obtained. Dry foams produced after overnight drying at room temperature had higher strength at 50% strain (272.8 kPa) compared to other studies by using freeze<sup>2</sup> (82.41 kPa) or oven-dried foam<sup>3</sup> (< 50 kPa). Our proposed methodology shows the potential to reduce the use of foam derived from fossil fuels without sacrificing the drying process, desired morphology, and strength properties.

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## **Fabrication of Hydrophobic Cellulosic Foam Using Sustainable Technology**

Biobased lightweight materials have received high attention from both academia and industry as a replacement for fossil-based materials.<sup>1</sup> Cellulose has risen as a very interesting resource to create a range of products that allow us to go from a fossil-based society to a bioeconomy.<sup>2,3</sup> Well-designed cellulosic foams can be applied in variety of applications including mattresses, sealing and insulation products, water purification and many other applications. Cellulosic fibre is highly water-absorbent; they need certain level of hydrophobization to be resistant to wetting by polar liquids such as water. Aqueous foam can be used as a transform medium to form a biobased foam from cellulosic fibres together with chemicals to design the structure according to the intended application. Here, we disclose how to use the biodegradable chemicals during the foam fabrication process to design a hydrophobic biobased foam with high mechanical properties. Chemi-thermomechanical (CTMP) and bleached chemi-thermomechanical (BCTMP) pulps are used for fabrication of hydrophobic foams in this study. We prepare an organocatalytic aqueous formulation (OAF), which contain a hydrophobic suspension derived from alkoxysilanes and an organocatalyst.<sup>4</sup> The combination of OAF, a sustainable polyelectrolyte and pulp, resulted in a hydrophobic foam with high strength property (E-modulus improved from 2.75 to 22 kPa).<sup>5</sup> Elemental mapping images show an even distribution of the OAF and the chitosan at the lignocellulose fibers of the foam. The fabricated biobased hydrophobic foam has a great potential for decontamination applications such as removal of oil from water. All ingredients are approved as non-toxic chemicals and can be readily provided in large quantities, which allows for scalability in a cost-effective manner.

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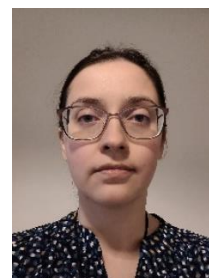
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**Investigation of the effect of the interaction between main components of  
bio-based barrier dispersion coatings on its film formation**

Nowadays, food packaging is a major source of global plastic waste, which is a significant environmental issue. One possible solution is applying barrier coatings, which enable the use of more environment-friendly plant-based materials like paper and paperboard for food packaging by significantly enhancing the barrier properties of these porous and moisture-absorbing materials. Water-based barrier dispersion coatings possess many advantages, such as facilitated repulping, recycling, and, in some cases, composting of the coated plant-based packaging. Their advantages also include low emissions of volatile organic compounds, some of which may be a health hazard. Additionally, barrier dispersion coatings are promising safe alternatives to fluorochemicals in food packaging. However, the film formation mechanism remains at the core of the design of dispersion coatings with good barrier properties, and the development of cost-efficient barrier dispersion coatings still requires substantial research efforts. Currently, interest in bio-based barrier dispersion coatings has increased due to the possibility of an even greater reduction in the use of fossil-based materials. It should be noted that bio-based dispersions demonstrate even more complex film formation mechanisms than synthetic dispersions due to the recalcitrance of many natural materials.

Our study delves into the area of the interaction between the components of bio-based barrier dispersion coatings, shedding light on these coatings' complex film formation mechanisms. We studied the interaction between the bio-based dispersion's core polymer cellulose acetate butyrate with the stabilizer poly(vinyl alcohol). Variations in chemical structure and molecular weight, the effect of the ratio of the components in dispersion, and the size of the particles on the film formation properties were studied. Fourier-transform infrared spectroscopy and quartz crystal microbalance with dissipation monitoring were employed for qualitative and quantitative evaluation of the interactions between the dispersion components. We employed surface-sensitive atomic force microscopy, a mainstay for investigating the film formation process at the nanoscale, enabling the evaluation of various parameters' impact. Moreover, some dispersions were coated onto paperboard, the obtained coatings' morphology was evaluated with scanning electron microscopy, and the coatings' barrier properties were tested. As a result, the effect of different coating dispersion parameters on film formation and coatings' morphology and barrier properties were analyzed, and main trends in the structure-properties relationship were revealed.

This work represents a step towards the development of bio-based dispersion coatings, a promising way for sustainability. By studying these coatings' complex film formation mechanisms, our research contributes to the ongoing efforts to find sustainable alternatives to plastic food packaging.

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### **Dispersion of bacterial cellulose in organic solvents and its assembly as an ultra-low grammage coating on woven fabric**

Current permeable system of chemical and biological (CB) protective suit is mostly multi – layered, consisting of a permeable outer shell fabric for evaporative cooling and an integrated layer of active carbon lining as an adsorbent layer against hazardous aerosol and gas. Aspiring for an enhanced particulate filtration efficiency in the shell fabric layer and hence an extended lifespan of the inner carbon adsorbent, a versatile method was pitched to explore the feasibility of incorporating bacterial cellulose (BC) in protective garment. In this study, BC suspensions prepared in different concentrations and with various organic solvents were first vacuum filtered onto woven cotton fabrics and nonwoven films of BC with grammage  $< 1 \text{ g m}^{-2}$  were achieved. Air permeabilities of these fabricated fabrics were characterised and particulate filtration efficiencies were also simulated with a sodium chloride aerosol challenge in a small – scale wind tunnel. For instance, a prototype coated with  $1 \text{ g m}^{-2}$  of BC – in – water suspension had a filtration efficiency of 83% while sustaining an air permeance of 20 mm/s. Despite the hydrophilic nature of BC, it was discovered that similar particulate filtration performance could be achieved with  $0.5 \text{ g m}^{-2}$  of BC when an ethanol – water mixture was employed during filtration. Filtration efficiency of woven fabric coated with  $1 \text{ g m}^{-2}$  of BC – in water/ethanol suspension also increased from 83% to 93%, hinting a better dispersion of BC in a ‘less polar’ medium. The solubility parameter between different organic solvents and BC was therefore investigated in this work and the potential of realising different porous structure in BC nonwoven films by controlling solvent polarity was demonstrated.

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## **Combined full-field tomography and SWAXS imaging at ForMAX**

With the rising demand for sustainable products, forest-derived materials are crucial for addressing societal and economic needs worldwide. Renewable natural composites, such as plant and wood fibers, exemplify these materials due to their exceptional mechanical properties [1], stemming from the hierarchical arrangement of cellular structures and the ultrastructural characteristics of cellulose microfibrils. Understanding the structural properties of these materials on micro-, nano-, and atomistic scales is essential when considering the development and manufacturing of biodegradable products with desired traits.

