Doktoratsinitiative Holz – Mehrwertstoff mit Zukunft

# DokIn'Holz

## *Zusammenfassung*
## *Wissenschaftlicher Endbericht*

1. Jänner 2014 – 30. April 2017

## Doktoratsinitiative *Holz – Mehrwertstoff mit Zukunft*

des

Bundesministeriums für Wissenschaft, Forschung und Wirtschaft

gemeinsam mit der

Kooperationsplattform Forst Holz Papier

Bundesministerium für
Wissenschaft, Forschung und Wirtschaft

FORST HOLZ PAPIER

## F1: Innovative Laubholztechnologien und Produkte

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The climate change and its consequences such as sustainable and ecologically sound forestry are leading to an increasing percentage of hardwoods in many regions in Europe. This puts pressure on an enhanced hardwood processing and utilization in order to utilize hardwood resources in a proper and economically viable way. A critical look at the current hardwood material utilisation reveals a gap between the growing stock and proper material utilisation. In contrast to the increasing forest stocks, the production and utilisation of hardwood lumber is decreasing. Historical data from 1992 to 2012, provided by FAO, shows an exemplary utilisation collapse of 30 – 50 % for Germany, Austria or Switzerland (FAOSTAT). On the other hand, the industrial and energetic utilisation of hardwood has developed progressively.

A reason for the drop in hardwood lumber manufacturing is seen in saturated market of solid hardwood products and the very slow product changes in comparison to the substitution of products by competitive materials in the various sectors of hardwood utilization. Traditional hardwood firm structures and processing technologies have developed in an evolutionary way over a long-time period. We assume that there is a significant optimisation potential in a re-design of the current hardwood processing and the hardwood process supply chains by introducing new solid hardwood production concepts focusing on roundwood disintegration processes (e.g. sawing) and the divergent material flows.

Therefore, the overall question was about the future production processes, production facility locations as well as the specific hardwood products.

With this background, the central question that motivated this study was: Are there different manufacturing concepts and strategies for the solid hardwood industry considering existing products, processes and technologies?

In this context, the report provides information about a more transparent process chain from sawmills via downstream companies to the final product and it supports an inter-firm consideration of alternative production approaches of selected products as well as alternative technologies by (i) literature research, (ii) hardwood product as well as process chain analysis and (iii) hardwood glued laminated timber facility-location allocation model in a network design.

After a thorough literature research, the second part of the work represents a conceptual framework of a methodology to analyse hardwood products and process chains on their individual characteristics by starting with the product. It is not an individual company-related solution for hardwood products thus it can be used from company managers and decision makers as a decision support tool. Relating to different aspects of production processes, in our case product, product components, manufacturing processes and technologies (Figure 1), a high amount of information is structured and provided for strategy decisions.

This second part of the work represents a methodology to redesign supply chain production processes. Quality Function Deployment and Process-Technology Matrix enable to make statements about the separation of the supply chain in different company units with key competences and about cooperation within the supply chain. Further, the user can elaborate an extended diversification strategy on vertical expansion.

The main limitation is based on the theoretical results of the case study although the underlying information is based on standards and existing scientific papers.

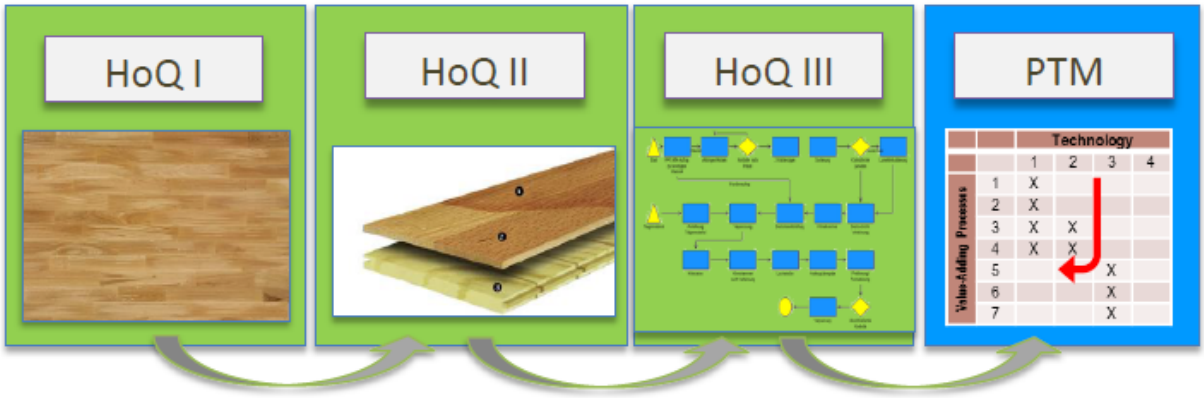


**Figure 1:** Multi-step Quality Function Deployment (QFD) and the Process-Technology Matrix are combined as a systematical methodology.

The methodology and the case study show that hardwood downstream firms should consider mainly the characteristic properties of hardwood. Due to biological, mechanical and physical reactions, the individual hardwood properties also influence product and component design as well as process chains. For future hardwood application in the field of construction, these requirements should be integrated in the product and process chain design. This does not necessarily result in the development of further technologies but it can reveal optimisation potential of current products, product components, existing processes and corresponding technologies.

Further, the study shows that alternative hardwood manufacturing approaches exist, which can be identified with this cross-company methodology.

The third part of the work consists of a mathematical optimisation model from the tool box of operational research. Because different material based topics were already covered, we decided to go further and think about the hardwood timber transformation process in a supply network system. This means to use the existing information to get answers about facility locations, material flows and capacities within Austria as a case study. We used mixed linear programming for setting up downstream facilities in a production network in order to achieve minimal network cost. The model consists of forests, sawmills, downstream facilities and customers with a specific demand (Figure 2).

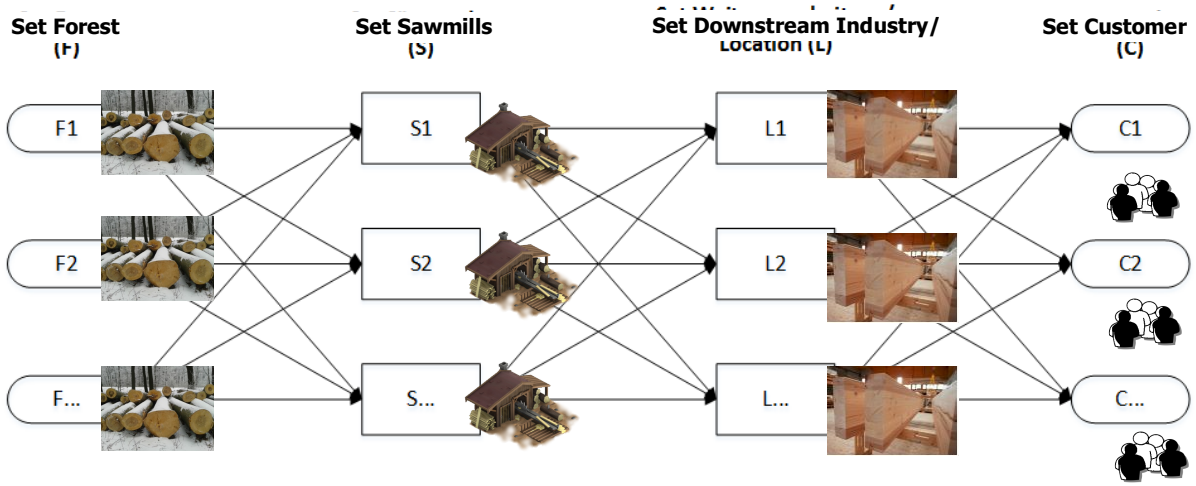


**Figure 2:** The network structure of the facility location allocation model in a network design is represented. For this model a set of 88 forests nodes, 26 sawmills, 16 locations and 88 demand zones/customer are used.

The results are the various material flows which were generated from different scenarios surveyed. The

comprehensive description of the optimization model and the results are described in the upcoming paper. This model can also be extended to other or combined primary processing solutions such as sawmilling and veneer peeling.

# F2: Sicherung der Rohstoffversorgung: Produktionsplanung und Ertragsregelung unter Berücksichtigung von Risiko und Ungewissheit

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Long-term production planning and yield regulation are the core elements of forest management planning at the strategic level. The former determines production targets and silvicultural programs, whereas the latter strives for a sustainable trade-off between the level and structure of the growing stock on the one hand and the cuttings on the other. The approaches and tools applied in practice mostly refer to concepts which ignore any risk and uncertainty irrespective of the fact, that risks of production and market risks are of great significance, the long time horizon of timber production implies a high degree of uncertainty and several tools for addressing these issued explicitly are documented in the scientific literature. The project's results shall help bridging the gap between the respective state of the art in science and practice.

