CROSSLINKED LIGNOSULFONATE AS BIOBASED MATERIAL OF THE FUTURE
A CARBAMATE LINKED POLYURETHANE LIKE POLYMERIZATION OF PURIFIED LIGNOSULFONATE WITH INTERESTING PROPERTIES

Although huge amounts of lignin derivatives (Kraft lignin and lignosulfonates) arise as a by-product from the pulp and paper industry, lignosulfonates are rarely used as a precursor for high-value products. This fact is a result of the heterogeneous and complex molecular structure of lignosulfonate, which makes it hard to modify, purify, and characterize.

In the course of project Lenz 1.5, a strategic project between cooperation partners Wood K plus and Lenzing AG, a purification procedure was established to isolate lignosulfonate from sulfite spent liquor. After adsorption onto an acrylic resin, washing off undesired compounds, and desorption, lignosulfonic acid in a reasonable purity is available to utilize it in modification reactions.

First polyurethane bond of lignosulfonate: A polyurethane synthesis was developed starting from lignosulfonate. Based on the large amount of hydroxyl groups in lignin, the lignosulfonate was reacted with glycerol carbonate to further increase the hydroxyl group content. These were then reacted with dimethyl carbonate to give cyclic carbonate functionalities, which upon reaction with hexamethylenediamine yield the polyurethane product. Interestingly, the synthesis of lignosulfonate cyclic carbonates as well as its amination hadn’t been reported before.
SUCCESS STORY

Nevertheless, the project team unambiguously proofed its formation by IR and solid state NMR spectroscopy, which was also used to verify the carbamate bonds in the lignosulfonate polyurethane.

Furthermore, a lignin model compound was subjected to the same polyurethane synthesis sequence. With the help of the very simple molecule, the products and by-products of each reaction could be separated and fully characterized. The obtained information is required to understand the reaction mechanisms of the model compound and allowed the researchers to interpret and optimize the lignosulfonate’s reactions.

Impact and effects

In established polyurethane producing processes highly toxic substances (e.g. disocyanate) are utilized, while known alternative procedures require expensive catalysts or explosive epoxide compounds. By contrast, the developed polyurethane synthesis is environmentally friendly, applies harmless reagents and is cheap, because glycerol carbonate can be produced from glycerol and \( \text{CO}_2 \) or urea, and dimethyl carbonate is commonly used as a solvent in industry.

The synthesis and characterization of lignosulfonate based polyurethanes is already prepared for publication and was submitted to an internationally acknowledged journal. Now the structure and associated material properties of the polyurethanes need to be studied in detail, to develop products with competitive physicochemical properties.

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©Mimini Vebi, Crosslinked lignosulfonate in a petri dish and 10x magnified particle

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Project coordination (Story)
Dr. Ireen Gebauer
Project Leader
Wood K plus
T +43 (0)7672 701 3181
i.gebauer@wood-kplus.at

Wood K plus
Kompetenzzentrum Holz GmbH
Altenbergerstraße 69
4040 Linz
T +43 (0) 732 2468 – 6750
zentrale@wood-kplus.at
www.wood-kplus.at

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Project partner

- Lenzing AG, Austria
- University of Natural Resources and Life Science, Austria

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Cellulose is the most abundant biopolymer in the world. As a renewable resource about a third of all plant material is cellulose and it is used in every area of our lives. A broad range of cellulosic fiber sources may be found in nature. The most common raw materials are cotton, wood, flax and cannabis. Today cotton is the most important natural fiber used in the textile industry worldwide. It is grown in countries with a warm climate and limited water supply. A draw-back is that cultivation of cotton requires large water consumption and an excessive use of pesticides, leading to massive environmental problems. Therefore, rayon (i.e. viscose) made from wood is a good alternative to cotton fiber. Especially viscose fibers leave a much lower water and carbon footprint compared to cotton.

For the production of rayon (Fig. 1), cellulose is treated with sodium hydroxide resulting in alkali cellulose formation. The alkali cellulose is then treated with carbon disulfide to form cellulose xanthate, which is subsequently dissolved in dilute sodium hydroxide. This solution is called viscose (see Fig). Finally the viscose solution is pumped through a spinneret into a dilute sulfuric acid bath, where cellulose is regenerated.
The number of xanthate groups (γ-value) and their distribution influence several properties of the viscose such as solubility, viscosity, filterability and spinnability. A new NMR method for determination of the γ-value and of the distribution of xanthate groups was developed.

Due to its not stable structure, viscose is not easy to analyze and not durable. The new method allows not only the analysis the viscose samples but also the comparison of viscose samples from different viscose factories.

