Transilvania
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Faculty of Wood Engineering

HABILITATION TREATISE

Valorisation of bio-based resources for innovative materials, products and applications

FH-Prof. Dr. rer. nat. Thomas Schnabel
Salzburg University of Applied Sciences
Department of Forest Products Technology &
Timber Constructions

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Summary

The habilitation treatise entitled "Valorisation of bio-based resources for innovative materials, products and applications" presents the author's contributions to the material science fields of theoretical, experimental and industrial research or implementation in production processes. This present thesis reflects the author's considerations and research merits after graduation the doctoral study at Technical University of Munich (Munich, Germany) in 2009. The habilitation treatise is divided into two main parts: i) scientific and ii) professional achievements with career development plans. Moreover, the different parts contain various chapters with some of the research conducted results from the author's finding during his research activity within Salzburg University of Applied Sciences and in collaboration with other institutes like University Salzburg, Åbo Akademi University in Turku (Finland), University of Ljubljana, University of Natural Resources and Life Sciences in Vienna, Technical University Munich, University of Applied Sciences Upper Austria in Wels (Austria), University of Belgrade, University of Lleida (Spain), FH Campus Wien - University of Applied Sciences in Vienna, and Holzforschung Austria in Vienna. The research activity is mainly related to circular economy and bio-economy. The transition from by-products materials to innovative raw materials and added value products is presented for various case studies. Based on the background from bio-economy some different materials were analysed and identified as possible new materials for different applications.

The first direction of research includes crop materials like different straw based insulation materials under the author's supervision (Schnabel et al. 2016b). Research with straw of various plant species resulted in low density insulation panels and in materials for blow-in insulation. The information about both insulation products was not published before the starting point of the research project BioInsPa in the year 2012 (Schnabel et al. 2016b).

The second main direction of research includes composite materials produced under the author's supervision from wood fibres and by-products from the leather manufacturing process (e.g. after tanning) (Wieland et al. 2010b;a). The possible combination of leather shavings/particles and wood fibres for in-

novative fibreboards showed improved material properties (Schnabel et al. 2019c). These first discussions of the possible material combination with different companies and the Salzburg University of Applied Sciences resulted in two further granted research projects (Schnabel 2015). Besides the compared mechanical properties with low leather amounts decrease the strengths of the panels with increasing leather ratio (Solt et al. 2015). However, enhanced fire resistance properties were only determined with a high leather portion of the fibreboards (Schnabel et al. 2019c).

The third research topic was about antimicrobial effects of wood and wood extractives as well as the condensation products from different wood processing steps (e.g. drying and steaming process) for the transformation from waste materials to added value products (Schnabel 2016). Fundamental knowledge was gained in the field of wood extractives and their influence on the microbial activities of wood. Wood samples as well as extractives from different wood spcies and wood processing steps were analysed for the identification of possible effects (Laireiter et al. 2014; Wagner et al. 2019).

Part 2 presents the progress and career development plans from the professional, scientific and academic point of view. Professional and teaching experience includes the short description of the personal development. Scientific research experience is reflected by the number and type of national and international projects which the author managed and supervised. These research and development projects are also important for the development of the department of Forest Products Technology & Timber Constructions at Salzburg University of Applied Sciences at the Campus Kuchl and they should strengthen the transnational and cross-sectoral collaborations between academia and industry within research and Erasmus projects. This includes further collaboration with the Faculty of Wood Engineering at Transilvania University of Brasov. Proposals of further research activities are based on nature-inspired subjects related to transfer technical into biological processes for the material and product development. The changes of these processes may play an important role for solving the challenges in future and to save energy and to reduce material consumption.

Contents

Part A

Scientific achievement

This present part reflects author's research activities and merits after graduation the doctoral study at the Department of Forestry Science and Resource Management at Technical University Munich in 2009 (Munich, Germany). The main focus was the innovative use of by-products from different bio-based industries, which were not used at the current state and could be suitable for new materials and/or products or the production of ligno-cellulosic composite materials with and without wood for different applications. General, the projects were in line with the concepts of the circular economy as well as the bio-economy. For innovative projects a fundamental knowledge of characterisation methods (Schnabel et al. 2014; Wagner et al. 2015) and a good overview of raw material (Wagner et al. 2020a; Schnabel et al. 2019c), wood processing processes (Meints et al. 2016; Schnabel et al. 2017; 2007; 2016a) and/or by-products in different industry sectors (e.g. tannery and wood industry) (Rindler et al. 2015; Schnabel et al. 2019a; Wagner et al. 2018a) are necessary. These research ideas were implemented into thirteen projects with academic, research & development as well as industry partners at different European countries and supported by public regional, national and European funding programmes (e.g. Austrian Research Promotion Agency (FFG), Danube Transnational Programme (DTP) and Alpine Region Preparatory Action Fund (ARPAF)). Within the various research projects the positions were taken over as expert and director with scientific, management and financial tasks. Furthermore, the author's research activities were mainly within Salzburg University of Applied Sciences, but also at University Salzburg, Åbo Akademi University, Elettra Soncrotrone Trieste, University of Ljubljana, University of Belgrade, University of Lleida, University of Natural Resources and Life Sciences, Technical University Munich, University of Applied Sciences Upper Austria, FH Campus Wien - University of Applied Sciences, and Holzforschung Austria. Nevertheless, companies were also involved in the research progress to give them new opportunities for the development of new materials, products and processes.

The various research projects and the collaboration with the mentioned institutes were fruitful for gaining new knowledge in the ligno-cellulosic material science area regarding the bio-economy of different industrial by-products and increasing the up-cycling. These experiences and knowledge were also transferred to the students in different study degree programmes and an international audience with different workshops and mentoring sessions to strengthen the cross-sectoral collaborations between different industry sectors for developing new value chains (Atena and Schnabel 2018; Morandini et al. 2018). In this process, scientific bachelor's, diploma's and master's theses were also supervised within the framework of teaching and research at Salzburg University of Applied Sciences. A doctoral thesis was co-supervised within the cooperation with University of Salzburg concerning the characterisation of wood extractives in different tree tissues and solvents (Wagner et al. 2018a; 2020a).

The research studies are related to the circular economy and bio-economy. The transition from lignocellulosic and tannery waste materials to innovative materials and value-added products is presented for various case studies. Based on the background from the bio-economy some different materials were analysed and identified as possible new composites and products for different applications (Schnabel et al. 2020a). The results presented below were generated during individual research projects in various scientific fields (Schnabel 2015). The product development of wood and other lignocellulosic based composites including issues related to material up-cycling, characterization and identification of specific use. This intellectual approach was also performed for development of high added value from waste products.

The introduction of the bio-economy concept in the timber value chain is presented through the results from Schnabel et al. (2020a) in chapter 1. The novelty of the bio-economy approach lies in rethinking the whole system of all various actors. The competitiveness through innovation is one objective that leads to economic growth and increases employment in compliance with environmental sustainability.

Chapter 2 of the part A is focused on the research results of crops materials and their applications as different insulation materials and it is based on different results from Schnabel et al. (2020a; 2019b;d); Nagl et al. (2015c); Krenn et al. (2017a). Research with straw of various plant species resulted in low density insulation panels and in materials for the blowing insulation. The information about the both insulation products was not published before the starting point of the research project BioInsPa in the year 2012 (Schnabel et al. 2016b). Within this project basic and applied research in the area of insulation panels and blowing insulation were performed by using crops materials (Nagl et al. 2015c; Krenn et al. 2017a; Schnabel et al. 2019a). Different properties of the material could be observed and new applications of the crops material were identified and proposed.