The general guidelines for risk management postulate, that effectiveness and efficiency of respective approaches hinge on a comprehensive as well as balanced concept. Consequently, a framework for risk management in forest enterprises has been developed, thereby taking into account the peculiarities of forest enterprises in general and timber production in special and highlighting the role of management planning within the overall system. Figure 1 illustrates this concept. A checklist is provided to assist the assessment of the individual conditions as well as the requirements for designing the risk management process in practice. The questions specifically address the different decision making levels making up a forest enterprise but also the owner. Accordingly, the checklist is structured to overall questions and questions related to the individual hierarchic decision making level. Thus, a context for comprehensive risk management is provided which is required for the evaluation of specific tools for strategic planning in forest enterprises.

Once the general framework for company-specific risk management (RM) is established, one may proceed to identify the appropriate tools for production planning and harvest scheduling. The decision, which of them is suitable and useful for a practical implementation is to be made individually and has to take into account the specific operational conditions. In order to support this process a two-step-approach for the assessment of planning devices has been designed. It consists of two sets of test questions referring to a total of ten criteria. One catalogue serves for the general characterization of any such tool. This general characterization is intended to serve as a starting point and reference for an individual assessment at enterprise level. The scheme for the user specific evaluation systematically confronts the profile of the tool with the requirements and frame conditions for practical application in the specific case.

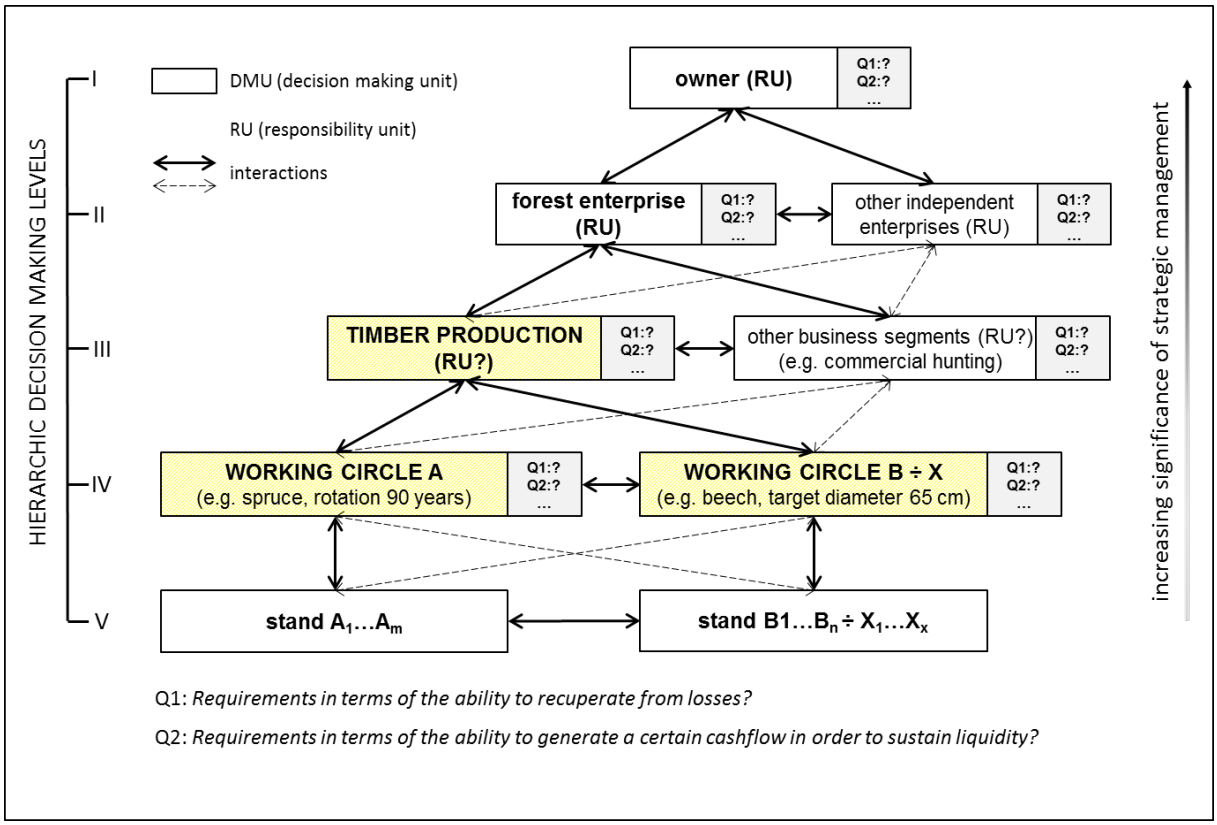


**Figure 1:** Framework of a comprehensive forestry specific system of risk-management (the focus of subsequent research is highlighted in yellow)

The evaluation scheme has been applied to five models which might be of interest for introducing explicit RM into strategic planning issues of timber production. The results indicate that the transdisciplinary approach is quite suitable but has to be developed further in order to effectively bridge the gap between theory and practice in this field.

An e-learning course called *'Risikomanagement im Rahmen von Ertragsregelung und Produktionsplanung im Forstbetrieb'* has been established in order to make the material and tools which emerged from this project available to all stakeholders (https://learn.boku.ac.at/course/view.php?id=11288 guest access with password 'risk'). A range of activities devoted to highlighting the topic and to the dissemination of the project's results shall foster an ongoing discourse about RM in forest management planning and serve as a basis for specific transdisciplinary activities.

# H1: Numerische 3D-Modelle zur Ermittlung von Biegefestigkeiten, Festigkeiten und stochastischer Informationen von Brettschichtholz und Brettsperrholz unter Berücksichtigung von Material- und Strukturnichtlinearitäten


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This work aims at a new numerical approach to understand and estimate failure mechanisms and strengths of wood and wood-based products. Since failure initiation and crack formation are strongly influenced by the complex material system of wood, exhibiting cellular and layered structures on different length scales, a mechanical concept, in which these different microstructural characteristics are incorporated, appears to be necessary. Introducing the division of wood into meaningful scales of observation, failure modes and failure stress states (strength properties) are to be determined at each scale by means of numerical methods, and the obtained information is to be transferred - and will serve as input - to the next higher scale of observation.

For this so-called upscaling, at the Institute for Mechanics of Materials and Structures, a numerical concept based on the extended finite element method is currently in development, which is able to describe failure (even cracking mechanisms) of wood very accurate. For a comprehensive description of the strength behavior over several levels of observation, however, this method alone seems to be insufficient and inefficient. For this reason, within this work, numerical limit analysis approaches are developed and applied for the first time to wood, complementing the overall multiscale 'damage' concept successfully. This method exclusively focuses on the time instant of failure, and delivers a lower bound and an upper bound for the ultimate strength of the considered material structure. Compared to conventional finite element approaches, where the complete load history has to be considered and, in order to predict the correct failure mechanisms, proper regularization techniques must be used, limit analysis approaches are much more stable and efficient.

The first application is applying the numerical limit analysis approaches for the estimation of effective failure surface and failure modes of wood at the annual ring scale, in which, the honeycomb like structure can be observed in the $R-T$ plane. For different cell structures for earlywood and latewood, two unit cell models are introduced. Due to the highly repetitive structural characteristics, periodic boundary conditions are enforced. The Tsai Wu anisotropic yield function describes failure of the cell wall layer and the von Mises isotropic yield function is applied to the middle lamella. Applying various loading states in numerical limit analysis calculations, lower bound and upper bound failure surfaces are obtained for both unit cell models, and different failure modes are identified for different effective stress states. Similar failure mechanisms and effective failure surfaces as delivered by the extended finite element method were obtained, although the numerical limit analysis calculations exhibit signif- icant advantage regarding computational efficiency. Unfortunately, at this scale no experimental data was available for validation of these numerical approaches.

Thus, in a next step, the numerical approaches were applied to the next higher scale of observation, the clear wood scale. At this scale, the material is modeled by a layered structure with earlywood and latewood layers in the $L-T$ plane. The Tsai Wu anisotropic yield function is assigned to

both layers with the strength parameters for the *T* -direction obtained from numerical results at the annual ring scale, and the parameters for the *L*-direction as well as in-plane shear from literature. Applying a similar procedure as at the previous scale, failure surfaces were obtained for different material principal orientations. The numerical results were compared to experimental data from biaxial loading tests on Norway spruce clear wood. The effective failure surfaces obtained from the limit analysis approaches enclose the experimental results almost perfectly, and thus, represent a good estimate for the effective strength of clear wood. In contrast, computational results from a continuum micromechanical model based on the shear strength of lignin, provide an inner envelop to the experimental results. In combination these two methods provide comprehensive information about the failure behaviour of clear wood in an computationally very efficient way.