Synchrotron-generated X-ray full-field tomography and scattering techniques are invaluable for generating multiscale structural information on wood and other hierarchical materials. At the ForMAX beamline at MAX IV Laboratory [2], the combination of full-field microtomography imaging ( $\mu$ CT) and small- and wide-angle X-ray scattering (SWAXS) allows for in-situ, time-resolved characterization of these materials.

In this project, we characterized the nanoscale properties of cellulose microfibrils in selected microscale structures of young birch branches using a combination of  $\mu$ CT and SWAXS imaging techniques. The microfibrils' orientation and crystalline properties were determined and matched with the cellular structures visible in tomography data. The results emphasize the outstanding suitability of the combined X-ray imaging techniques for the multimodal structural characterization of hierarchical materials and the unique capabilities of the ForMAX beamline.

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**Enhanced Material Analysis through the Integration of Rheological and Scattering Methods**

We present an experimental methodology that integrates rheology with temperature and moisture control in conjunction with Small-Angle X-ray Scattering (SAXS) and Wide-Angle X-ray Scattering (WAXS). This approach allows us to investigate material properties at various length scales simultaneously.

The core of our experimental setup is the Anton Paar MCR702 rheometer, outfitted with a custom-modified SWAXS cell that enables temperature and moisture control. This versatile cell can accommodate different sample holders, making it suitable for a wide range of materials. For solid samples, we use holders for bar-shaped specimens, while for softer materials, extensional fixtures can be used. Liquid samples can be measured using parallel plate, cone and plate or Couette cell configurations. The rheometer is mounted on a movable table at the MAX IV synchrotron in Lund, Sweden, facilitating the investigation of material properties in both time and space during a single experiment. Our system operates within a temperature range of approximately 25 °C to 200 °C. When combined with moisture control, the temperature range is naturally limited but can manage humidity levels from a few per cent up to around Rh = 90%.

The presentation will highlight the versatility of our setup by showing examples of different materials exposed to various humidity and temperature conditions. We will illustrate how this integrated approach provides new insights into the interaction between mechanical properties and structural changes at different length scales, offering a deeper understanding of material behavior that was previously out of reach.

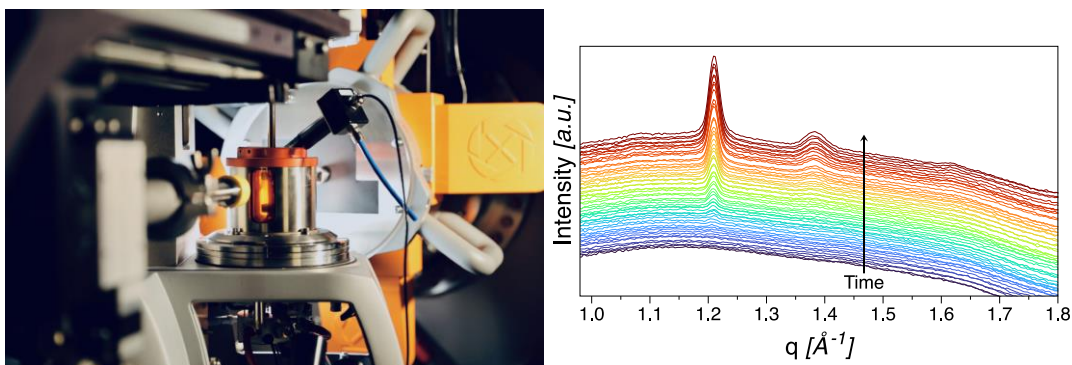


Figure 1 Rheometer with SWAXS cell at MAX IV (left). Example of a time evolution of WAXS signal (right).

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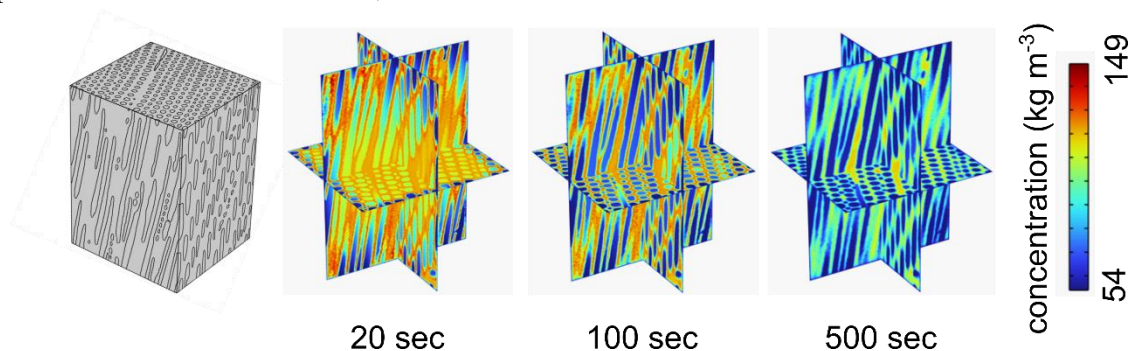
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## **X-ray Computed Tomography aided Finite Element Modelling to Analyse the Hygromechanical Behaviour of Wood**

Microscopic X-ray computed tomography aided finite element modelling (XCT FE modelling) is a popular method within material science to predict material properties and behaviour of heterogenous materials, such as wood. The method uses information from synchrotron or lab-based tomograms to build, calibrate and validate finite element models. This approach allows for situation specific simulations through incorporation of material variability and complexity of geometry. XCT FE modelling can be categorised in five subsequent steps: *source*, *experiment*, *image processing*, *model development*, and *output* (Florisson et. al. 2023). This process is far from automated, which can result in human-induced errors.



**Figure 1:** XCT FE moisture flow simulation of a Norway spruce branch compression wood microstructure (image courtesy to Emmy Lano and Isac Norberg)

At Uppsala University, an XCT FE modelling methodology for wood was developed and the bottlenecks within this process were identified. Two case studies were used to test the methodology and some of its bottlenecks. The first study focused on segmentation and meshing strategies to geometrise the microstructure of wood for finite element modelling. The second study focussed on the proper identification of hygroexpansion properties of opposite wood (OW) and compression wood (CW) found in Norway spruce branches. The properties were obtained with an ex-situ post-mortem scanning approach. By incorporating the variability of material and geometry into a numerical model through XCT, a more naturalistic material behaviour could be simulated and a better prediction of material properties could be obtained.