The next chapter 3 deals with the research of innovative composite materials produced under the author's supervision from wood fibres and by-products from the leather manufacturing process (e.g. after tanning) based on the study from Rindler et al. (2015); Schnabel et al. (2019c); Solt et al. (2015); Tondi et al. (2015); Wieland et al. (2010b;a). The possible combination of leather shavings/particle and wood fibres for innovative fibreboard were developed and characterized by Wieland et al. (2010b). These first discussions of the possible material combination with different companies and the Salzburg University of Applied Sciences resulted in two further granted research projects (Schnabel 2015). The composite panels and fibreboards were produced with various ratios of leather and different glue amounts and compounds. Besides the compared mechanical properties with low leather amounts decrease the strengths of the panels with increasing leather ratio (Solt et al. 2015). However, fire resistance properties were determined with a high leather amounts of the fibreboards (Schnabel et al. 2019c).

Chapter 4 is regarding antimicrobial effects of wood and the use of wood extractvies as well as the condensation products from different wood processing (e.g. drying and steaming process) for the transformation from waste materials to added valued products based on the results from Kavian-Jahromi et al. (2015); Laireiter et al. (2014); Schnabel et al. (2016a); Wagner et al. (2019; 2020a). Fundamental knowledge was gained in the field of the wood extractives and their influence on the microbial activities of wood. Wood samples as well as different extractives were analysed for the identification of possible effects (Laireiter et al. 2014). Only a few compounds of the huge amount of extractives from the larch wood and/or bark have antimicrobial effects on different stains and bacteria (Wagner et al. 2019).

General, the project concepts were in line with the approaches of the circular economy as well as the bio-economy. An interdisciplinary thinking in different subjects are crucial to develop new value chains (Atena and Schnabel 2018; Morandini et al. 2018; Schnabel et al. 2020a).

1 In the interplay between material science and bio-economy*

Bio-economy is a concept that targets to replace fossil resources with renewable raw materials in as many areas and application as possible. Hereby, the collaboration between different industry sectors are necessary for saving natural resources (Bezama 2016; D'Amato et al. 2017). One by-product from one industry sector may use in another industry sector for the substitution of fossil resources and/or new materials as well as applications, which is very important today and for the future (Jarre et al. 2020; Mair and Stern 2017; Zabaniotou 2018). Agroforestry residues can used as main sources of the basic building blocks for chemicals and materials, and especially bark is the most promising sources with a high bio-economic potential (Thorenz et al. 2018).

The total European bio-economy (the EU-28) amounts to about 2.1 trillion euros in turnover and 18.3 million employees (Piotrowski et al. 2018). It includes the food, feed and beverages sectors, which are responsible for roughly half of the turnover. The bio-based industries - chemicals and plastics, pharmaceuticals, paper and paper products, forest-based industries, textile sector, biofuels, and bio-energy - contribute with 600 billion euros (28.6 % of total turnover in the European bio-economy) and 3.2 million employees. According to this general picture it must be assumed already a well-established position of bio-based industry in European bio-economy with an evident relevant role of bio-energy (Lewandowski et al. 2019; Piotrowski et al. 2016a). It is not always easy to track biomass flows for energy purposes if much information is available, due to the regional and national rules which are quite stringent (Becker and Brunsmeier 2013; Pieratti et al. 2020). It is beyond doubt that bio-energy is one of the main tools to mitigate climate change; nevertheless, in some regions, the energy market has changed due to the increased value of wood chips. Moreover,

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*The author managed and did the research in the field of innovations to foster the circular-/bio-economy in the forest-based value chain between 2017 and 2020 within two European research projects with 23 international partners. These research results were presented in 3 scientific articles and books as well as in 2 international conference contributions. The author contributed to the funding acquisition, design and implementation of the research, to the analysis of the results and to the writing of the manuscript these articles.

wood biomass for energy also needs to use wood material of lower quality (not only chips or side stream) with the risk of diverting attention from other possible uses (Teischinger 2017). The generation of energy has created new jobs like the external service, which is in charge of collecting residues from forests and sawmills in order to sell them to public and private energy plants (Pieratti et al. 2020). The potential of forests in the framework of bio-economy is quite important, the most part of biomass is growing in the forest and it covers about 40 % of land in Europe (https://ec.europa.eu/eurostat), in which the potential of available forest biomass is spatially distributed across the European continent (Verkerk et al. 2019).

The estimated amount of available global biomass in the year 2011was 11.4 billion tons of dry material for food, feed, energy and material use (Chirat 2017). This amount was supplied firstly by agriculture - harvested agriculture biomass (40 %) and grazed biomass (31 %) and harvest residues (12 %) - and then by wood with 18 % and 2.12 billion dry tonnes. However, only 10 % of this amount with the exception of wood was used for the production of materials and products (Piotrowski et al. 2016b). Many scenarios in different studies indicated a decrease in biomass supply in Europe in the future (Stern et al. 2015). The decrease of wood availability is coming from exterior due to the establishment of new and large wood processing capacities in the arising countries. This process will enforce the use of local biomass both form agriculture and forestry in order to satisfy different industry requirements and the need of new value chains. The harvesting of wood from natural forest is considered a sustainable process, and cannot be easily increased in a short period of time. Therefore, other strategies are necessary for fulfilling the needs of biomass for different industry sectors.

An intelligent use of the natural resources starts from saving the raw material usage, extending an efficiency production process, a lengthening service lifetime and include a possible energy-saving recycling. In the last years it has been becoming more and more important to insulate a building in an ecological way. A well-insulated home reduces the energy consumption by keeping the building warm in the winter and cool in the summer. This in turn cuts down carbon emissions linked to global climate change. A further point is to ensure and secure that stock of fossil fuels which remain available and use currently unused waste materials to produce new materials and products.

Therefore, it needs a research-based knowledge gain to understand different materials and/or processes and use it for innovations in technology to developing sustainable and competitive bio-based products (Schnabel et al. 2020a; Tondi and Schnabel 2020). This strategy has a long tradition in the bio-based industry and in the wood-based industry and is an ongoing process. The importance of resource efficiency will become more pronounced in the future as populations grow in large mega-cities and competition from natural resources, energy and water become more intense (Hill 2006; Becker and Brunsmeier 2013; Knigge and Schulz 1966). The resource-efficient manufacture and use of bio-products reduces the amount of waste materials as well as allowing the recycling of materials (Mair and Stern 2017). In the industrial symbioses established by co-operation partners, production side streams are processed into high-grade products and at the same time natural resources are saved. This is the circular economy approach at its best (Atena and Schnabel 2018). The principles of circular economy have been used for a long time in the forest industry. The results of various research studies and laboratory scale applications show that almost every part of the tree can be used and processed to give high-quality sustainable products. The products can be recycled many times and finally used for bio-energy production, which should be only the last step in the forest timber value chain. Also, the work of the author is settled in this research field.

New approaches were developed and analysed in detailed, when the author's doctoral study was over in the year 2009. One of author's main research focus was the up-cycling of different by-products, which were not used in this time and have great potential for the substitution of other raw materials in different applications (e.g. insulation and fibreboard) (Schnabel et al. 2016b; Wieland et al. 2010b). The characterization of wood extractives and their influence on the wood and the interaction with further properties for different applications were also analysed (Eckardt et al. 2020; Schnabel et al. 2014; Wagner et al. 2018b). The current presentation of these research areas are in line with the bio-economy approach. The transformation of traditional industrial processes to sustainable and innovative role models is compulsory given by limited resources and adverse environmental effects (Atena and Schnabel 2018; Morandini et al. 2018). These research works were granted of various national and international projects in cooperation with various universities, research institutes and companies.