In order to assess the potential of the numerical limit analysis approaches for strength predictions of wood-based products used in timber engineering, the approaches were applied to bending tests on cross-laminated timber plates with concentrated loading. Taking experimental results from the Institute for Mechanics of Materials and Structures as reference, in which correlations between load bearing capacity of cross-laminated timber plates and wooden boards are studied, geometry of plates, boundary conditions and loading conditions are defined accordingly, and material strength parameters are employed from Eurocode 5 in order to be consistent with the experimental work. For the three different strength classes of wooden boards, C18, C24, and C35, used in the experimental program, the effective failure loads from numerical approaches are discussed, which agree very well with the experimental bending test results. It can be concluded that, the numerical limit analysis approach is able to provide good estimates on effective strengths of cross-laminated plates. Due to the high time efficiency, the implemented approaches provide possibilities to study failure mechanisms and the influence of raw material on the final products by means of comprehensive numerical calculations.

Finally, the upper bound formulations were extended by velocity discontinuities, which enable velocity jumps between finite elements as additional degree of freedom. It has been proven using numerical examples that, the velocity discontinuities are very powerful in capturing very localised failure. If discontinuity interfaces are properly defined, good upper bound results can be obtained even with very coarse meshes. Thus, the computational time efficiency can be further increased significantly. In timber engineering, failure in wood-based products and structures is often localised,

e.g. shear failure around ply bounding, and for this reason, this implementation has great application potential.

In conclusion, numerical limit analysis is a powerful tool estimating structural strengths based on ductile failure. Although failure of wood is often characterised by brittle cracking, especially wood- based products exhibit the ability to utilise their plastic potential, and thus, limit analysis deliver usable strength estimates very efficiently. The capability of this method in providing good quality of strength estimations with high computational time efficiency has been discussed in different scales of observations.

## H2: Das akustische Verhalten von Wand- und Deckenverbindungen im Massivholzbau

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In many cases, commonly used prediction methods for calculations of the sound insulation between adjacent rooms are of limited applicability for timber constructions. This observation is caused by the fact that relevant measurement and calculation standards are based on homogenous, isotropic materials like concrete. For timber constructions a different acoustic behaviour is expected, especially, in the junctions of the involved components. Therefore, the competitiveness of the material timber compared to other building materials is significant decreased.

During the PhD-project “The acoustic behaviour of junctions of walls and ceilings of cross-laminated-timber”, research concerning the dynamic processes in the junctions has been conducted to get a deep understanding of these problems for future solutions. Essential physical effects have been identified and their influence on the acoustic energy transmission between adjacent components was studied. Proposals for additional calculation methods have been developed. Based on these findings, general designs of timber junctions may be improved in future. As a consequence, the deep knowledge of constructions using panels of cross-laminated-timber may be increased. Using such alternative prediction methods, the need for expensive measurement procedures can be reduced which allows more efficient developments of innovative new products.

Computer based calculations using approaches like the finite element method and the statistical energy analysis allow comprehensive analyses of the structure-borne sound transmission. The calculations allow an insight into the acoustic energy distribution within a component and a determination of the energy flow from one component to another one. Using experimental data of equivalent test setups, values of connection stiffness of common junctions are identified and implemented in the calculation models. In terms of practical aims this allows a calculation of the resulting acoustic energy transmission (see figure 1). The resulting values are integrated in standardized prediction methods for calculations of the total sound transmission, e.g., in multi-storey dwellings (see figure 2).

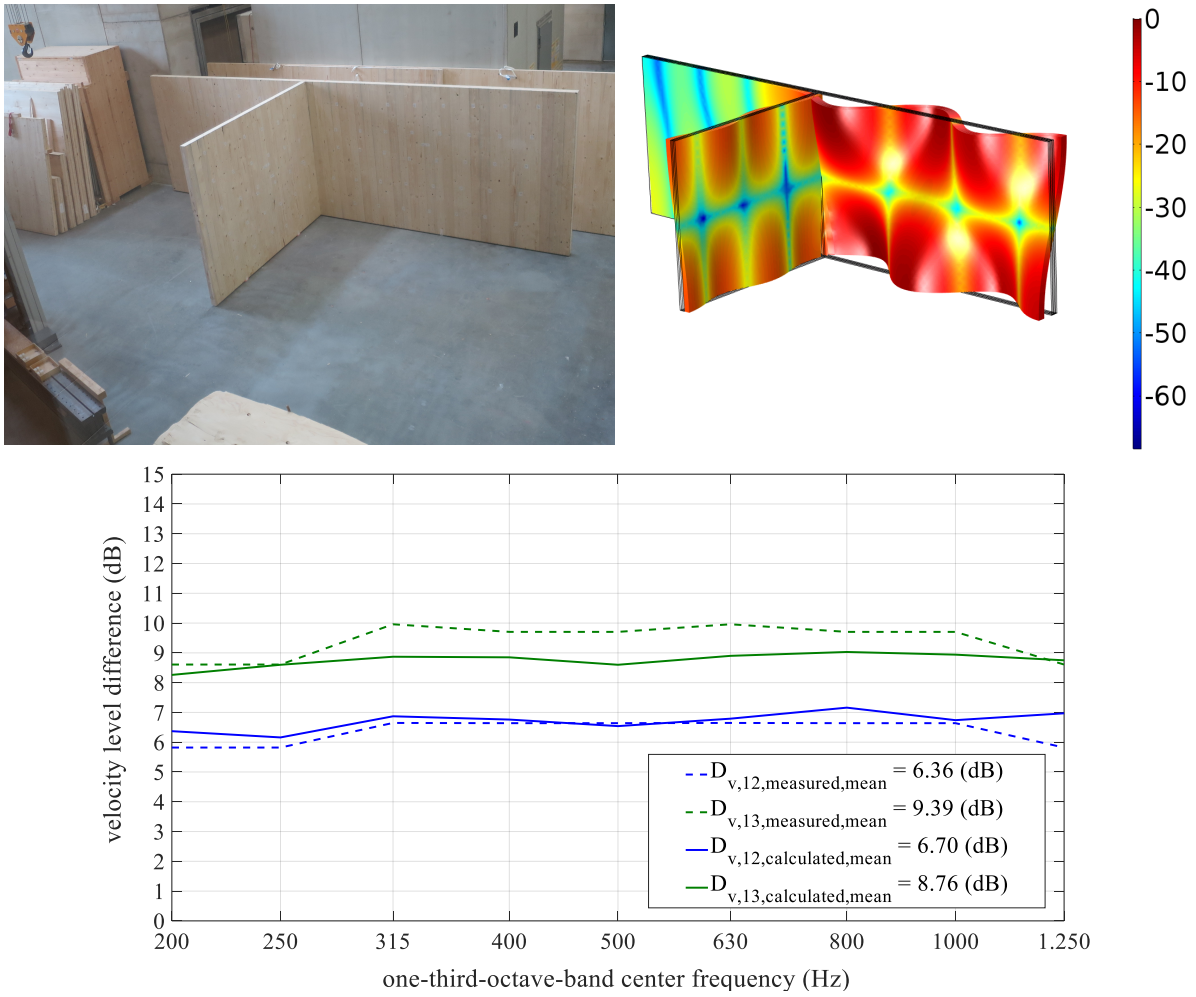


**Figure 1** – Calculation of the structure-borne sound transmission through a T-junction (upper left) using the Finite Element Method (upper right); Comparison of measurement and calculation results (lower graph)