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**Synchrotron techniques as a new way to look into the fundamentals of the  
pulping process and wood fibre properties**

Considering modern environmental demands to replace fossil-based materials with renewable and biodegradable analogues, the scientific community started to put more attention to understanding the pulping process and its impact on wood fibre structure and properties. Wood is one of the most abundant natural materials, the market value of which is rapidly dropping due to the emergence of electronic and plastic substitutes. In order to be able to use wood components in the production of smart materials and be competitive economically with synthetic analogues, it is necessary to have a good understanding of structure-property relationships. We want to assess this by means of different synchrotron techniques, including Wide Angle X-Ray Scattering (WAXS), X-Ray 3D Nanotomography, and X-Ray Multiprojection Imaging (XMPI).

Kraft cooking is one of the well-established methods for pulp production, and numerous studies were performed previously to characterize the impact of each step on the wood structure and properties [1]. Usually, for characterization such studies require sampling before and after treatments. One needs to stop the experiment at different steps to perform sampling and characterization during the experiment. This may induce false visualization of chemical and mechanical changes. In this work, we attempted to study pulping in situ with WAXS using a specially customized reactor without the need for termination of the experiment. First tests in water were already performed at DESY, P03 beam line, and have proven the potential of the reactor.

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## **Understanding the impact of lignin types on the yield and morphology of cellulose-lignin based fibers: advanced characterization of lignin leaching with magnetic resonance methods**

The shift towards forest-based products aligns with the growing emphasis on sustainability and the need to reduce dependency on fossil resources. Cellulose–lignin-based carbon fibers (CFs) have emerged as a promising alternative to expensive fossil-based polymers used for precursor fibers. However, the performance of the bio-based carbon fibers needs further improvement. One of the persisting challenges is the lignin leaching during fiber spinning which limits the carbon fiber yield and the efficiency of the process.

This work focuses on comparing lignin leaching by varying the molecular weight and sources of lignin: soft wood kraft lignin (SKL), low and high molecular weight fractions of SKL and wheat straw lignin are used.

Magnetic resonance imaging (MRI) is used for an in-situ characterization of the lignin leaching on a model set-up for filaments coagulation<sup>1</sup>. The MRI visualizations and evaluation of the mass transport during the coagulation are complemented with UV absorbance measurements on coagulation baths during a spinning process to determine the amount of leached lignin. The spun fibers are analyzed using Klason analysis and gel permeation chromatography (GPC) to assess the lignin content. Additionally, liquid-state NMR is used on the different lignins and on dissolved spun fibers<sup>2</sup> to examine structural changes in lignin and cellulose, as well as any potential linkages or interaction points between the two molecules.

The combined methodologies suggested that the lignin leaching is very dependent on the molecular weight and the chemical structure of the lignin. A substantial lignin content decrease is noticed when lignin with higher content of carboxylic acid groups is used – i.e. wheat straw lignin - followed by the low molecular weight fraction of SKL.

Another part of this study demonstrated that lignin leaching likely results in defects, defibrillation, and pores in the spun fiber. Conversely, a lower lignin content in the spun fiber led to an apparent increase in tensile strength, likely due to the relatively higher cellulose content.

Maximizing the potential of bio-based carbon fibers remains challenging. Emphasis should be placed on minimizing lignin leaching and optimizing the operating conditions for fiber spinning to improve their quality and performance.

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**Highly thermal conductive bio nano composite with anisotropic properties**

Wood polymer composites (WPCs) are essential in the move towards renewable materials, as they significantly reduce reliance on plastics. However, WPCs typically lack high thermal conductivity, limiting their application. This study presents the pioneering manufacture of 2D Hexagonal Boron Nitride (HBN) and wood fiber (provided by Stora Enso) reinforced Poly Lactic Acid (PLA) composites using extrusion. Initially, the wood fibers were coated with HBN, then compounded with PLA, and subsequently the used as input material in capillary extrusion. Prior to extrusion, the rheological properties of the composites were examined using a rotational rheometer, and extrusion parameters were adjusted based on the composite viscosity. The composites exhibit excellent anisotropic properties due to the orientation of wood fibers and HBN in the extrusion direction. Various compositions, including 15 wt% wood fiber (WF), 45 wt% HBN, 30 wt% WF-30 wt% HBN, and 45 wt% WF-15 wt% HBN, were analyzed. Thermal conductivity tests revealed that the 30 wt% HBN-30 wt% WF composite achieved a 1000% improvement in thermal conductivity compared to pure PLA in the flow direction. Notably, the perpendicular direction to the flow showed a 100% improvement, underscoring the remarkable anisotropic characteristics of the extruded composites.

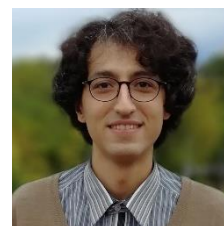


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**Transforming Poplar Wood to high performance bio composites**

**Abstract:**

Wood is a renewable, CO<sub>2</sub>-storing natural material with widespread use in the construction sector, thanks especially to its favorable mechanical properties. Its combined high specific strength and stiffness make it useful as a core material for advanced structural sandwich panels, and its unique structure allows its use for developing advanced functional materials such as membranes and nanogenerators [1]. However, compared to other engineering materials and composites, wood has important disadvantages such as moisture-driven swelling and shrinking, and low mechanical properties in the wet state [2, 3].

We report a novel, fully bio-based high-performance composite obtained by reconstructing wood. This composite was developed by the delignification of poplar wood followed by its relignification i.e. impregnation with Kraft lignin. The physical and chemical properties of the reconstructed wood, and especially its microstructure, were investigated using a combination of scanning electron microscopy (SEM), fluorescence imaging, X-ray diffraction (XRD), and small-angle X-ray scattering (SAXS), and tensile experiments. The microstructure of reconstructed wood displayed strongly deformed cell walls, which helped forming cohesive interfaces. Compared to native wood and densified wood, the reconstructed wood exhibited significant superior mechanical properties, reaching a tensile strength of about 250 MPa and yield stress of 130 MPa. In addition, this material displayed a very high-water resistance. Reconstructed wood, born from the essence of wood, holds great promises as a more sustainable solution for construction and manufacturing of high-performance materials.