Impact and effects

A new NMR method for the determination the number of xanthate groups and their distribution in viscoses works on real-world samples and that it is possible to measure the γ-value and substituent distributions at different time intervals during viscose process. This method allows the comparison of viscose samples from different viscose factories.

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**Project coordination (Story)**

Dr. Katja Wöss
Project Leader
Wood K plus

T +43 (0) 732 2468 5672
k.woess@wood-kplus.at

**Wood K plus**
Kompetenzzentrum Holz GmbH
Altenbergerstraße 69
4040 Linz
T +43 (0) 732 2468 – 6750
zentrale@wood-kplus.at
www.wood-kplus.at

**Project partner**

- OMV Refining & Marketing GmbH, Austria
- Lenzing AG, Austria
- Metadynea Austria GmbH, Austria
- Sandoz GmbH, Austria
- DPx Fine Chemicals Austria GmbH & Co KG, Austria

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SUCCESS STORY

Wood K plus
WOOD: next next generation materials and processes - from fundamentals to implementations

Programme: COMET – Competence Centers for Excellent Technologies

Programme line: COMET-Centre (K1)

Type of project: Functional surfaces, 01/2015 – 12/2018, multi-firm

BRIGHT PROSPECTS DUE TO NEW MULTI LAYER COATINGSYSTEM

THE RESEARCH RESULTS ARE BASIS FOR A 13 MIO EURO INVESTMENT AND 25 NEW JOBS

The furniture industry is always subject to trends. In the last few years, the tendency for high-gloss and high-quality surfaces has been manifested. To meet these needs, various technologies have been used in the past. These methods offer different advantages and disadvantages – but in detail they are not able to combine a perfect surface appearance with high scratch resistance or to be appropriately inexpensive. To fit in with these customer requirements, wood-based panel manufacturers have to offer new solutions.

With the results presented here, it has been possible to combine all the important criteria as “functionality of the surface”. A perfect gloss effect and high surface resistance have been linked with easy processing and the possibility of easy-to-implement color combinations with, for example existing melamine surfaces.

The most important parameter of the project success was the achievement of the highest possible “depth effect” of the gloss combined with associated smooth surface where both, the carrier material used and the layer structure of the coating, played an essential role.

Since wood-based materials tend to age due to changes in environmental conditions (temperature, humidity), this phenomenon was already taken into account during the development work. Furthermore, the necessity of finding a balance between the hardness and the flexibility of the coating material was an important step.
SUCCESS STORY

Impact and effects

Based on the research results, the company partner made an investment of around 13 million Euros. With the implementation of the new technology, production capacity in the field of coated wood-based panels will take place and increase by around 40% from 10,000 million m$^2$ to 14,000 million m$^2$. With this development it is expected to generate sales of around 60 million Euros per year, with an export share of around 90%. Linked to this is a strengthening of the production site, whereby 25 new jobs will be generated in the area of the new production plant in the final expansion.

Project coordination (Story)
DI Dr. Edith Zikulnig-Rusch
Area Manager
Wood K plus
T +43 (0) 4212 494 8017
e.zikulnig-rusch@wood-kplus.at

Wood K plus
Kompetenzzentrum Holz GmbH
Altenbergerstraße 69
4040 Linz
T +43 (0) 732 2468 – 6750
zentrale@wood-kplus.at
www.wood-kplus.at

Project partner
• FunderMax GmbH, Austria
• Reutlingen University, Germany
• Johannes Kepler University Linz, Austria

Diese Success Story wurde von der Zentrumsleitung/ der Konsortialführung und den genannten Projektpartnern zur Veröffentlichung auf der FFG Website freigegeben. Weitere Informationen zu COMET: www.ffg.at/comet
Knowledge on the interface between wood and adhesive is of fundamental importance to improve binders, processes and composites. “Understanding new interfaces”, a project of common interest to our partners, aimed at an increased fundamental understanding of surface properties and their interaction with adhesives. Therefore, this project was organized as “strategic dissertation”. Such dissertation was largely independent of short-term company interests and characterized by high independence, an open right to publish, and an especially deep integration with academics.

The performance of adhesives depends on its cohesion as well as its adhesion to the wood surface. These surfaces are, however, inhomogeneous and complex. Wood mainly consists of parallel hollow tubes, the wood cells. The cell walls are built up of a layered structure, where each layer differs in structure and chemical composition. Adhesive may bond to the compound middle lamella (“the outside of the tube”), a cut surface across the cell wall (mainly the S2-layer), or the inner cavity termed lumen (“the inside of the tube”).