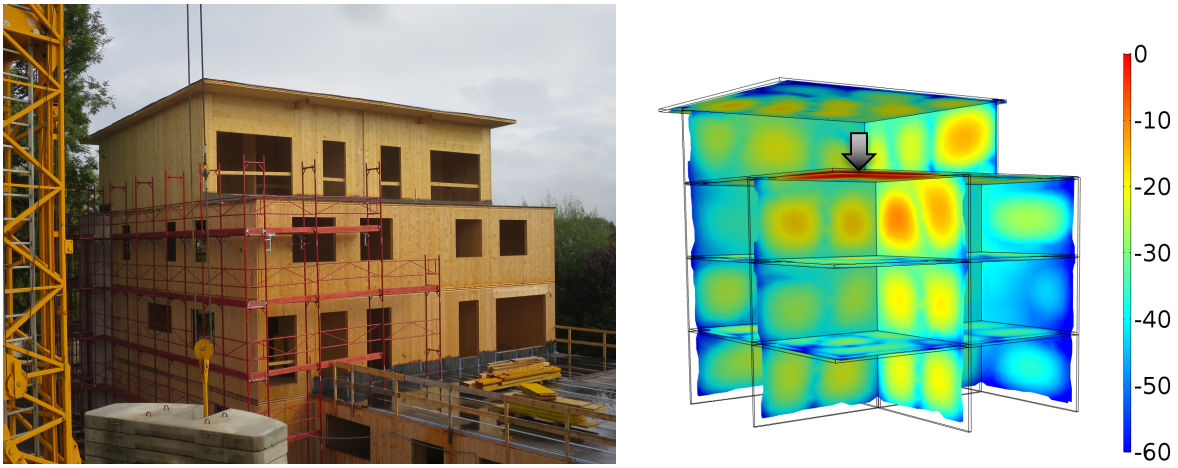


**Figure 2** – Building shell of a multi-storey dwelling (left); finite-element calculation of the principle of structure-borne sound transmission in the building caused by impact noise at 200 Hz (right)

## H3: Grundlagen für einen neuen mechanischen Holzaufschluss als Basis für verbesserte bzw. neue Holzwerkstoffe und Engineered Wood-Products

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Round wood is used in huge quantities and is typically fragmented by tradition based technologies like sawing, peeling, hammer milling, refining and others in a first process step. A compulsory need of radical innovation was identified in order to amend current fragmentation technologies. Existing ones are not able to maintain the mechanical potential of the material. A couple of these technologies exhibit insufficient yield of high quality material. We hypothesize that it is possible to fractionize wood in a different and also more efficient way than the existing ones most notably by non-cutting based technologies maintaining the natural optimized structure of wood. Wood composites produced out of these optimized particles are assumed to have improved mechanical properties. In a pre-project alternative disintegration of wood, several technologies were identified by a cross innovation patent research. In total approx. 26.000 patents were screened and 250 were selected for further investigation. From this initial number, 50 patents were evaluated during a workshop with industrial and scientific partners due to their high potential. At the end six technologies were identified and selected by the industry partners as the main topics for the DokIn´Holz programme. Selected disintegration technologies were as follows: i) laser cutting technology, ii) wedge cutting supported by laser, iii) waterjet and wedge cutting supported by waterjet, iv) d) squeezing technology, v) chipless cutting technologies (i.e. slicing), vi) disintegration and debarking by means of shockwaves.

During the first part of the project a detailed literature research of all these alternative cutting technologies was performed. Specific questions arising from the literature research were answered by performing expert interviews. Detailed results of the different disintegration technologies were compiled to a presentation and short report including a SWOT analysis. The results of the literature research and SWOT analysis were discussed with the industry partners involved. Following technologies were selected for further experimental work: i) squeezing as a mechanical pre-treatment of chips for the refiner process, ii) shockwave technologies and iii) waterjet for debarking. Furthermore, it was decided to summarize all information of laser cutting technology in a review article.

Further investigations of squeezing and shockwave pointed out, that shockwave will cause similar effects than the dynamic impact of a solid body into the wood material. Due to the viscoelastic properties of wood high differences between static and dynamic insertion of a solid body is expected. Systematic test runs were performed to investigate the effect of total energy absorption and the speed of the solid body smashing the wood material. Results of these first trials directed to the idea to use dynamic smashing as a pre-treatment of chips before refining, in order to reduce the energy consumption in the refiner process.

Figure 1 and 2 show industrial chips and chips after smashing as well as SEM micrographs of both kinds of chips, proving that dynamic smashing yield a high number of micro-cracks parallel to the fiber direction. Hypothetically according to other authors micro-cracks should reduce the energy consumption during refining compared to unaffected chips.

**Fig 1:** Reference industrial chip and internal structure.

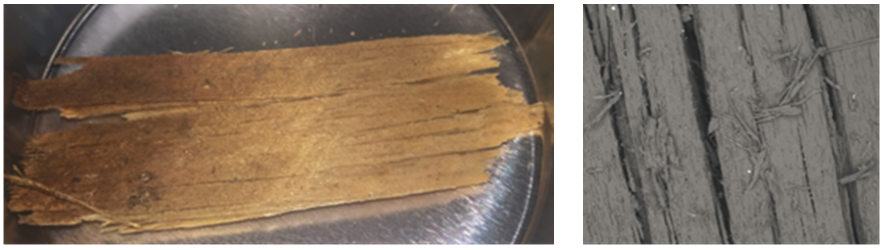

**Fig 2:** Smashed industrial chip and internal structure.

For waterjet debarking experiments with changing water pressure were performed. Waterjet was adjusted in different directions and angles to the surface of small logs. Additionally water and energy consumption, as well as possible feed speeds were evaluated. Results pointed out that waterjet debarking can be applied only on thick logs, but this method is not efficient from an economic point of view for small diameter round wood.

In the review article laser cutting technology was compared to circular saw, to point out the advantages and disadvantages, as well as limitations of laser cutting.

The main conclusion of the project is that there is no alternative cutting technology available, comparable to the circular saw. Meaning that circular saw is the most suitable tool for produce timber with defined dimension. However, optimization of strand, particle and fiber production can be still assumed.

## H4: Innovative Laubholzverwendung

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The inherent mechanical properties of some hardwood species indicate a high potential for applications in the building sector. In this context the major challenges for a wider and more effective and sustainable use of hardwood are to find (i) a competitive product and (ii) adapted connection techniques. Current industrial processes using wood as raw-material are mainly focusing on the production of prismatic panel or bar-shaped elements with rectangular cross-sections. Another approach, which may lead to a new product category, competing currently used steel- and aluminium-elements, is to form thin sheets of wood (veneers) with interposed adhesive to create light-weight components with optimized cross-sectional shape, called **U**ltra **H**igh **P**erformance **P**lywood - UHPP (Figure 1).

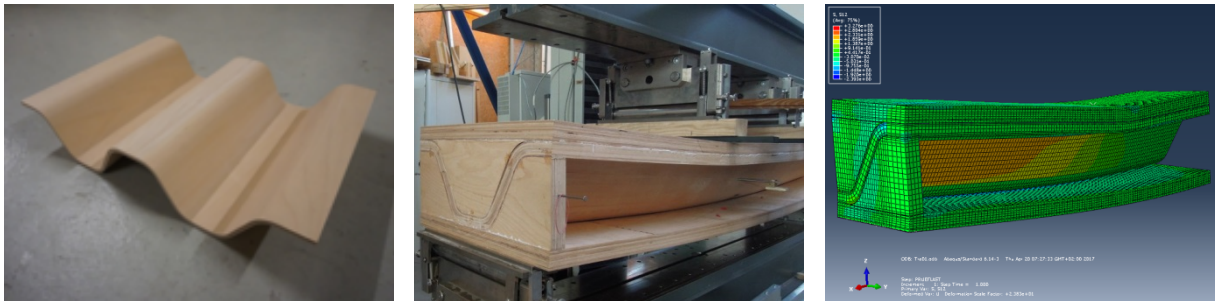

**Figure 1:** Two-waved trapezoid (left), 4-point-bending test (mid), parametric FE-model (right)

Therefore the emphasis of my research in this field is on providing knowledge for forming methods and identifying main aspects with influence on the properties and the load-bearing behavior of a final product. Investigations on forming techniques have shown that in particular, beech and birch veneers are suitable for the mentioned production processes. The required material properties of the raw material for theoretical investigations on structural components produced with a matched-die forming technique were determined and implemented in a parametric finite-element model. The model is currently validated by experimental tests and will enable to determine the effects of different layer-setups and cross-section designs on the load-bearing behavior and to define an optimal design for a structural application (Figure 1, right).

The design and load bearing capacity of structures made of hardwood are strongly related to the performance of applied component connections, which are often limiting the minimum cross-section dimensions. In the frame of the project two types of fasteners as components of end-grain joints were investigated: (i) self-taping screws and (ii) glued-in plates. The study on self-taping screws focused on the determination of the value of efficiency (ratio of the load-bearing capacity referred to the load-bearing capacity of the gross-section of the component) of an end-grain joint for a hardwood component loaded in tension. The results indicated that the current morphology of screws traditionally used for softwood connections has to be adjusted for hardwood applications, which is currently investigated in an ongoing project.