An overview of current wood-based products, materials, processes and applications are shown in Figure 1.1. Normally, the first step in the timber value chain after tree harvesting is the processing of the round

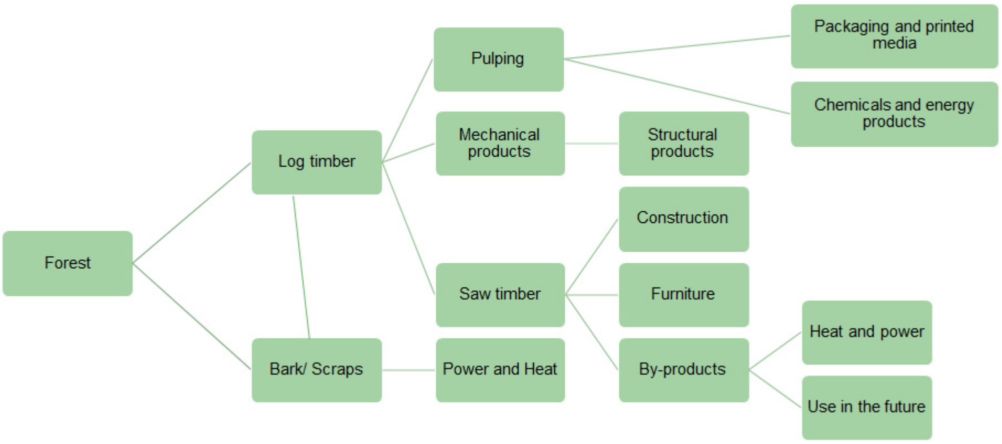

Figure 1.1: Sketch of current forest-based value chain in the Alpine region (Wagner et al. 2018a)

wood in sawmills (Schnabel et al. 2020a). The opening cut of the timber is carried out by using various cutting methods and devices (e.g., frame saw, band saw or chipper). Therefore, the yield is quite different and is depending on the sawing technology and wood quality. However, the products for this first processing step are sawn wood, sawdust, wood chips and bark for further applications (e.g. constructions, pulping, or energy production).

This traditional timber value chain has been developed according to the local circumstances since a long time. Based on the circular economy and bio-economy approaches new ideas concerning the utilization of current by-products of this value chain has move to the forefront (Schnabel et al. 2020a). Especially, the bark and other tree tissues (e.g. branches, and knots) are interesting as a supplier of bioactive compounds with high pharmaceutical and cosmetician potentials for further applications (Laireiter et al. 2014; Wagner et al. 2019; 2020a). A new bio-refinery approach could be developed for these special tree tissues to extract valuable substances. This extension of the current timber value chain is shown in Figure 1.2, where the innovative section is connected with dotted line. Also, other substances form forestry biomass are used for various products and applications (Table 1.1). Different sugars (e.g. glucose) are used for different building blocks of bio-based plastics materials (e.g. polyhydroxyalkanoates (PHA), polylactic acids (PLA)) (Piotrowski et al. 2018). Nevertheless, tree extractives play an important role in the recovery of valuable bioactive compounds from natural resources. These secondary metabolites can divided into different main chemistry classes, in which the polyphenols are very interesting for a wide number of applications (Fengel and Wegener 2003; Wagner et al. 2019).

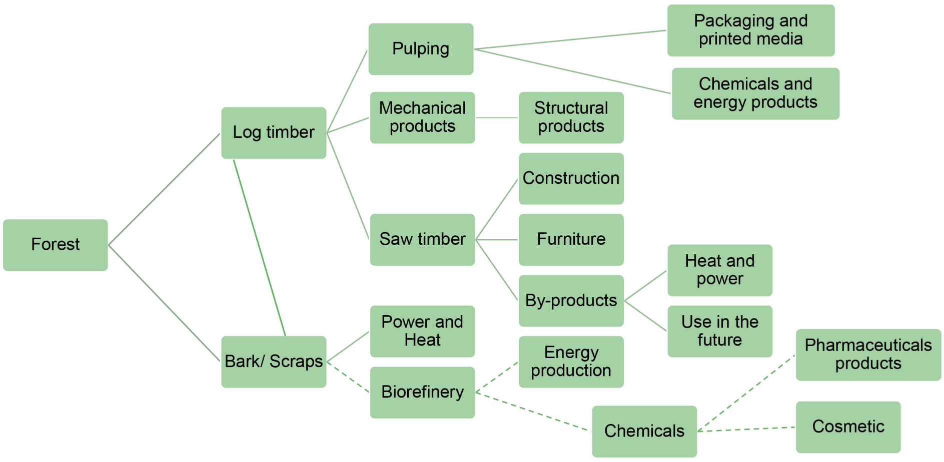

Figure 1.2: Overview of an innovative section (connection with dotted line) of the traditional forest-based value chain in the Alpine region, modified from Wagner et al. (2018a) according to Schnabel et al. (2020a)

All these innovative products for different applications were collected from the bio-refinery concepts of the pulp and paper industry as well as research projects. Nevertheless, there is no real limit to expand the bio-refinery concepts of virgin wood to other tree tissues and/or recycled wood (Stern et al. 2015).

Table 1.1: Summary of innovative products derived from forestry biomass (Schnabel et al. 2020a)

wood constitutes	products & applications
cellulose	textile, fibres, glucose, ethanol, building blocks
hemicellulose	food encapsulation, cattle feed, hydrogel
lignin	adhesives, carbon fibres, foams
extractives	essential oil, food and dietary additives, bioactives, cattle feed, tannin foams

Therefore, the future of an Alpine circular-/bio-economy is strongly connected to the material use of wood residues in these regions (Atena and Schnabel 2018). Regions across Europe are currently establishing their own development paths towards more sustainability or a comprehensive bio-economy strategy, or have done so already. The potential of innovation of the material use of wood-based residues thus hinges on the design and implementation of these strategies and how these could be connected, eventually (Schnabel et al. 2020a).

Products, applications and research projects in this report offer competition in a number of markets. When wood residues can be effectively and efficiently sourced, companies are able to provide alternatives to fossil-based products and materials. Markets, where competition from wood-based materials exists, include among others granules for injection moulding, fuels, landfill material made from paper mill residues, or carbon-rich soil additives. Therefore, the use of residues from forestry and wood processing potentially provides added value for foresters and the wood-working sector as a whole. An investigation on innovation diffusion of new wood-based materials demonstrates that, within the forestry and wood processing sector, additional skills and/or partners need to be included for the success of new materials and wood-based products. Moreover, it is held that market expertise of relevant forestry and wood processing actors needs to extend beyond the knowledge of the next step in the value chain (Schnabel et al. 2020a). To succeed in the commercialisation of new materials and novel wood-based products and applications, different skills and mind sets are required from those needed to sell the standard volume products of the forest and wood-working sectors.

Clearly, the market potential of wood-based residues lies in innovation beyond the classical timber value chain. What is therefore needed is the successful connection of primary biomass producers in remote Alpine areas with processors and producers of intermediate products and applications (Atena and Schnabel 2018). As of today, only wood that adheres to specific quality criteria can be processed into timber used for construction or carpentry, furniture and frames. Therefore, huge potential for innovation and marketisation lies in the valorisation of lower quality wood or unused materials in biorefineries and bio-product mills, for example. To this end, a network of decentralised processing facilities across remote areas would be best to cater for the needs of biomass producers, biomass processors and manufacturers of bio-based products alike.