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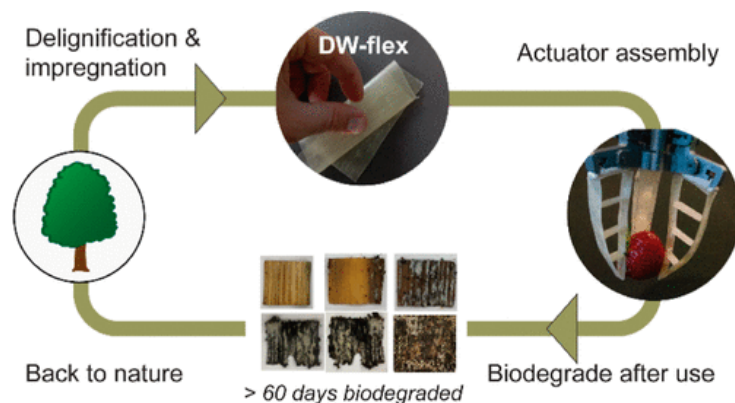
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## Biodegradable and Flexible Wood-Gelatin Composites for Soft Actuators

Compliant materials are essential for many emerging soft robotics applications. Concerns about sustainability and end-of-life options for these materials are increasing, as they are mainly petroleum-based and non-recyclable. Although alternative bio-derived soft materials like gelatin have been explored, they often fail to meet the mechanical performance requirements for soft actuating systems. To address this, we reinforced a compliant and translucent gelatin-glycerol matrix with structure-retained delignified wood, creating a flexible and fully bio-based composite called DW-flex. This DW-flex composite exhibits highly anisotropic mechanical behavior, with greater strength and stiffness in the fiber direction and high deformability perpendicular to it. Introducing such anisotropy into otherwise isotropic soft materials enables more complex movement patterns. To demonstrate DW-flex's capability and potential, we developed and modeled a fin ray-inspired gripper finger that deforms based on a twist-bending-coupled motion, adjustable by changing the fiber direction. Additionally, we designed a proof-of-concept demonstrator suitable for gripping a soft object with a complex shape, such as a strawberry. We show that this composite is entirely biodegradable in soil, offering more sustainable solutions for soft actuators in robotics applications.



**Figure 1.** Graphical abstract of this work. Wood is delignified and infiltrated by a gelatin-glycerol matrix. Then, a gripper is assembled for gripping soft objects with a complex geometry. The composite can finally be biodegraded and returned to nature.

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## **Development of active polylactic acid (PLA)-based packaging film containing artichoke (*Cynara scolymus*) leaf extract and enzyme**

The global population growth has led to an increased demand for minimally processed food, resulting in a limited shelf life of food products and high consumption of synthetic plastic packaging [1]. Consequently, the food packaging industry is seeking bioactive packaging materials that can prolong the shelf life of food while providing a sustainable alternative to non-degradable plastics [2]. Meanwhile, plant residues, such as artichoke leaves, are often discarded, creating environmental concerns. Global artichoke production is estimated at 1978 Kton annually, with 70-80% discarded in the form of leaves, stems, and roots [3]. However, artichoke leaves are rich in bioactive compounds with potential use in active packaging. Therefore, this study aims to formulate a compostable and active PLA-based packaging film by incorporating a PLA-degrading enzyme and bioactive artichoke leaf extract (ALE). For this purpose, ALE was recovered using subcritical water extraction based on previously optimized conditions in a two-cycle process (solid-to-liquid ratio of 1:10, temperature of 180 °C for 15 minutes). The bioactive PLA-based blend was formulated with 0, 2.5, and 5% wt (of total solids) of both ALE collected from the second cycle and PLA-degrading enzyme, and a homogeneous mixture was prepared using a co-rotating twin-screw extruder. Stretch films with a thickness of around 240-270 µm were produced by compression molding and characterized for their mechanical properties and bioactivity. The antioxidant results for the PLA-based films showed the highest activity for the film containing 5% of both ALE and enzyme, with 81.07, 61.14, and 74.94% DPPH scavenging activity after three oxidative cycles for 15-minute intervals. Mechanical analysis indicated that the addition of ALE and enzyme decreased the tensile strength from 80.36 MPa in the control film to 54.98 MPa in the film containing 5% of both enzyme and ALE. Similarly, Young's modulus values decreased from 2138.93 MPa in the control film to 1797.84 MPa in the film containing 2.5% of both enzyme and ALE. The softening effect of the ALE and enzyme was beneficial for stretch films, intended for wrapping food products of various shapes and sizes. Additionally, the bioactivity results are promising for enhancing the shelf life of food products and reducing food waste.

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**Keywords:** Active packaging, Food loss, circular economy, Plant waste

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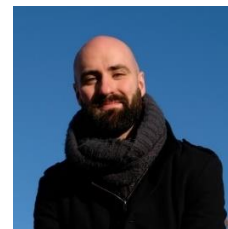
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**Utilizing wheat straw as a lignocellulosic feedstock for thermoplastic materials: pulping, upscaling and modifications**

Wheat (*Triticum*) is an annual cereal grain plant in the grassy family, constituting almost 50% of the total grain harvest in Sweden. Within the Vinnova Competence Centre: FibRe, we are devoted to turning wheat straw biomass into thermoformable materials with as little modification as possible. The aim is to gain in-depth understanding of how to create a lignocellulosic fibre feedstock from straw and further make the material thermoformable, based on the unique spatial and compositional structure of the straw tissue.

First, we devised a systematic methodology for extracting lignocellulosic feedstock from wheat straw in a mild yet efficient process that safeguards wheat straw's cellulose fiber integrity, while minimizing lignin content. Straw was pulped via the kraft process and hydrothermal soda treatment. Systematic variations of process parameters were made according to a five-level and three-factor central composite design (CCD). Response surface modeling (RSM) revealed factor-response relationships and synergic effects between time, temperature, and NaOH concentration on the lignocellulosic yield and composition. The wheat straw lignocellulosic pulp could position itself on par with its wood pulp counterpart by optimizing these parameters.<sup>1</sup> Alkalinity was shown to have the largest individual effect on all the responses. Alkalinity-time emerged as the most influential interacting factor to enhance pulp yield. Regarding cellulose content, the interaction between alkalinity and temperature exhibited a notable effect while interactions between alkalinity-time and temperature-time had a synergistic effect on reducing lignin content.

The CCD-RSM work provided input to make an informed decision on parameter settings for pilot-scale production of wheat straw pulp with the highest cellulose content combined with low lignin content and high yield, yielding a total of 1.7 kg of wheat pulp.

Different chemical paths for bringing thermoplastic properties have been investigated within the project. However, the heterogeneity of the feedstock presents a challenge to determine at which level the modifications occur. Here, the polymerization on fibres' surface is discussed to provide thermoformability and processability to the material. Different polymer modifications were explored and analysed by FT-IR, SEM, DSC. Films has been prepared by hot-pressing to simulate an application for the packaging industry.