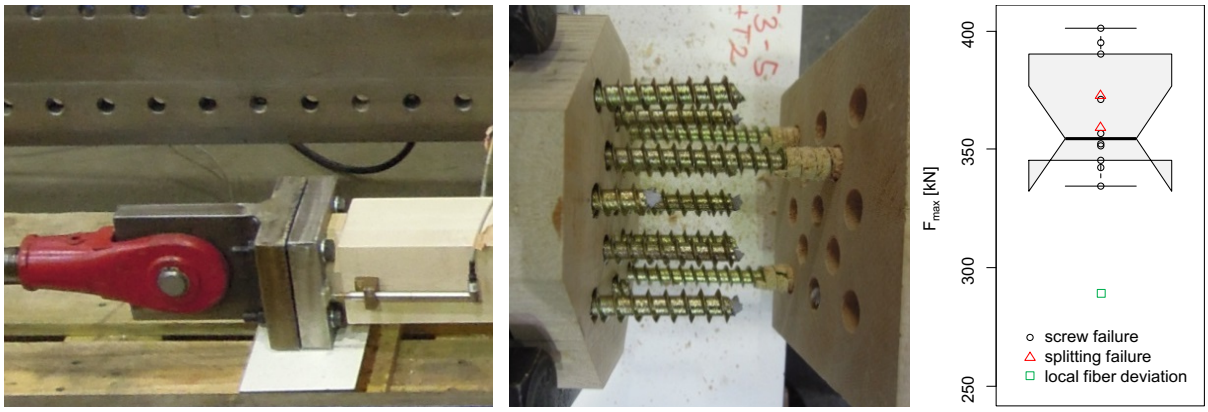


**Figure 2:** Test configuration (left), failure (mid) and extract of results (right)

Taking the promising approach of creating light-weight components as shown in Figure 1 into account, a major challenge is to find an adequate connection technique for such products. One of the possible solutions is glued-in plates, which can be implemented in the narrow faces of an element. In order to evaluate the suitability of glued-in plates, experimental tests were carried out. The pull-out strength and the load bearing behavior of perforated and unperforated plates considering parameters such as (i) the type of adhesive, (ii) the surface treatment were determined. It was found that the short term pull-out strength of adhesively bonded end-grain joints made of glued-in plates can be significantly improved by the type of applied adhesive, the joint geometry (in particular related to used perforation), the surface treatment and the reinforcement (Figure 3).

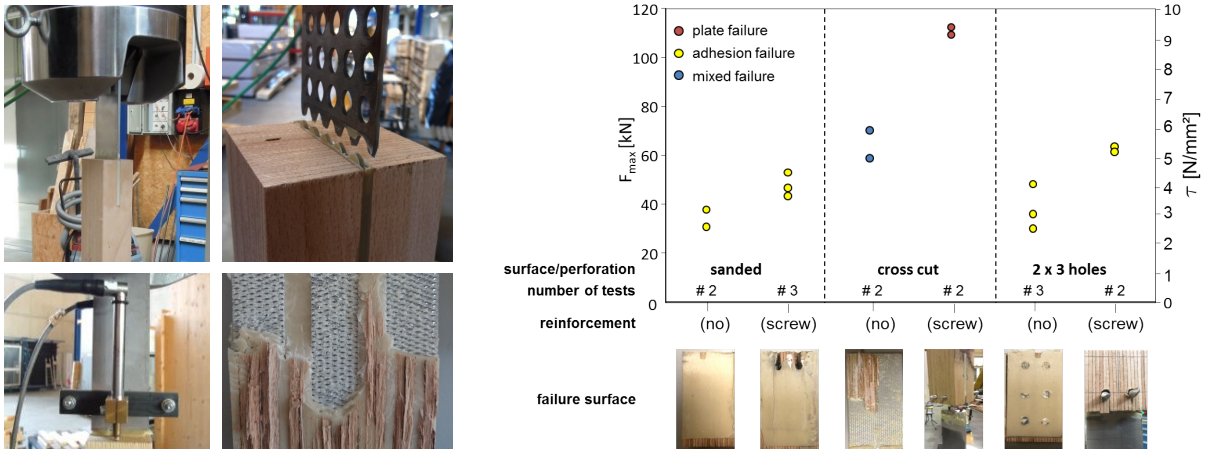


**Figure 3:** Test configuration (left),failure (mid), extract of results (right)

# H5: Grundlagen für ein vorgefertigtes und integriertes Fassadensystem zur thermischen Sanierung an bestehenden Gebäuden mit variabler Geometrie


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| Wirtschaftspartner: | proHolz Tirol<br>Cadwork Informatik GmbH<br>Schafferer Holzbau GmbH<br>Freisinger Fensterbau GmbH | Harrer GmbH<br>Saint Gobain Isover Austria GmbH<br>Isocell GmbH |


The thermal refurbishment of the building stock is one of the most fundamental challenges of sustainable urban development. Particularly the use of natural and local materials gets an increasing relevance, regarding the embodied energy. The focus of this work is the development of systematised solutions for thermal refurbishment with ecological, prefabricated and integrated façade elements.

The (Ecological-Timber) E.T.-prefab façade is developed to get a classified and systematised modular façade system, which can be used for buildings in building class 4 and 5 up to a fire escape level of 22 m without the demand of a fire protection concept (figure 1). The façade elements are self-supporting and can be mounted on the existing building in different ways. Depending on the building structure and material the element might be hanging, storey wise mounted, standing at the bottom or storey wise standing.

**Figure 1:** The E.T.-prefab façade

For an easy and fast mounting process special joint solutions are developed. They provide the prefabrication of the façade elements including the external render system in the factory, regardless of the weather conditions and with a high accuracy. These joint details enable an assembly on-site like a modular construction system and can be realized in few days (figure 2).

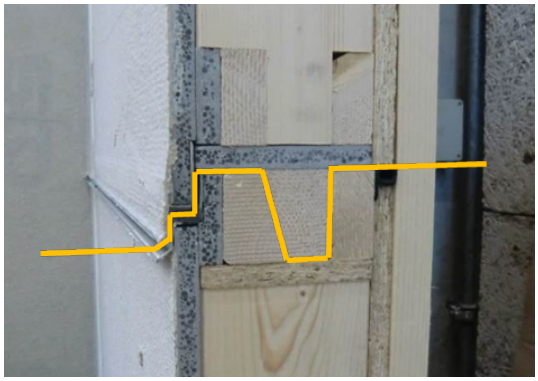

**Figure 2:** Sample of a horizontal joint detail

A façade system connector (figure 3) is evolved, which is applicable to variable building types and geometries, to mount prefabricated timber framed façade elements for building refurbishment or hybrid timber buildings. Recently project Partner Vinzenz Harrer GmbH received the property rights of the connector and named it SHERPA Efcon. The System-Connector has to fulfil several criteria. It must be multifunctional and easy to assemble, so that the façade elements are mounted in a very short construction time and it should be able to absorb tolerances in all three directions to compensate an uneven existing wall, inaccuracies in building survey or in prefabrication as well.

An important factor is the possibility to lead vertical and horizontal loads storey wise in the existing building, so that at the bottom no concentrated load introduction occurs. Thereby the application of prefabricated façade elements gets more flexible. A central role plays the accessibility of the connector during assembly. Its position on the upper part of the façade element and a detailed consideration of dimensions allow the handling from the top of the respective element.

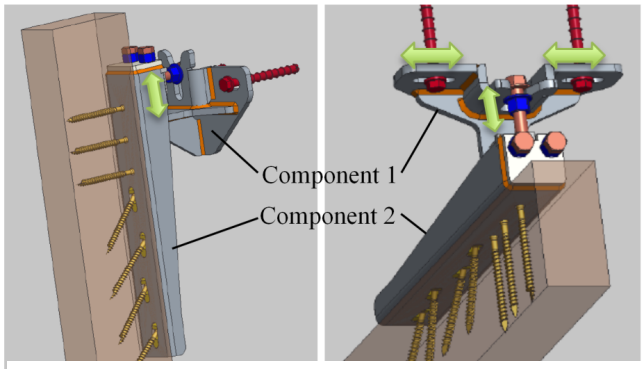


**Figure 3:** The SHERPA Efcon Connector

Due to the fact using ecological and combustible materials the fire safety plays an important role. To fulfil these fire protection requirements and to get a classified façade system for renovations of buildings in class 4 and 5 with more than 6 storeys tests are conducted at the IBS-Austria in Linz. To get a system solution and not only a product, the aim of this project is to classify an independent façade-system, so that different render-systems, external and internal panels but also insulation materials can be used.