The effective utilisation of materials and energy resources is becoming an increasingly important challenge for the economy and society. There are various approaches to solve this challenge. One option for the companies can be to react with this situation and they can develop new materials and products from available and sustainable natural resources. Improvements in efficiency (cost effectiveness) as well as the modernisation of existing methods and technologies are important and will also be crucial for leading companies. Resource efficiency involves the consideration of all the stages of the life cycle of a product, including extraction of resources, production, the use of the product and recycling.

On the other hand, the academia can work on different levels to transfer the knowledge and technology into enterprises (Morandini et al. 2018). Besides the basic research projects, the universities have carried out applied research projects in collaboration with companies. Nevertheless, the knowledge transfer to the student is the most important goal of educational institutes, and this can support the development of innovative products. Young people work in well-equipped laboratories during the study degree courses and can develop their own skills, personality and experiences. Many of the students are multiplicators; they will work in the future for companies and they are enthusiastic for development of new materials and products as well as their own business (Schnabel et al. 2020a).

2 Ligno-cellulosic based materials for insulation products[*]

The scarcity of resources can be a motor of motivation for material-driven changes within the existing value chains. The development of new products through the utilisation of unexploited bio-based resources is one way where innovation can happen. By using these unknown raw materials, the product properties of the composite materials can change; thus, characterisation is necessary to determine the material properties of the composite materials. Furthermore, changes in material behaviours can also results in innovative applications and the discussion about new materials, products and processes (Schnabel et al. 2015; Huber and Schnabel 2015; Huber et al. 2019; Schnabel et al. 2020b).

This study deals with many different pre-treatment processes (e.g. mechanical, enzymatic or thermal-mechanical processes) of the raw materials of various plant species. Here the influence of the different treatment processes on the chemical, structural and technological changes of the material properties was analysed. Subsequently, the matter was used for the production of insulating materials with natural adhesive systems on a laboratory scale, used to possible suitability of the treated ligno-cellulosic fibres for insulating (Schnabel et al. 2020b). Up to now, technical obstacles with different material pre-treatments, among others with a straw digestion by a steam explosion process. This treatment combines the effects of a mechanical pre-treatment with a thermal pre-treatment.

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[*] The author managed and did the research in the field of product development of cereal-based materials between 2010 and 2015 in the frame of two research project with five Austrian companies. These research results were presented in 8 scientific articles and books as well as in 6 international conferences. The author contributed to the funding acquisition, design and implementation of the research, to the analysis of the results and to the writing of the manuscript of these articles.

2.1 Research approach

The usage of bio-based material for various products to save and protect buildings against heat loss has an old tradition. Today up-cycling and recycling of products are become more and more important. One possible opportunity to save raw materials (e.g. wood) and to substitute oil-based products is the usability of cereal straw as thermal insulation materials (Adolph 2014; Nagl et al. 2015c). In Figure 2.1 is shown the research approach for the usage of unexploited raw straw materials from the agricultural areas. Straw as feed stock is inexpensive, fast growing, locally available and easily manipulative (Ashour et al. 2011). Normally, if the soil needs plant nutrient matter, then the straw materials are left/burned in the field.

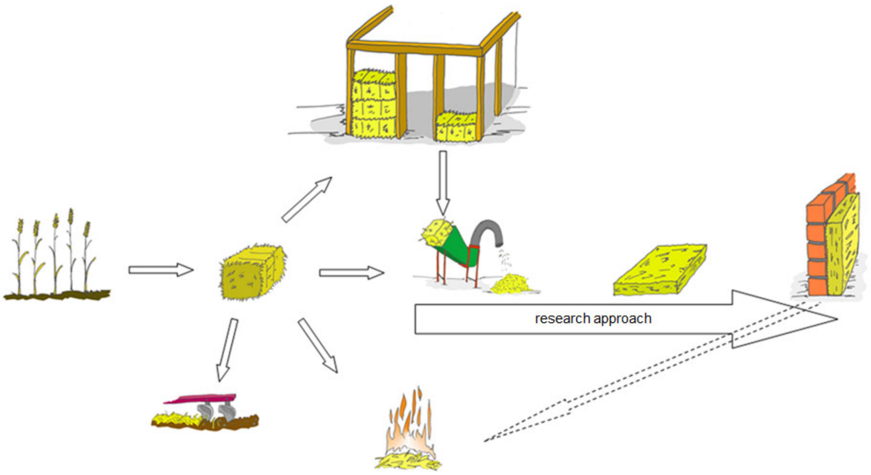

Figure 2.1: Research approach for the development of new insulation materials from different straw species (Schnabel et al. 2019a)

However, there are some agricultural fields that do not need the supply of additional substances. For this area the straw can be used for other application. On the one hand the straw is also used for bedding of livestock breeding. On the other hand, this cereal straw can be used to develop innovative products (Lewandowski et al. 2019). The harvesting and production of the straw bale in the field is important for the further usability. Currently, straw bales are used either as load bearing structure or as infill wall (e.g. wood beam structures). Moreover, the increasing of the market share of the bio-based insulation materials may yield a positive effect on long-term CO_2 fixation (Jarre et al. 2020).

Effective insulation materials support climate protection due to their energy saving properties. The most common materials are made of synthetic foams or mineral wool both of which are based on crude oil. Natural insulation materials out of hemp, flax, straw or sheep wool have a much lower share in the market. Social rethinking and public awareness for sustainable products are essential to increase and strength their market share (Schnabel et al. 2020b). Gellert (2010) concludes that insulation based on herbal or animalistic raw materials have a market share in Europe of about 4 to 6 %. Straw as stating material is locally available, inexpensive, fast growing and has been used as building material for centuries. Beside these advantages also considerable drawbacks like thickness of the wall if different size of bale of straw are used for the load transfer and/or insulation issues (Nagl et al. 2015c; Krenn et al. 2017a). Also, the hierarchic structure of the plant material results in anisotropic material properties and has to consider for the applications. The construction of a house with prismatic or cylindric straw bales needs some work experience and good material knowledge for the application, then the bales showed also anisotropic behaviour with the vertical strain for vertical orientations higher than those for horizontal orientation (Schnabel et al. 2016b). Therefore, a thin insulation panel produced with standardized material properties and low density (<300 kg m^{-3}) can boost the practicable materials for re-constructions and retrofitting of buildings (Huber et al. 2015; Nagl et al. 2015c;a). This idea has been increased focused on the research since the last years within urbanised areas. However, raw materials of annual plants particle/fibres are less homogeneous due to their structure and also the adhesion of straw boards does not yield good results with standard adhesive systems (e.g. UF) (Nagl 2014; Krenn et al. 2017a; Zhang et al. 2003).