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**Dynamical mechanical analysis of isolated and plasticized native, residual,  
and technical lignin: molecular structure, phase morphology, and glass  
transition.**

Lignin in biomass, pulp, and isolated during pulping all have great potential as renewable and inexpensive thermoplastic components and materials. Still, widespread utilization is hindered by high glass transition temperatures ( $T_g$ ), brittleness, and poor flow properties. This study aims at elucidating what molecular structures govern lignin thermoplasticity and how these can be efficiently manipulated. To accomplish this, lignin was first isolated of high yield and purity from wheat straw, Norway spruce, and softwood kraft pulp, and, in addition, a softwood kraft lignin was fractionated into molecular-weight groups. These lignins were characterized by size-exclusion chromatography, Fourier transform IR and,  $^{31}\text{P}$  nuclear magnetic resonance spectroscopy. The  $T_g$  of these lignins and their relative dynamic moduli were determined using a novel dynamic mechanical analysis (DMA) approach for non-self-supporting materials. Applying the Flory-Fox equation for modelling the  $T_g$  revealed that residual lignin in pulp had an apparent free volume more similar to that of kraft lignin than isolated native lignin. However, the chain length had a larger influence on the flow properties than the degree of condensation. To elucidate the role of lignin structure in plasticization, the four different lignins were blended with three plasticizers of different functionalities (protic, aprotic, and aromatic) selected based on the hypothesis that various lignin structures would be susceptible to varying types of plasticizing mechanisms. The viscoelastic properties of the resulting blends were studied using the DMA setup. Contrary to the hypothesis, the plasticizers exhibited remarkably consistent  $T_g$ -depression and mechanical damping profiles across all four lignins – except when phase separation occurred. The phase separation of lignin plasticization is surprisingly understudied, as most literature is focused on  $T_g$  and specific interactions. The solubility of the plasticizers in the lignin, which could only be hinted at using Hansen solubility parameters, appears to be the governing factor of lignin plasticization at higher (> 20 wt%) concentrations. Additionally, the impact of external plasticization was not limited to reducing  $T_g$ ; it also led to a narrower transition, suggesting a more homogeneous ordering of the material.

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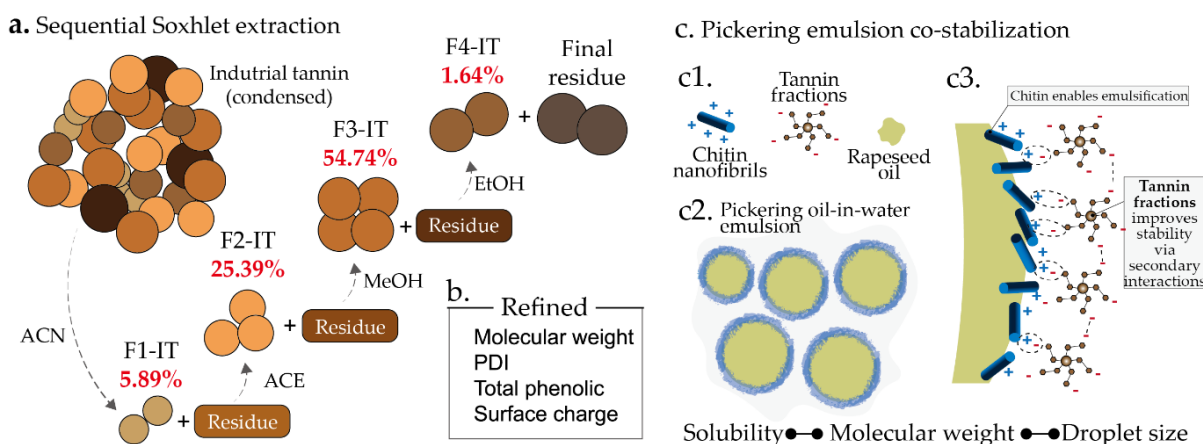
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**Refined industrial tannins via sequential fractionation: exploiting well  
defined molecular structures for controlled performance in Pickering  
emulsions co-stabilized with chitin nanofibrils**

Tannins from *Acacia mearnsii* (black wattle) are one of the few industrially available sources of non-lignin polyphenols.<sup>1</sup> The polydispersity found in industrial tannins (IT), related to chemical functionalities and molecular size, limits their high value-added applications.<sup>2</sup> In this work we employ a solubility-centered sequential fractionation (SCSF) to obtain homogenous tannins fractions, which are then used to control the microstructure and properties of oil-in-water emulsions co-stabilized with chitin colloids. The molar mass distribution and polymerization degree of IT were controlled simply via solubility, with a direct correlation with the emulsion droplet size. Addition of fractionated tannins to chitin stabilized Pickering emulsions was furthermore shown to be a viable strategy to engineer the viscoelastic properties of the emulsion, control and enhance its stability, as well as introduce antioxidative properties. Overall, we demonstrate a facile method to finely control the properties of IT and enabled the customization of tannin, allowing broader utilization in multiphase systems.



**Figure 1** Refining of industrial condensed tannins by sequential extraction and use of well-defined fractions in Pickering emulsion co-stabilization with chitin nanocrystals.

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## **Transparent and water-resistant cellulose nanocrystals (CNC) films with conductivity organic polymers**

Sulfated cellulose nanocrystals (CNC) is a kind of renewable biopolymer owning Hiral nematic structure in the water and good transparency when making films. In this study, we combined the conductive organic polymers (COPs) with CNC to create conductive CNC nanofilms. In-situ and ex-situ polymerization of COP monomers in the CNC template were employed to produce the transparent conductive films. A series of crosslinkers including sodium hypophosphite (SHP), 1,2,3,4-butanetetracarboxylic acid (BTCA), and ethylene glycol diglycidyl ether (EGDE) were selected to improve the water resistance of resulting CNC film. Combining with triethanolamine (TEOA), as a plasticizer, can improve the flexibility of CNC/COPs films. Various characterization techniques, including polarized optical microscopy, ATR-FTIR spectroscopy, and sheet resistance measurement, were utilized to analyze the films. The lowest sheet resistance achieved for the CNC/Pyrrole film was  $2.2 \times 10^8$  ohm/sq using the ex-situ method. Overall, this study creates flexible films with transparent and water resistance properties, paving the way for optoelectronic applications.