In order to compare the strength of an adhesive to all possible surfaces, an elaborative method was needed. Single wood fibres were isolated, straightened, frozen onto an ice cube, cut along its longitudinal axis, embedded into adhesive, and finally cut perpendicular to the fibre axis. Thus, samples of half wood cells with adhesive contact to all three surfaces were obtained. These samples were then investigated by nanoindentation: a very
small cone diamond tip was pressed into the interface. The energy spent is related to the adhesive strength. For example, no differences in adhesion were found at the interfaces of the different surfaces and MUF adhesive. MUF penetrated into the cell wall leading to a strengthening superimposing adhesion differences.

Furthermore, influences of technical processes were investigated. MDF fibres were produced in a thermomechanical refining process, leading to higher content of relatively apolar lignin at the outside fibre surface. These fibres were halved and embedded in adhesive as well, followed by nanoindentation. A trend indicating higher adhesive strength between less polar PUR adhesive and the less polar compound middle layer was observed. Nanoindentation was also performed at the interface of PUR with the relatively polar surface of cellulosic fibres now leading to a lower adhesive strength.

Impact and effects

In conclusion, the adhesive strength can be steered by adapting the polarity of the wood fibre. This knowledge was also implemented. Cellulose nanofibers containing residual lignin were obtained by mechanical fibrillation and were used to stabilize aqueous styrene emulsions. Upon polymerization of the styrene, polystyrene microspheres coated in fibres were obtained. Also polyester microspheres were obtained this way.

The work of the Ph.D. student, who also completed an outgoing research stay at the University of Maine (USA) for 4 months, was published in six publications. The impact of this fundamental work is illustrated by the fact that these have been already cited several times in only a short period of time.

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Project coordination (Story)
Dr. Erik van Herwijnen
Team Leader advanced bonding
Wood K plus
T +43 (0)1 4277 89137
e.herwijnen@wood-kplus.at

Wood K plus
Kompetenzzentrum Holz GmbH
Altenbergerstraße 69
4040 Linz
T +43 (0) 732 2468 – 6750
zentrale@wood-kplus.at
www.wood-kplus.at

Project partner
- University of Natural Resources and Life Science, Austria
- Egger, Austria
- Henkel, Switzerland
- Johns Manville, USA
- Metadynea, Austria

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SUCCESS STORY

INDUSTRY 4.0: POTENTIAL FOR THE DIGITIZATION OF THE AUSTRIAN SAWMILL INDUSTRY

SURVEY OF SMALL AND MEDIUM Sized SAWMILLS SHOWS THAT THE FIRST LEVEL OF DIGITISATION IS ONLY PARTIALLY REACHED

Industry 4.0 refers to the integration of Information and Communication Technologies (ICT) into production systems, which can optimize production flows. However, as a first step, the application of ICT requires the digitization of processes. This project examined whether small and medium-sized enterprises of the Austrian sawmill industry fulfil the requirements for the Industry 4.0 (“Internet of Things”). Thereby a better understanding of the need for action for sawmill enterprises, but also for associations and political institutions to ensure the competitiveness of the Austrian timber industry, was achieved.

Digitization criteria: three levels

The state of digitization was assessed by means of a survey based on digitization criteria. The questions dealt with the existence of basic data processing (first stage of digitization), digitally networked information and communication (second stage of digitization) and digitally networked products and services (third stage of digitization).

By cluster analysis, the 87 surveyed small and medium-sized sawmills are divided in three groups. The first group (n= 35), the “manual small business”, did not reach the first stage of digitization: computer supported production programs (0%) are not
SUCCESS STORY

available. The second group (n=26), the "the automated business", has partially reached the first stage of digitization. Half of the companies use production programs (55%), but only a quarter of the enterprises have their own server (27%). The third group (n=26), the "solid developed business" group, has reached the first stage of digitization as the basic hardware and software is available. However, there is a lack of further software equipment and cross-company strategy for digitization, to reach the second level of digitization.

Information, financing models and IT-Consulting recommended.

Only one group of enterprises reached the first level of digitisation. The remaining companies did not reach the first stage of digitisation or reached it only partially. This shows that there is great potential for the digitisation of the Austrian sawmill industry. In order to foster digitisation, the provision of information and financing models as well as the support of external IT experts is recommended.

©Wood K plus, Achieved digitization criteria by identified group

Project coordination (Story)
Dr. Franziska Hesser
Team Leader
Wood K plus
T +43 (0) 1 47 654 – 73518
f.hesser@wood-kplus.at

Wood K plus
Kompetenzzentrum Holz GmbH
Altenbergerstraße 69
4040 Linz
T +43 (0) 732 2468 – 6750
zentrale@wood-kplus.at
www.wood-kplus.at

Project partner
- All COMET partner companies

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