2.2 Characterisation of cereal straw and plant

Based on the mentioned consideration the properties of four various plant materials were analysed. The air-dried hemp, corn and miscanthus have been shredded using a customary shredder (Figure 2.2). Wheat straw has been mulched resulting in slicing the stems in longitudinal direction. Especially hemp created a woolly-fibrous material when shredded. A high portion of leaves of the corn plant can be seen in Figure 2.2b. The particle size distribution of the materials used, based on weight which varies significantly (Figure 2.3). The portion of material fractions below 250 μm were almost zero for the chopped raw materials except

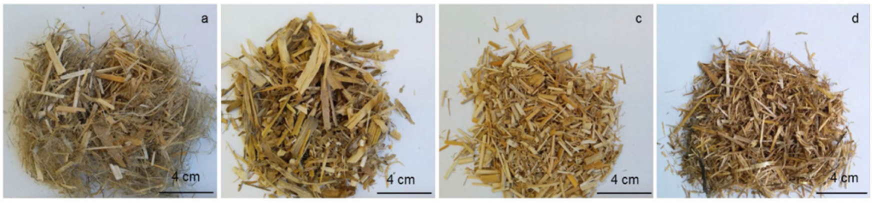

Figure 2.2: Selected stalks from various plant species: a) hemp, b) corn, c) miscanthus and d) wheat (Nagl et al. 2015b; Krenn et al. 2017a)

for the wheat straw. Also, in the next fraction the wheat materials presents a higher amount of small particles (0.25 - 1.0 mm). In the particle fraction between 1.0 and 4.0 mm the different weight amounts of the materials from miscanthus and wheat are higher than from the other two materials. While chopped hemp and corn <4 mm have a higher amount of the materials in these fractions than the miscanthus and mulched wheat. However, due to the hemp fibre interaction to a conglomerate a small falsification in this value can exist by using the shaking- or vibrating procedure for the determination of the particle size distribution. The distribution of the particle size of the raw materials has a large influence on the further material properties (e.g., insulation board) (Schnabel et al. 2019c). The average moisture content of the four various materials in this study was 9.9 % with a standard deviation of 3.02 %, where no material had a large difference. A woolly fibrous and inhomogeneous material was formed during a chopping of hemp. This inhomogeneous material structure creates additional cavities that cannot be completely filled with fine particles and resulting in the lowest value of bulk density of 29.72 kg m^{-3}, followed by maize stalks (48.62 kg m^{-3}) and wheat straw (56.56 kg m^{-3}). Miscanthus results the highest value of bulk density of 137.63 kg m^{-3}.

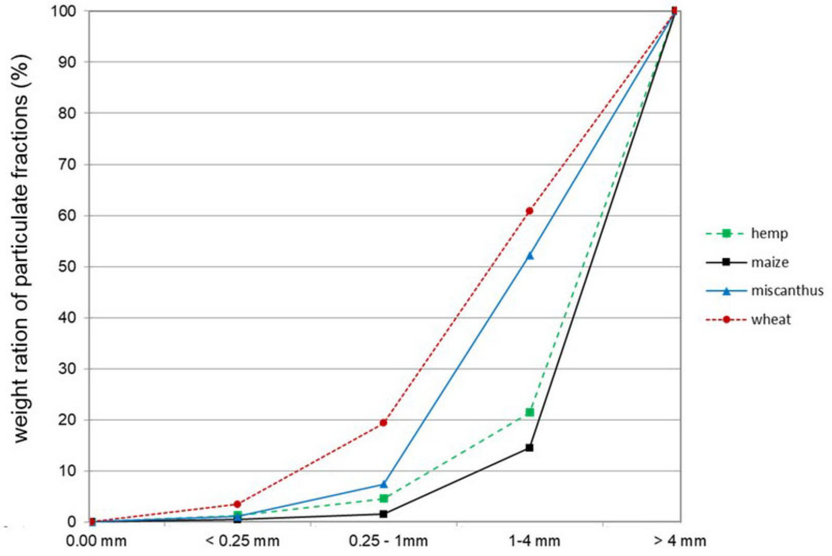

Figure 2.3: Proportion of weight of the four various materials as a function of particulate fractions

This density also influences the board properties, as no dimensionally stable boards with low bulk density than the bulk density could be produced by hot pressing (Nagl et al. 2015c).

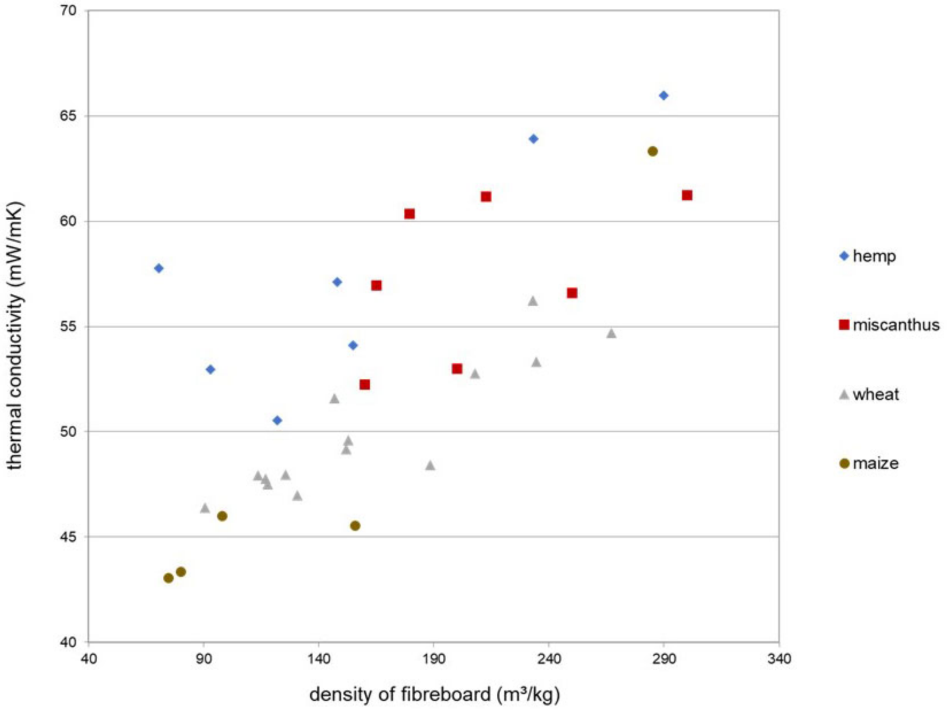

Figure 2.4: Thermal conductivity of the used panels subject to the density at a measurement temperature of 10 °C and a temperature difference of 15 K

2.2.1 Development and analysis of insulation materials

The aim of this study was the production of dimensionally stable insulation boards below 300 kg m^{-3} and the investigation of thermal conductivity. Therefore, board insulating material with different raw densities (Figure 2.4) and dimension with 45 x 45 x 3 cm^{3} were produced with the four various raw materials. Before measuring the thermal conductivity, the boards were stored in a standard climate at 20 °C and 65 % relative humidity until their weight remained constant.

The production of the insulation boards was carried out without difficulties, only when using the hemp material the used plough blade mixer had to be rebuilt and the ploughs had to be removed in order to be able to glue the fibres successfully. The lowest board density of 80 kg m^{-3} could be achieved from maize stalks, below which it was not possible to manufacture the laboratory boards in a dimensionally stable manner. Miscanthus required a bulk density of at least 160 kg m^{-3} for a stable insulation board. Even wheat straw as an extremely difficult raw material to bond (Krenn 2016; Krenn et al. 2017a; Zhang et al. 2003) could be produced here as an insulating board with raw densities from 90 kg m^{-3}. Further investigations, the UF adhesive used could be replaced by a tannin/hexamine binder (Krenn et al. 2017a). All materials show a dependence of thermal conductivity on board density over the investigated density range (Figure 2.4). A reduction of the bulk density has a favourable effect on the thermal conductivity. Low thermal conductivity values mean higher thermal insulation properties. The lowest thermal conductivity values with comparable raw densities were determined with the boards made from wheat and maize straw. With the inhomogeneous material of hemp, no dimensionally stable insulation board could be produced by using hot pressing process below 70 kg m^{-3}. However, a local optimum of the thermal conductivity function of the hemp insulation board was found by approximately 122 kg m^{-3}. If the board density is lower than the mentioned values then the material distribution of the board was inhomogeneous and some open pores may exist resulting in increased heat convection and high thermal conductivity values. The investigated boards from raw straw materials from annual and perennial plant can compete with the insulation materials on the market with regard to thermal conductivity (Nagl et al. 2015a).