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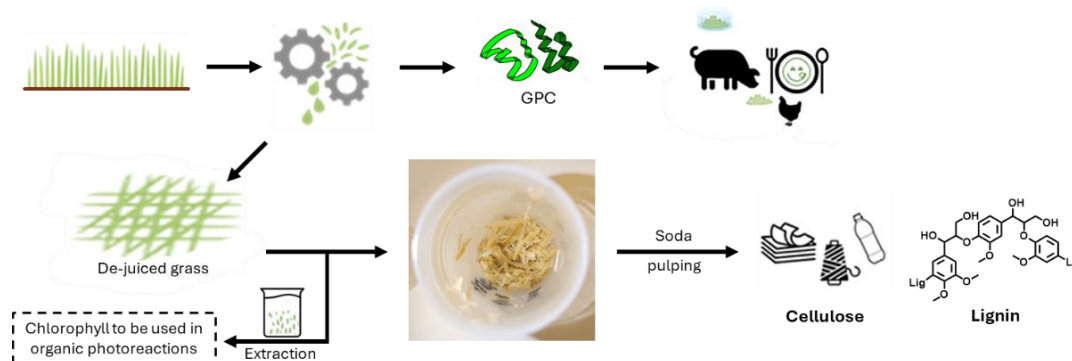
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## Refining cellulose and lignin from de-juiced grass-clover side-stream

Green Protein Concentrate (GPC) is an extract from green perennial biomass - such as grass-clover, alfalfa and ryegrass - used to replace feed ingredients for animals.<sup>1</sup> The fibrous de-juiced biomass is a side-stream containing chlorophyll pigments, cellulose, hemicellulose, and lignin. Chlorophyll can be extracted using organic solvents, while the isolation of the remaining biopolymers requires further pulping to obtain materials with added value. Cellulose, for example, can be used in textiles and packaging, as an alternative to fossil-based sources.<sup>2</sup> Our main focus is to optimize a soda pulping of this side-stream to produce a so-called dissolving pulp that meets the requirements to be used for production of textile fibers.



We tested different conditions in soda pulping, varying parameters such as temperature, time and reactants ratio. We also used different organic solvents during the chlorophyll extraction to evaluate the effect on the structure of the final pulp. Our goal is to find an optimal balance between the chlorophyll extraction and the isolation of cellulose, as well as preserving the lignin obtained from the pulping liquor. We assess the quality of the pulp and lignin based on several characterization techniques: gel permeation chromatography, NMR and UV-Vis spectroscopy, intrinsic viscosity, optical microscopy, as well as the contents of ashes and sugars.

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**Changes in conformation and dynamics of arabinoxylan after modification via periodate oxidation, reduction and etherification**

Chemical modification of polysaccharide chains has been reported to affect their conformation and dynamics [1-3]. In this work, we are interested in how the conformation and dynamics affect their solubility, in the context of chemical modification and coating applications, and in thermoplasticity, for applications where they could be an alternative to fossil-fuel based plastics. To understand how these changes contribute to modified arabinoxylan's (AX) thermoplasticity, we utilize small-angle X-ray scattering (SAXS) to determine changes in the chain conformation in dispersions. To investigate changes in the materials' dynamics, we have measured the relaxation times of AX and their modified counterparts in the solid-state with NMR ( $T_{CH}$ ,  $T_{1\rho}(H)$ ,  $T_{1\rho}(C)$ ).

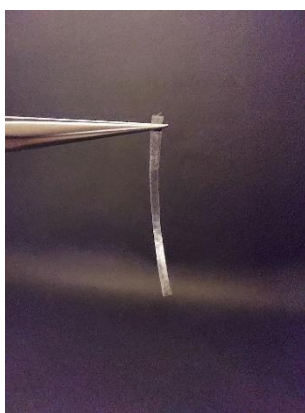


Figure 1: Compression molded film of modified AX after tensile testing

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## **Mycelium-bound material from carrot waste for packaging application**

As economic expansion accelerates and resources are significantly reduced, there is an urgent need to shift industrial products toward sustainable alternatives. Materials bound with mycelium are particularly notable in this context due to their biocompatibility, and environmental friendliness. In such products, mycelium plays a vital role by binding components within composite structures. Residues generated in juice production process (fruit and vegetable wastes), contain sugars and other nutrients essential for growth of microorganisms, at the same time, they contain large fraction of cellulose which can be used for development of materials. This study employed carrot pomace to cultivate *Aspergillus oryzae* and *Rhizopus delemar* via submerged fungal cultivation in bench-scale bioreactors. The harvested materials, comprising fungal biomass and carrot pomace non-digested fraction (containing cellulose), were used to produce mycelium-bound material with paper-like properties through a wet-laid process, followed by post-treatment with glycerol. The physical and mechanical properties of the resulting materials were assessed. The mycelium-bound material containing *Aspergillus oryzae* demonstrated an average porosity of 36  $\mu\text{m}$  and tensile strength of 48.8 MPa, whereas that containing *Rhizopus delemar* showed a porosity of 45.7  $\mu\text{m}$  and tensile strength of 31.5 MPa. The SEM image of the surface of *Aspergillus oryzae*-material revealed mycelium with a long microfibrillar structure distributed in the matrix of carrot pomace particles, whereas *Rhizopus delemar*-material exhibited mycelium with a broader diameter and shorter length. Moreover, the material containing *Aspergillus Oryzae* displayed a higher water contact angle ( $57^\circ$ ) than that containing *Rhizopus delemar* ( $23^\circ$ ), indicating a higher hydrophobicity. The glycerol post-treatment reduced the average porosity and negatively affected the tensile strength. However, it increased the elongation at break by 60%, resulting in a flexible material suitable for use as a wrapping paper. The materials property charts verified that the mechanical properties of mycelium-bound material are comparable to those of conventional paper products. This study provides a foundation for a fungal biorefinery strategy that unlocks the value of carrot pomace, enabling the creation of innovative, paper-like materials with potential applications in the packaging sector.

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**Extraction, structural characterization and bioactivity of polysaccharides from agro-industrial by-products towards functional packaging**

A relevant amount of by-products is generated during the agro-industrial operations, which have a significant negative impact on the environment and also on the social and economic perspectives [1]. These by-products are considered wastes or low value-added products, and they are frequently disposed in landfills, incinerated, or partially used in animal feed [2]. As source of dietary fibers and bioactive compounds, distinct by-products appear as valuable resources for the food, cosmeceutical and pharmaceutical sectors [3,4]. In the present study, microwave-assisted extraction was applied to investigate the recovery of bioactive polysaccharides from wheat, citrus and coffee byproducts. The influence of different extraction conditions in terms of time (5, 10, and 15 min) as well as temperature (140°, 160°, 180°C) on the structure-functionality of the recovered polysaccharides were investigated. The different extracts were investigated by size-exclusion chromatography (SEC), Ion-chromatography (IC), high-performance liquid chromatography-photodiode array (HPLC-PDA) elucidating the molecular weight, monosaccharides composition, and to identify attached phenolic compounds, respectively. Polysaccharides bioactivity was investigated in function of its antioxidant activity. The results revealed the potential of the extraction technique on the recovery of agro-industrial by-products polysaccharides, which can be fine-tuned to improve the recovery yields and the functionality of the target molecules for further applications in functional packaging materials.