**Figure 4:** Fire classification of the prefabricated façade system and prove the required fire protection goals in Austria

In a demonstration project the advantages and practicability of this refurbishment method are proved, the façade of the farmhouse in Trins of Prof. Flach is renovated by using the SHERPA Efcon connector and prefabricated façade elements with integrated windows and insulation.

A comparative ecological study of ETICS compared to the E.T.-prefab-façade shows its ecological advantage from Cradle to Cradle and the reduction of the global warming potential ($CO_2$-e) of 40%.

**Figure 5:** mounting process of the prefabricated facade elements, refurbishment of the farmhouse in Trins

The developments and research results contribute to raise the use of natural insulation materials and prefabricated timber framed façade elements for thermal renovation. An implemented refurbishment project, offered the opportunity to attempt, analyse and demonstrate the newest developments and to prove the user-friendly feasibility. Thus this work will contribute to a new standardised and cost-effective refurbishment measure for ecological thermal renovations.

# P1: Experimentelle Bestimmung und numerische Modellierung von Festigkeiten von Einzelfasern und Faser-Faser Bindungen in Papier

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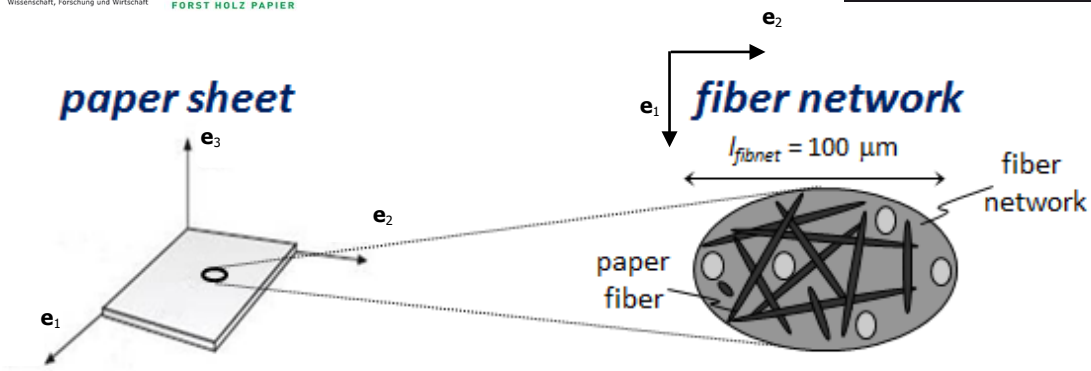
The project was devoted to the deciphering of the microstructural interactions governing the mechanical properties (in particular elasticity and strength) of paper. Our scientific hypothesis was that this would allow us to eventually predict overall paper strength from the strength properties of pulp fibers and/or their bonds.

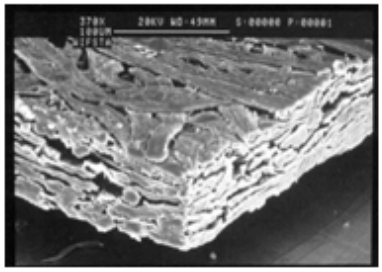
In order to cast the aforementioned microstructural interactions in a clear and efficient mathematical form needing only such input parameters which are directly accessible from experiments, we resorted to the theoretical framework of continuum micromechanics or random homogenization.

We resorted to micromechanical formulations based on infinitely many material phases oriented in all space directions, a concept which turned out as particularly successful and efficient for modeling the mechanical behavior of porous polycrystals made up of needle- or disc-shaped solid elements.

As regards the adaptation of these theoretical concept to the case of paper, the starting point was to restrict the orientation directions from those possible in the three-dimensional Euclidean space, to those possible within a plane in such a space, namely the paper plane (see Fig. 1).

Crucial breakthroughs of the project were: first, the identification of a morphology tensor function for the limit case of a disk embedded in-plane with respect to a transversely isotropic medium; second, the understanding that singularities in the transition from three- to two-dimensional Euclidean space emerged due to the vanishing of both aspect ratio and stiffness of the pore phase; third, the understanding that an appropriate two-dimensional representation of paper microstructure should involve a vanishing aspect ratio not only for the pore phase, but also for the infinitely many fiber phases; fourth, the understanding that such a representation only crudely captures the mechanical behavior of paper; fifth, the understanding that an accurate description of fiber geometry, as well as of the physical and chemical parameters at nanoscale relating to its elasticity, is absolutely critical to capture the mechanical behavior of paper. These findings ultimately led to a general model capable of simulating and accurately describing the linear elasticity and strength of virtually any planar transversely isotropic fiber network, paper included (see Fig. 2), and, very important, with high computational efficiency.




*interpenetrating network*
uniform orientation distribution of fibers *within paper plane*
***self-consistent scheme***

**Fig. 1** – Micromechanical representation of paper: (a) position with respect to orthonormal base frame ($\mathbf{e}_1$, $\mathbf{e}_2$, $\mathbf{e}_3$);

(b) Scanning electron micrograph of paper; and

(c) representative volume element (RVE) for paper micromechanics

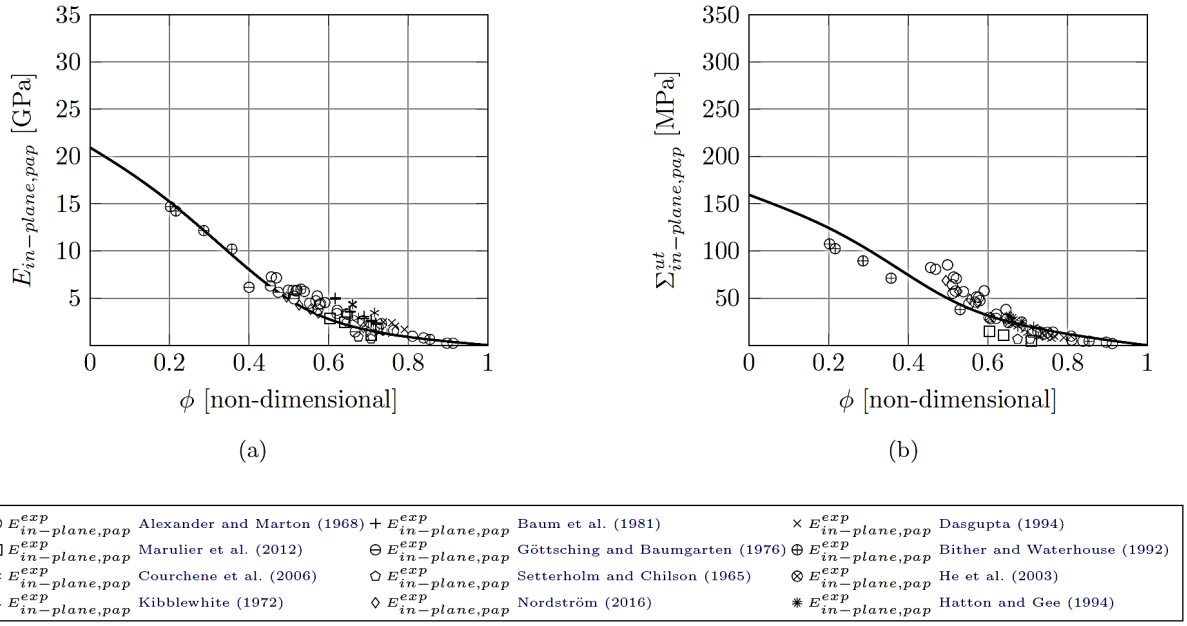



**Fig. 2** – (a): In-plane Young's moduli of paper micromechanically predicted for various porosities from single fiber Young's moduli of single fibers in fiber direction and compared to corresponding paper experiments; (b): In-plane uniaxial tensile strength of paper micromechanically predicted for various porosities from back-fitted in-plane (mode II) shear strength of single fiber-fiber bonds and compared to corresponding experiments

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It is well known that the strength of paper mainly depends on the strength of individual fibre as well as the strength of individual fibre-to-fibre bonds (Page 1969). In order to get better understanding about the influence of these two factors, the research work at the Institute of Paper, Pulp, and Fibre Technology, at Graz University of Technology is dealing with the experimental determination of the mechanical properties of the smallest constituents of the paper network. The work was carried out with respect to the type of wood (soft-/hardwood), influence of varying relative humidity and influence of refining.