The studies from Nagl et al. (2015a) and Nagl et al. (2015b) deal with the investigation of straw boards using UF-glue produced at densities lower than 300 kg m^{-3}. However, Krenn et al. (2017a) used sieved fractions >2 mm of maize, miscanthus and wheat straw for production of insulation boards glued with renewable materials (e.g. tannin) (c.f. Figure 2.5). Because the fine part absorbs a relative high amount of the used adhesive compared to the other fractions; this behaviour does not result in better adhesion of the insulation boards. For wheat straw 92.6 % of the material was used for the manufacturing of the boards, whereas for maize 97.2 % and miscanthus 95.4 % of the materials were used, respectively. Further, for different types

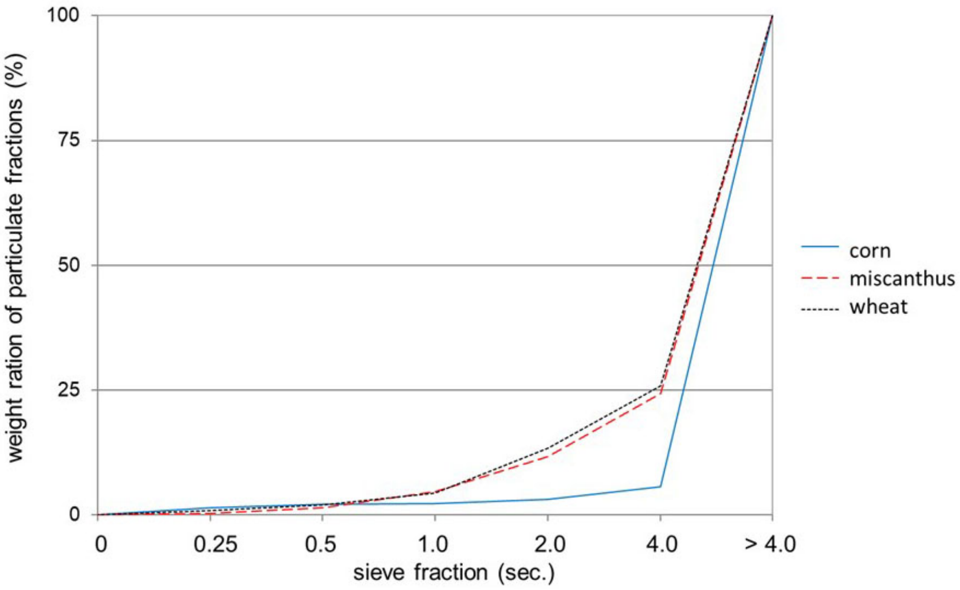

Figure 2.5: Particle distribution of straw varieties maize, miscanthus and wheat (Krenn et al. 2017b)

of boards, beside the low thermal conductivity important parameters were recorded e.g. water absorption and the fire behaviour, then assessed or compared.

The manufacture of the insulation boards from various ligno-cellulosic raw materials and adhesives took place without difficulties. In addition to the UF adhesive used by Nagl et al. (2015a) and Nagl et al. (2015b) for their experiments, a tannin mixture and water glass were used as glues. Before testing the thermal conductivity, water absorption and fire behaviour, the insulation boards were stored in a normal climate at a temperature of 20 °C and a relative air humidity of 65 % until the constant weight was achieved. The tannin binder (mimosa tannin) was mixed with 50 parts by weight each of tannin powder and water in a laboratory mixer. In the following step, the solution was adjusted to pH 8.5 with a 10 % sodium hydroxide solution using a pH meter. Shortly before gluing, 3 % hexamine was added to the solution as hardener. Furthermore, for the comparisons, a urea-formaldehyde resin (UF) from Methadynea with a solids content of 66 % and an ammonium sulphate solution were used as hardeners, and a sodium silicate from the Merk Group with a solids content of 50 % was used as the third binder. For all manufacturing tests of the laboratory boards, a proportion of 15 % per binder was used in relation to the absolutely dry (atro) mass of the particles (Krenn et al. 2017b).

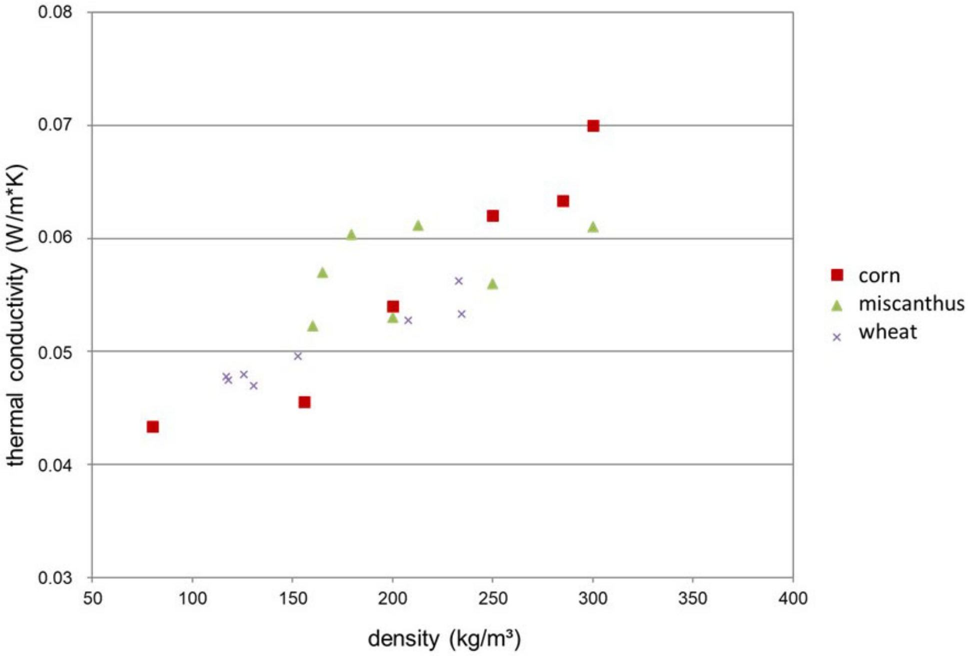

Figure 2.6: Thermal conductivity of the used panels subject to the density at a measurement temperature of 10 °C and a temperature difference of 15 K (Krenn et al. 2017b)

Figure 2.6 shows the thermal conductivity of the analysed boards as a function of the bulk density. The spans of the raw density from maize stalks (80 - 300 kg m^{-3}) or wheat straw (50 - 300 kg m^{-3}) have higher values than the boards from miscanthus (160 - 300 kg m^{-3}). This can be explained by the low bulk densities of the two materials (Krenn et al. 2017a; Nagl et al. 2015a;b). In addition, it can be observed a linear dependence of the thermal conductivity on the density of the different boards in the tested area. Dimensionally stable insulation boards could be produced with densities around 80 kg m^{-3} from maize stalks. For the boards based on other raw materials, this low bulk density could not be achieved. In addition to the favoured low thermal conductivity in a possible application as an insulation material, further material properties such as water absorption and fire behaviour were investigated. For these tests, were produced boards of 45 x 45 x 2 cm^3 with a bulk density of 250 kg m^{-3}, in order to ensure a sufficient comparability of the different raw materials. The Figure 2.7 shows the water up-take and release of the wheat straw board (250 kg m^{-3}) with different binding agents. After 24 hours the water absorption is 270 to 280 % when UF and water glass (WG) are used as binding agents. The water up-take value of boards with tannin binder (TA) in the same period is 250 %. The highest percentage of water inside the material was found in wheat straw panels with UF as binder. When considering the actual water up-take as a function of the sample area, the findings are put into perspective.