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## **White photothermal textile by incorporating pectin, a fruit-waste compound**

Preserving human thermal comfort in a cold environment is important for various activities. Currently, photothermal textiles are being developed by using carbon-based materials to harness solar energy as a heat source to tackle the cold climate. However, dark coating hinders practicality in visual possibilities and sustainability issues resulting from the production and disposal of inorganic materials encourage utilizing renewable biobased resources. Here, we introduce bio-based photothermal fabric by integrating pectin into the man-made cellulosic fibers which can absorb ultraviolet light (UV) from the sun for heating, offering warmth to the wearer and can be a solution for energy-saving outdoor clothes. We introduce bio-based photothermal fabric by incorporating pectin derived from fruit waste into man-made cellulosic fibers obtained by dry-jet wet spinning. These fibers can efficiently absorb UV light from the sun for heating. This thermal energy can be retained by the fabric, offering warmth to the wearer. The obtained photothermal textile tackles the challenges of current photothermal textiles, which explained above. The photothermal fibers were obtained by dispersion of pectin particles into the ionic liquid and subsequently adding cellulose pulp. Then, the photothermal fibers spun with the dry-jet wet spinning method via Ioncell technology. Based on the photothermal characterization of the fibers, it was observed that by adding only 1 wt% pectin into the cellulosic dope, the temperature of the obtained fiber contributed to a 6.2 °C greater due to the photothermal conversion capability compared to the pure cellulose fiber under 100 mW/cm<sup>3</sup> sun power. In addition to this functionality, the obtained photothermal fibers have white color which can be died in desirable colors. The development of photothermal textiles by the addition of pectin as a renewable biowaste enhances the value proposition of wood biomass by expanding its application range beyond traditional uses. Integrating biowaste into the cellulosic textile provides smart functionality while reducing reliance on non-renewable resources and adding value to the traditional textile on the market.

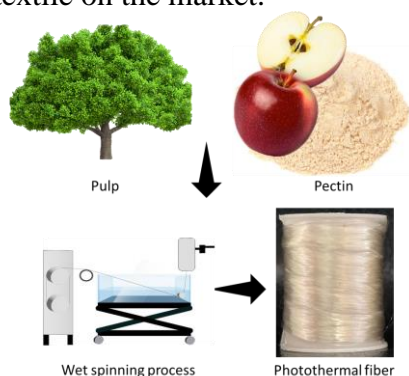


Figure 1. Schematic illustration of photothermal fiber.

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### **Effect of fines on cellulose-rich materials modified by a combination of glycerol and ultrasonication**

Polymeric materials are essential in today's modern society and their production has increased exponentially since their commercialization in the 1940s. One of the largest industrial sectors is packaging, estimated to be 146 million tons in 2015. A large fraction of the packaging material is single-use and ends up in landfill or is incinerated, as most plastics used are non-degradable and recycling is still challenging in large parts of the world. Material research has focused on decreasing the environmental impact of these kinds of materials by reducing and replacing them with, for example, cellulose-based materials like cardboard, but also more innovative materials like bio-based foams. Cellulose is an abundant biopolymer already widely used and bio-degradable and recyclable. One limiting factor for conventional cellulose-based materials is the limitations when shaping cardboard and paper-based materials. Although cellulose is a polymer it does not show typical polymeric behaviors such as thermo-processability which most synthetic polymers do. Our approach, inspired by the plasticization of gluten, is to incorporate a small amount of plasticizer into the lignocellulose matrix. We hypothesize that the small plasticizing molecules will reduce the strong intermolecular interactions within cellulose and enhance molecular mobility. Successful implementation will increase the lignocellulose's thermo-processability and yield a material that can be processed through conventional thermo-processing such as extrusion or hot-pressing allowing more advanced shaping opportunities than existing methods.

During the last three years, we have been working with cellulose to see how it may be modified with the help of glycerol, sorbitol, and starches to achieve a behavior that imitates thermoplasticity. Our work has shown that glycerol that is incorporated into fibers through ultrasonication and then formed into sheets can increase their ductility from 0.5% to 30% without losing significant mechanical integrity.<sup>1</sup> However, that material is still quite paper-like in its structure and therefore we have combined this approach with a high-sheering mixer to increase the homogeneity of the material. To increase the processability of the material small amount of starch is added to form a fully bio-based material with interesting properties that can be hot-pressed into partially translucent sheets, consisting of >60 w% lignocellulose.

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# **Ionic liquids and penetration pathways in wood: methodologies development for mass transport analysis**

**P45**

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In recent years, the ionoSolv process has gained attention as a promising wood pretreatment technology within the biorefinery sector. This relies on low-cost, thermally stable protic ionic liquids (ILs) selectively dissolving lignin and hemicelluloses from wood, leaving a cellulose-rich pulp and enhancing processing efficiency. This process has been successful for a variety of biomass feedstocks, such as softwood[1]. Mass transport events play a crucial role in governing the rate at which wood is delignified, determining the overall efficiency of the pretreatment process. Previous work analyzing the mass transport of ILs into wood, highlighted that ILs transport into the wood structure is the rate-limiting step in the ionoSolv pretreatment[2]. Due to the different solubility of these ILs for the wood components, both the diffusion pathway and mechanism could differ from those observed in traditional wood fractionation processes. Therefore, it is important to examine where the ILs are located and how they are distributed within the fiber structure. The aim of this research is to develop methodologies to investigate the mass transport of ionic liquids in the fiber structure of wood pieces and the subsequent diffusion of lignin within cellulose fibers. Wood samples (Norway spruce, softwood) are impregnated with protic ionic liquids (e.g. N,N-Dimethylbutylammonium hydrogen sulfate, [DMBA][HSO<sub>4</sub>]), and further reacted at 150 °C. Three key aspects are examined: depth of ILs penetration into wood; uniformity of their distribution within the wood matrix; specific location of ILs within the cell walls across different wood sections. Chemical and morphological profiles from different structural levels will be obtained by combining characterization techniques such as Time-of-flight secondary ion mass spectrometry (TOF-SIMS), and wide/small angle X-ray Scattering (W/SAXS). By integrating the findings from these characterization methods, new insights can be gained to understand the transport pathways involved in the ionoSolv process for wood delignification. Establishing and optimizing a solid methodological framework could ultimately allow to take full advantage of the unique properties of ILs and their application in wood fractionation processes.