Up until now, most of the studies dealing with the determination of mechanical fibre and bond properties have been performed using softwood pulps. Although papermaking properties of hardwood pulps differ from those of softwood pulps, both of them, when using blends, contribute to the strength of the final paper product. In the effort to obtain data for hardwood fibres and bonds, strength measurements using eucalypt kraft pulp (Jajcinovic et al. 2016) have been performed using the micro bond tester (Fischer 2013) developed at Graz University of Technology. In addition to that, the influence of varying relative humidity on the mechanical properties of fibres and bonds was determined. The results are shown in Figure 1.

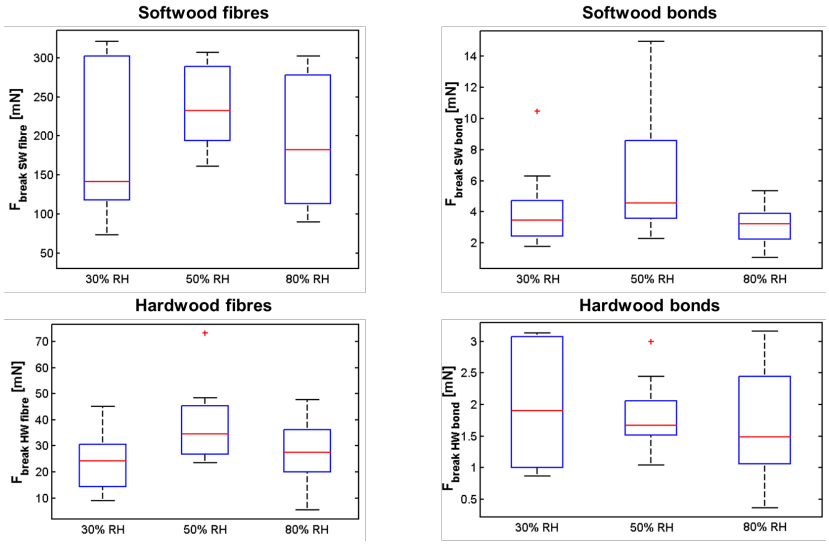
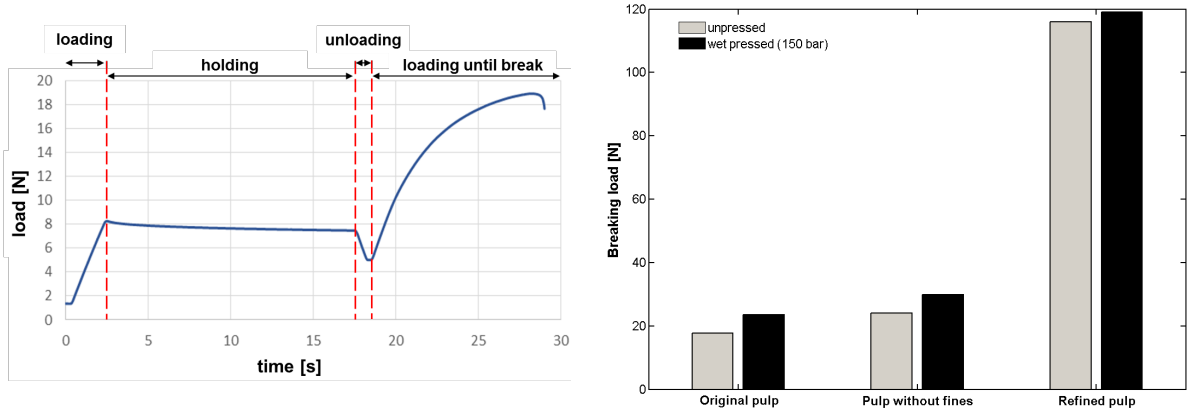



**Figure 1.** Results fibre and bond strength measurements.

Refining – mechanical treatment - increases the strength of the final paper product. This is attributed to internal (increased flexibility of fibre) and external (increased surface area of fibre) fibrillation of the fibres (Sirviö 2008). In order to better understand how this treatment changes the mechanical properties of the paper sheet, its impact on the fibre and bond level needs to be investigated. For this, different types of pulp (kraft and sulfite pulp) have been refined using a PFI mill. Afterwards the fibres and bonds were tested using the micro bond tester. In both cases, an increase in bond breaking load was visible upon refining (↑ sulfite pulp ~40%, ↑ kraft pulp ~ 28%). When it comes to individual fibres, a decrease in the breaking load was observed.

Besides fibre and bonds, fines, which are defined as the pulp fraction that is able to pass through a screen having a hole diameter of 76 µm, are known to promote the mechanical properties of the final paper product (Retulainen et al. 1993). To study the effect of these cellulosic microparticles, handsheets with and without fines have been prepared and analyzed. Furthermore, a part of the

sheets was pressed in the wet state before drying (10, 80 and 150 bar). Due to pressing, the sheets are getting denser which in turn also influences paper strength (Kajanto 2008). In the next step the sheet were tested using a Zwick tensile tester. The principle of testing as well as results obtained from these tests are shown in Figure 2.




(a) Principle of sheet testing (b) Results tensile testing

**Figure 2.** Mechanical testing of handsheets

Fischer, W.J. (2013). A novel direct method for mechanical testing of individual fibres and fibre to fibre joints. PhD thesis, Institute of Paper, Pulp and Fibre Technology, Graz University of Technology, Graz, Austria.

Kajanto, I. (2008). Structural mechanics of paper and board. In Niskanen, K. (ed.), Paper Physics, Book 16 of Papermaking Science and Technology, Chapter 6, pp. 229-264. Finnish paper Engineers Association/Paper ja Puu Oy, 2nd Edition.

Page, D.H. (1969). A Theory for the Tensile Strength of Paper. Tappi Journal, 52(4):674-681.

Retulainen, E., Moss, P., Nieminen, K. (1993). Effect of fines on the properties of fibre networks. In Transaction of the 10th Fundamental Research Symposium, pp. 727-769, Oxford, UK, Pira International, Leatherhead.

Sirviö, J. (2008). Fibres and bonds. In Niskanen, K. (ed.), Paper Physics, Book 16 of Papermaking Science and Technology, Chapter 2, pp. 59-92. Finnish paper Engineers Association/Paper ja Puu Oy, 2nd Edition.

Jajcinovic, M., Fischer, W.J., Hirn, U. and Bauer, W. (2016). Strength of individual hardwood fibres and fibre to fibre joints. Cellulose, 23(3):2049-2060

# P2: Auflösung, Regeneration und Funktionalisierung lignocellulosischer Formkörper

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One of the most pressing issues of today is the global waste challenge. Due to their longevity, plastics contribute to global pollution and accumulate not only in the oceans and onshore, but enter as well the food chains in form of micro- and nanosized particles. In this respect, not only better recycling and waste disposal strategies have to be developed, but also a fundamental rethinking is required aiming at the reduction of oil-derived and non-degradable plastics in everyday products. Cellulose as the most abundant polymer source shows great promise as an alternative to plastics. Nanocelluloses obtained from native cellulose fibers (cellulose I) have extensively been explored due to their sustainability, biodegradability, biocompatibility, high specific surface area, excellent mechanical and heat-insulating properties. Still, certain obstacles remain to be overcome by researchers and industry: the high production and energy costs of native fibrillated cellulose and the difficulties in drying due to its tendency to agglomerate irreversibly in this procedure and to lose many of their beneficial properties.

Keeping these issues in mind, this project P2 focused on a novel nanomaterial from the cellulose II allomorph, namely TENCEL® gel, its first-time in-depth physicochemical characterization and novel ways for chemical modification. In comparison to native cellulose nanofibrils, this gel is produced with much higher energy-efficiency from a precursor obtained directly out of the lyocell process, which manufactures cellulosic textiles and non-wovens by a direct dissolution approach. The gel is composed of individual microparticles forming particle-like aggregates with a uniform nanostructure consisting of nanofibrils of 40-60 nm diameter. In this thesis, water-redispersible TENCEL®[1] gel was obtained according to a new drying protocol, based on simple oven drying in the presence of negatively charged polysaccharides. Furthermore, the gel was used as precursor to produce highly porous aerogels with specific surface areas of up to 423 m2/g. The powders featured low thermal conductivity, low bulk density and high acoustic absorption at low frequencies and these properties render them promising as insulating material.

The introduction of negative charges onto the amorphous regions of the cellulose II gel via carboxymethylation caused a reorganization into spherical nanoparticles. Dependent on the amount of introduced charges, the particle size was tuned from a mean diameter of 73nm to 129 nm. The small nanoparticle (73 nm) fraction was found to be easily redispersible after drying and forms densely aggregated and transparent films with possible application as oxygen barriers in packaging.