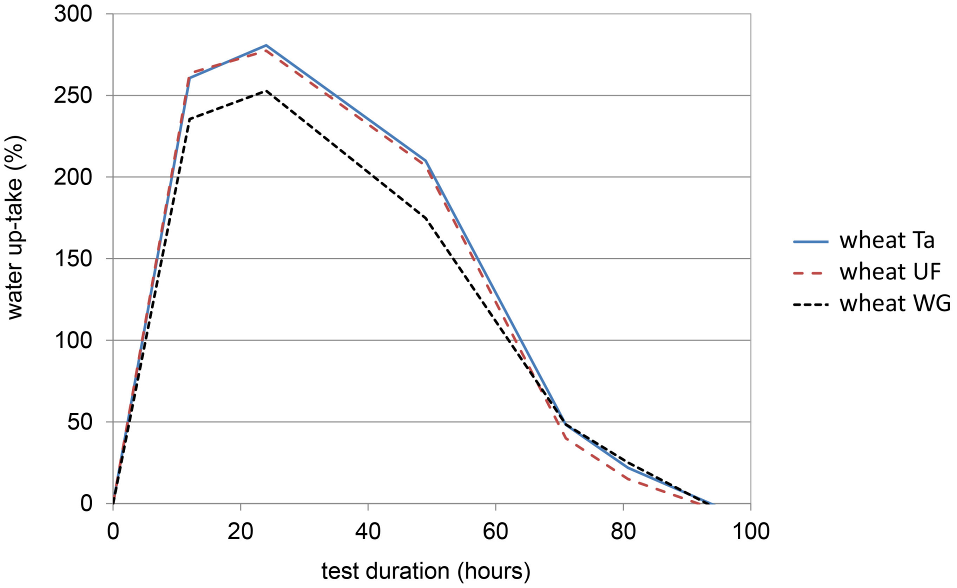

Figure 2.7: Influence of various binder systems of panels made of wheat straw on the water up-take and release (Krenn et al. 2017a)

The measured values of the plates with the water glass binder are 38.63 kg m^{-3} ahead of the samples produced with UF (39.53 kg m^{-3}) and tannin (44.52 kg m^{-3}). Values of this order of magnitude were able to be by Nagl et al. (2015a). After the 24-hour water storage, the samples were dried at 50 °C in a drying oven. After all straw insulation panels had a drying time of 69 hours until they had reached again their initial weight. Thereby, no significant differences in the drying times of the boards with different binding agents were determined. The water up-take and water release processes of the various boards are comparable. The binding agent used shows only a small influence on these properties. Figure 2.8 shows the water absorption and water release of various boards with regard to the raw materials maize stalks, miscanthus and wheat straw using a tannin glue system. The values of the total water up-take of the samples after water storage of 24 hours for the laboratory panels produced with the wheat 44.52 kg m^{-3}, maize 41.88 kg m^{-3} and miscanthus 25.77 kg m^{-3}. The maize stalks and wheat straw dried very irregularly off. Miscanthus rejected the slightest water absorption on. This behaviour was also confirmed by Nagl et al. (2015a) at the water absorption of loose miscanthus stem components was detected. The highest value was approx. 140 % (25.76 kg m^{-3}). Miscanthus also dried most evenly. The boards of miscanthus and corn stalks were dry after the water absorption and drying. The boards from wheat straw dissolve after testing the water up-take (Krenn et al. 2017a).

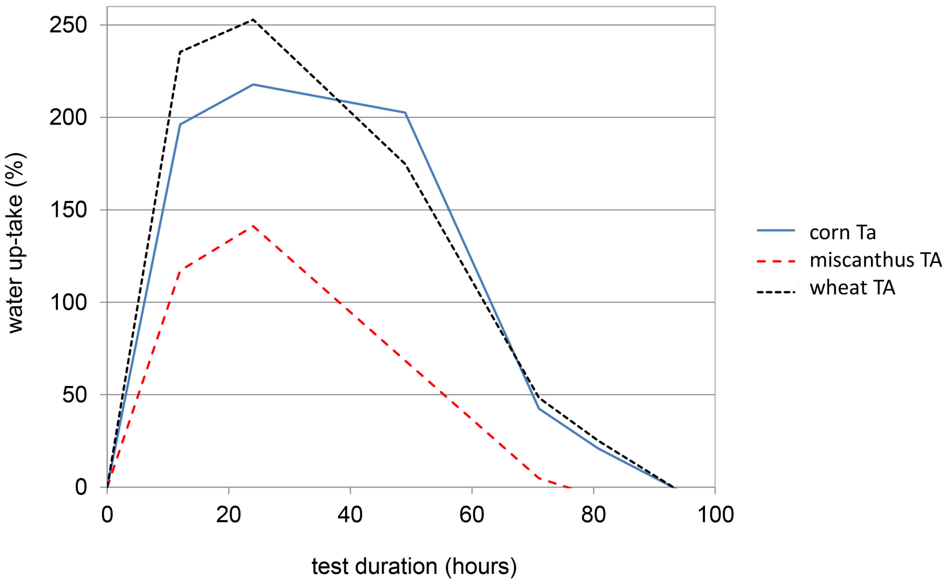

Figure 2.8: Influence of different insulation panels (250 kg m^{-3}) from corn stack, miscanthus and wheat straw with the binder tannin-hexamine on the water up-take and release (Krenn et al. 2017a)

2.2.2 Additional material properties of straw based insulation panels

In addition to water absorption, the poor fire behaviour is often for insulating materials made of natural raw materials as possible obstacle to the application. In the present study the fire behaviour of tannin-based, water glass based and UF glued insulation boards were examined. For the analysis of the fire behaviour of the boards a proper method had to be developed (Krenn et al. 2017a). Different samples sizes, devices and trail constructions were used for the pre-tests and the final test arrangement can be seen in Figure 2.9. The mass loss due to fire impact through a Bunsen burner was carried out and analysed. Here the variable loads of the equipment (e.g. Bunsen burners), which influence the weight measurement, were not taken up by the balance, but were derived by an external load transfer, so only the weight of the sample (40 mm x 40 mm) and its mounting on the balance was measured. Figure 2.10 shows the fire behaviour of wheat straw panels (250 kg m^{-3}), represented by the change in mass of the material associated with tannin (Ta), urea-formaldehyde (UF) and water glass (WG) bonded materials (Krenn et al. 2017b). After minor weight losses and stable values between 30 and 60 seconds there can be seen a constant weight reduction of the panels up to the defined testing after 5 min. The small decrease after 30 s can be attributed to the heating of the sample and evaporation (Kollmann and Côté 1968). The stagnating value up to the test time of 60 s is associated with the full evaporation of free and bound water because during this process, the temperature of this stage is not increased in the material and in a range of 102 °C ± 2 °C remains constant (Kollmann and Côté 1968). At this temperature there is no material degradation, but only loss of material by the dehydration of the material. In the further course of the test, the mass was continuously decreased by the thermal degradation. The wheat straw insulation board with tannin adhesive shows a higher mass loss after 5 min fire load than those with the two other adhesive types (UF and WG). The mean value at the end of the test for wheat straw slabs with tannin adhesive still amounts to 38.3 % of the initial mass.