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## Transforming Lignocellulosic Materials into Melt-Processable Plastics Using Microcompounding and Compression Molding Processes

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In this study, we present a comprehensive methodology for transforming lignocellulosic materials into melt-processable plastics, using periodate oxidation and sodium borohydride reduction of arabinoxylan (AX) and delignified wheat bran. The periodate oxidation of freeze-dried AX, followed by NaBH<sub>4</sub> reduction, yields dialcohol AX, which is subsequently etherified with butyl glycidyl ether (BGE) to produce BGE-DiolAX. Similarly, delignified wheat bran undergoes periodate oxidation and NaBH<sub>4</sub> reduction, followed by BGE etherification.<sup>1,2</sup>

The BGE dialcohol AX and BGE dialcohol cellulose-AX materials were successfully processed into extrudable filaments and compression moldable films using microcompounding and compression molding techniques (see Fig.1). Resulting analysis revealed that both materials exhibited suitable melt flow indices for extrusion and compression molding. Scanning electron microscopy (SEM) showed denser morphological structures, particularly in BGE dialcohol cellulose-AX, which also demonstrated superior thermal stability in thermogravimetric analysis (TGA). The modified AX exhibited thermoplastic behavior, becoming highly flexible and flowing above 130 °C, making it a viable alternative for oil-based thermoplastics.

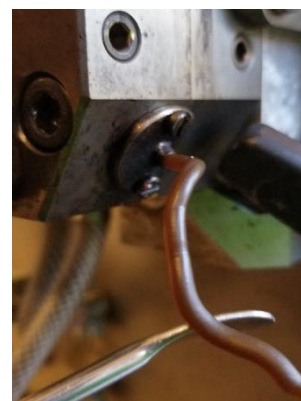


Fig. 1. Processing of BGE Dialcohol AX using microcompounder (Mc)

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## **Effect of fiber aspect ratio on the extrusion instabilities in wood polymer biocomposites**

Wood polymer composites (WPCs) are key industrial ingredients in the context of overarching societal efforts to promote and increase the use of renewable materials. Specifically, the use of wood fiber-based biocomposites as renewable materials can lead to a significant reduction in the consumption of plastic materials. The WPCs employed in this study are custom compositions based on the DuraSense® by StoraEnso, comprising polypropylene reinforced with different wood fiber concentrations (up to 40 wt%) and different aspect ratios. Usually, the extrusion flow of unfilled polymers exhibits a smooth surface (no instability) at low shear rates, followed by the appearance of instabilities such as sharkskin, stick-slip, and gross melt fracture at higher shear rates. However, in stark contrast to the dynamics of neat polymers, the extrusion flow of WPCs can be dominated by the presence of so-called surface tearing instabilities, even at very low shear rates. Melt flow instabilities (MFIs) of WPCs were mapped in single-screw extrusion via inline optical spectral analysis for the first time in this study. The MFIs were detected and quantified by applying an inline image analysis with the help of an optical visualization setup placed at the die exit. Further, space-time diagrams were constructed, subsequently, the temporal and spatial periodicities of the MFIs were determined as a function of shear rate, and Weissenberg number using 2D-Fourier transform analysis. Fourier transform spectrograms represent different characteristic frequency peaks for various instabilities. Surface tearing appeared from the lowest shear rates investigated, followed by a gradual decay in spectral intensity with increasing shear rates/slip velocities. Increasing shear rates, drying, wood fiber content, and increasing fiber size showed mitigating effects on surface tearing. However, surface tearing in undried samples was still present even at the highest shear rates and high wall slip velocities. A regime where the extrudate surface is dominated by bubbles at high shear rates and low wood fiber contents in undried WPCs was identified [1].

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### **Detection of extrusion instabilities using machine learning**

Instabilities during the extrusion process can lead to product defects due to factors such as screw speed, die dimensions, and material. The instabilities such as sharkskin, stick-slip, melt fracture for neat polymers, and surface tearing for highly filled composites can be observed in extrusion. To tackle these instabilities, we utilized machine learning algorithms on recorded extrudate videos. We varied the screw speed (shear rate) from 1 to 150 rpm at a ramp rate of 0.1 rpm/s to observe its impact on extrudate surface patterns. Our study involved three different materials: 5 wt% wood fiber polypropylene composites (undried), and 40 wt% wood fiber Polypropylene from Stora Enso AB, and linear low-density polyethylene (LLDPE), each exhibiting different extrusion instabilities. We employed the VGG16 network, Principal Component Analysis (PCA), and the k-Nearest Neighbors (KNN) algorithm for unsupervised learning to classify these materials. Our approach successfully developed an unsupervised clustering algorithm for materials, contributing to materials science and computer vision with a novel classification method. The consistency of our classification results is in good agreement with our previous study, which involved spectral dynamics analysis obtained from 2D Fourier Transform on the extrudate videos. [1].

[1]. Pashazadeh, Sajjad, et al. "Mapping surface defects in highly-filled wood fiber polymer composite extrusion from inline spectral analysis." *Composites Science and Technology* 242 (2023): 110133.

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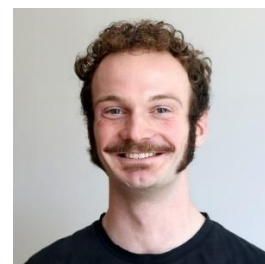
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## **Fungal Valorisation of Recalcitrant Forestry Side-Streams**

The adoption of a circular bioeconomy is vital for countering the many environmental challenges facing contemporary society in the 21st century. Of particular focus is the transition from fossil-derived materials to biologically derived materials. In Europe, forests have been attributed a high importance for this bio-material transition due to their high biomass production capacity [1]. Currently, forestry is regarded as a relatively low-tech industry with large room for development. Conventional processes in mills and biorefineries produce large volumes of low-value and recalcitrant side-streams which are regarded as waste and often burned as biofuels, landfilled, or decomposed [2].

The applicability of fungal biotechnology for this side-stream valorisation will be investigated. Specific focus will be paid to fungi with the capacity to produce carbohydrate active enzymes (CAZymes) to aid in the processing of recalcitrant lignin-carbohydrate complexes. Unlike the energy-intensive Kraft process, which solubilizes biomass in harsh chemicals [3], fungal CAZymes can break down biomass more efficiently, enabling the creation of innovative products from these complex macromolecules [4]. Further, the resulting composites of fungal hyphae and solid biomass will be studied on their capacity to produce ‘mycomaterials’ to replace unsustainable conventional materials, or to be used as a feedstock for further biorefinery processing.

This study aims to assess how different lignocellulosic feedstock and pretreatment affect fungal growth and enzyme secretion. The performance of these enzyme extracts will be evaluated on relevant biomasses and mycomaterial generated will be explored as source for a biorefinery scheme. Finally, a lifecycle analysis will compare the sustainability of the approach compared to the existing technology.

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