A novel, straightforward and generally applicable functionalization strategy was established to introduce chemical anchor groups onto never-dried cellulosic surfaces. Based on an aqueous silanization protocol, never-dried nanocelluloses were decorated with azido groups and the resulting functional materials were successfully post-modified by a click chemistry approach.

In summary, the economic and straightforward production of this gel, its high surface area and porosity, the possibility to influence its particle size and to derivatize it in non-dried form; make it a promising contribution to the family of nanocelluloses with lots of scientifically interesting and economically feasible future applications.

[1] TENCEL® is a registered trademark of Lenzing AG.

## P3: Alterung von Lignocellulosen – molekulare Mechanismen und Analytik

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The introduction of Gutenberg's printing with movable letters was the first step in Europe towards mass production of printed goods, which were before that all handwritten, and allowed for a wider distribution of printed matter. Printing ground was rag paper and as the name suggests, rag paper was made of old rag pieces made from annual plants, such as hemp, linen or cotton.

Storing and preserving printed information is a big concern of conservation scientists also today. They are especially interested in the prevention of degradation, deterioration and yellowing of documents of historical value. The degradation of cellulosic substance is wide and complex field in material science, conservation aspects just being a minor part of the whole story. With regard to yellowing, the past decade has brought crucial progress insofar as the chemical structures of many of the chromophoric – literally "color-carrying" – substances have been identified in several cellulosic materials (Korntner et al. 2015; Rosenau et al. 2005; Rosenau et al. 2007; Rosenau et al. 2014; Rosenau et al. 2011; Rosenau et al. 2004).

The focus of this work lies on the investigation of chromophoric materials by mass spectrometric analysis, with the focus of non- to micro-destructive measurements of the sample material as needed in case of all valuable, historic paper objects. Since the compounds of interest are only present in minute amounts in the sample material, lying in the range of ppm to ppb, several attempts to enhance the detection capability of the method had to be made to overcome this main obstacle. Ambient ionization techniques were tested, and focus was laid on the application of desorption ionization mass spectrometry (DESI-MS) and paper spray-MS (PS-MS) PS-MS turn out to be superior to DESI-MS: it was used to characterize the sum of the signals of the cellulosic key chromophores along a yellowing gradient on book pages. In the second part, a key feature of ambient ionization techniques, the accelerated droplet reaction was exploited in the form of reactive paper spray-MS. This method enabled derivatization of the key chromophores in microseconds for enhanced sensitivity in a combined derivatization and detection process. Employing this method to naturally as well as artificially aged paper samples, the monitoring of specific compounds during aging of cellulosic substrate became possible for the first time. Since the equipment needed for ambient ionization techniques is still far from being common and widely available, a stable and robust method based on gas chromatographic separation with enhanced detection was introduced for monitoring cellulosic pulps industrial applications.

Thorough elaboration towards matrix removal, sample clean-up step, and quantitative derivatization increased sensitivity and improved separation, rendering this method attractive to both paper conservation science as well as application for pulp monitoring in commercial cellulose production.

In summary, two methods based on the application of paper spray mass spectrometry were developed, enabling the study of key chromophores and specific markers of cellulose degradation. Additionally, the applicability of DESI-MS as a non-invasive technique was demonstrated by measurements on highly degraded historic paper samples.

# P4: Enzyme Refinery von Lignozellulose

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| Betreuer: | Georg GÜBITZ (BOKU) |
| Ko-Betreuer: | Gibson Stephen NYANHONGO (TU Graz)<br>Rupert WIMMER (BOKU) |
| Wirtschaftspartner: | ACIB GmbH – Austrian Centre of Industrial Biotechnology |

The thesis within the project P4 entitled “Enzymatic synthesis of functionalized phenolic materials” focused on the establishment of modern enzyme based and environmentally friendly techniques for the up-grading of phenolic biomolecules. The resulting enzymatically modified materials, based on lignin and natural phenolics, should find applications in the medical and in the chemical industry.

The enzyme laccase was the biocatalyst of choice, since it is inter alia involved in the lignification process in plants together with other enzymes. Hence, laccases belong to the family of oxidoreductases and they oxidize a broad substrate range (phenolics, aliphatic and aromatic amines, etc.) with a concomitant reduction of oxygen to water. With the help of the laccase-mediator system (LMS), also non-phenolic materials can be oxidized. In this system, the laccase is oxidizing the mediator and the mediator acts as an “electron shuttle” and is further oxidizing the substrate. Since laccases are known to oxidize phenolic materials, their potential on the cross-linking of lignin and natural phenolics with other functional biomolecules like chitosan was investigated within this project.

Lignin is a by-product of the pulp and paper industry and is nowadays mainly burnt. Only 2% of this biopolymer is used for industrial applications and therefore maximum exploitation of new applications are of high interest for the science and industry community. Therefore, this carbon-rich, aromatic hetero-polymer can be modified to create upgraded lignins with certain properties. The laccase mediated modification of lignin causes a subsequent polymerization, which improves the properties and also the performance of lignin, which makes it interesting for the use as dispersants, adhesives, etc. Additionally, we investigated the laccase mediated cross-linking between natural occurring phenolics, which are also building blocks of lignin, and the biopolymer chitosan. Therefore, cross-linking results in the formation of bioactive hydrogels with medical relevance.

In a first step, the potential of laccase on the polymerization of lignosulfonate was investigated. Therefore, the presence or absence of external oxygen supply in combination with or without synthetic and natural mediators was examined. The polymerization process was monitored by analyzing the oxygen consumption, phenol content, fluorescence intensity, as well as the molecular weight. All these parameters are intrinsic properties that were analyzed during laccase mediated polymerization of lignosulfonates. For the first time, we demonstrated that in presence of external oxygen supply even low-redox potential laccases can efficiently polymerize lignins without the need for expensive and/or toxic mediators. This strategy also leads to a shortening of the reaction process and to a high polymerization degree with more than ten-fold increase of the molecular weight of lignins.

In industry, the costs of production are always a critical factor, and therefore we further tested the potential of immobilized laccase on the polymerization of various lignins. Immobilization of enzymes is gaining an increasing interest, since it allows reuse of the enzyme and it can increase the stability of the enzyme, broaden the solvent range, and can result in a reduction of temperature and pH sensitivity, etc. Therefore, we immobilized the laccase on a polymeric support (Accurel® MP1000 polypropylene beads). During this immobilization procedure, the enzyme activity and also the protein concentration in the reaction mixture was investigated to determine the amount of protein absorbed to the carrier. Around 70% of the protein was bound to the carrier and further tests demonstrated the remarkable activity on lignin polymerization as confirmed by analyzing the phenol

content, the fluorescence intensity, size exclusion chromatography, and oxygen consumption.

In another application, the same laccase was used for the cross-linking of natural phenolics and the biopolymer chitosan, which resulted in the formation of hydrogels without the use of any potential cytotoxic cross-linking agent (e.g. glutaraldehyde, formaldehyde, etc). Hydrogels are commonly used as wound dressings and they consist of a tri-dimensional polymeric network. Most of the hydrogels are made of synthetic polymers, however, biopolymers are gaining an increasing interest due to excellent properties like low cytotoxicity, anti-microbial behavior, biocompatibility, and biodegradability. In the present study, the cross-linking between the natural phenolics and the chitosan was confirmed by ATR-FTIR spectroscopy and HPLC-ESI-TOF mass spectrometry. Characteristics of all phenolic-chitosan hydrogels were elucidated by evaluating the cross-linking degree, the rheological behavior, the swelling, the potential to release a drug, and their porosity. The enzymatic cross-linked phenolic-chitosan hydrogels demonstrated excellent mechanical properties that are comparable to those of synthetic polymers. The cytotoxic effect of the phenolic-chitosan hydrogels was further investigated with a human cell line. The results demonstrated a great potential to release drugs and also no cytotoxicity for all phenolics (except for sinapic acid) was shown.

Furthermore, enzymatically cross-linked phenolic-carboxymethyl chitosan hydrogels were investigated on their anti-oxidant and anti-inflammatory behavior. The anti-oxidant property is important, since reactive oxygen species are the underlying cause of many oxidative stress related human diseases and can lead to an impairment of wound healing. The anti-inflammatory potential of hydrogels is also an important issue that was investigated within this study. Our hydrogels demonstrated radical-scavenging behavior and an anti-inflammatory effect, which for the latter was detected by inhibition of enzymes that are over-expressed in chronic wounds.

Overall, the thesis investigated the potential of laccase based processes for the synthesis of functionalized phenolic materials, to demonstrate their potential for biomedical and also bulk applications.