Figure 2.9: Own experimental setup and detailed view to determine the mass loss due to fire (Krenn et al. 2017a)

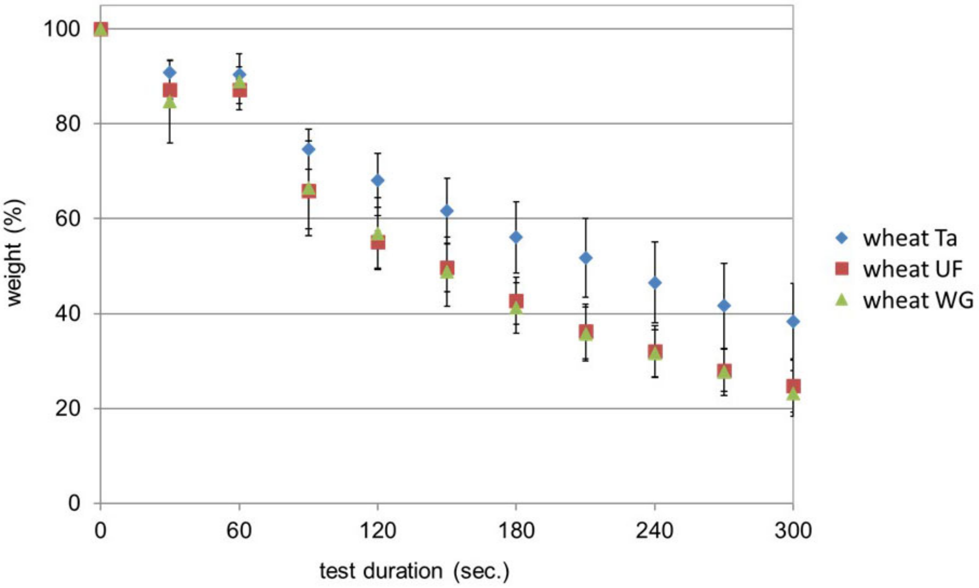

Figure 2.10: Mass loss of the panels of wheat straw (250 kg m^{-3}) with various binder system due to the effect of fire (Krenn et al. 2017b)

For panels with UF adhesive it is still 24.8 % of the initial mass and in the case of insulating panels with water glass it is 23.1 % of the initial weight. A similar course of the mass decrease could be observed for the samples made of the three used materials maize stalk, wheat straw and miscanthus and with the binding agent tannin (Ta) (Figure 2.11). At the end of the test period of 5 min, the mean value shall be of the maize stalk panels still 44.4 % of the initial mass. For wheat straw plates, the remaining final weight is still 38.3 % of the take-off weight and for the insulation boards from miscanthus it is 38.5 % of the initial mass.

Figure 2.12 shows the visual changes during a test of the different insulation boards at the end of the fire test. Two thirds of the maize stalk board burnt after the test. After 300 seconds, the underside glowed over its entire surface and was completely burnt. Only the leaves and stems of the maize plant were rudimentarily present. With the boards from miscanthus only more ashes and some intact stems were recognizable after the completion of the examinations. When the sample was taken, only individual fragments could be grasped by hand. This rapid combustion is due to the difficult bonding of the miscanthus. Due to the poor absorption or wetting of binding agents, the quality of bond is unstable due to the effects of fire and individual parts can easily become detached. The wheat straw panels were completely charred after completion of the test. Furthermore, some stalks came loose during the test. After completion of the tests, the specimens were no longer sufficiently bonded and disintegrated when the test device was removed.

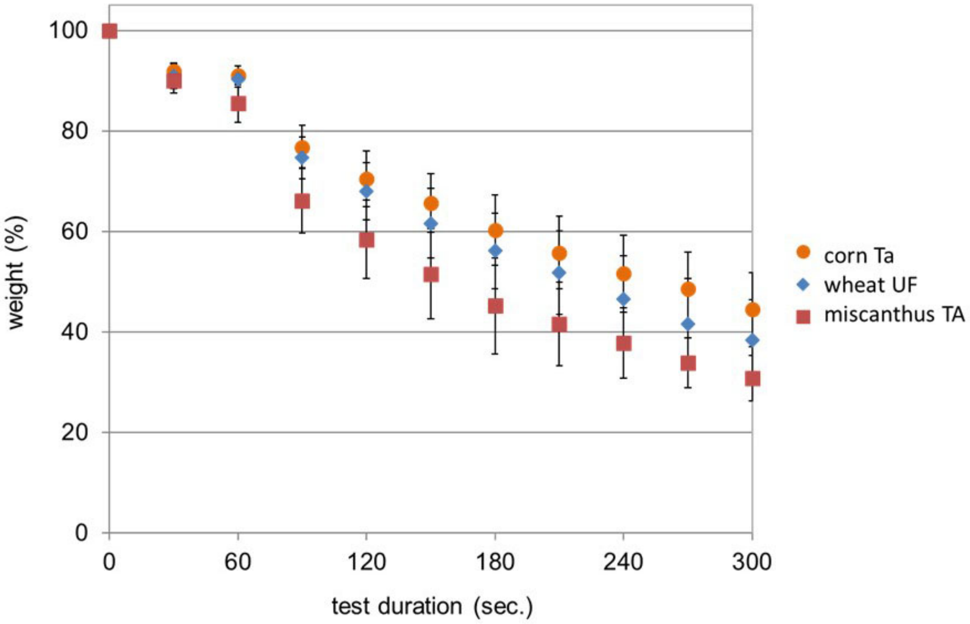

Figure 2.11: Mass loss of the various insulation boards (250 kg m^{-3}) due to the effect of fire (Krenn et al. 2017b)

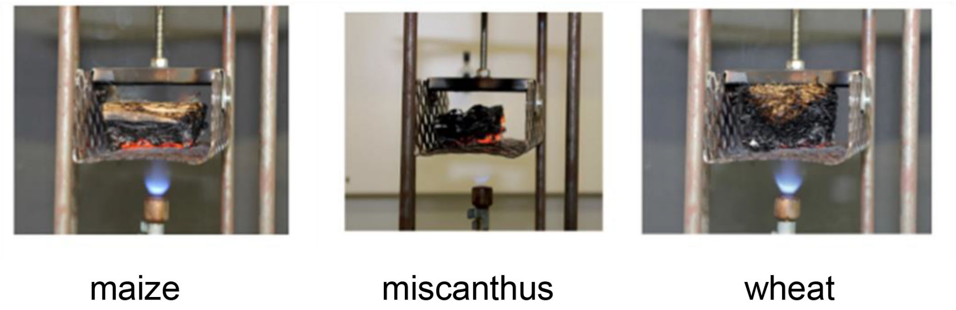

Figure 2.12: Visual observation of the changes of the insulation boards after the fire test (Krenn et al. 2017b)

2.3 Analysis of thermal-mechanical treated plants for insulation

This section deals with the influence of pre-treatment on the quality of annual plant particle/fibres for insulation materials. Even the insulation product can be a panel or/and a material for blowing insulation. The detailed results of the analysis of the both insulation products were not given before the starting point of the author’s research project BioInsPa in the year 2012 (Schnabel et al. 2016b).

To improve the bonding between the gluing system and different plant materials species various treatments can be performed using methods of chemical, enzymatic and thermal-hydro activations by destruction of the wax layer and increase of the accessible surface area of the material. Tumuluru (2018) give a detail overview about various treatments of annual plants fibres. The steam pressurized treatment modified the chemical composition, sorption behaviour and mechanical properties of wheat straw fibres (Nagl et al. 2015a; Schnabel et al. 2019d). For insulation materials the steam exploded straw fibres were produced by using a batch system in experimental laboratory scale set-ups (Huber et al. 2015).

2.3.1 Changes in material structure

Steam explosion process is widely used for the pre-treatment of plant biomass (Schnabel et al. 2019d). The performance of steam explosion process depends on temperature (indirectly the steam pressure), retention time and particle size of the materials. For the preliminary study, the biomass particle was put into a small vessel, pressurized and heated with steam under different temperature conditions (e.g. 160, 180 and 200

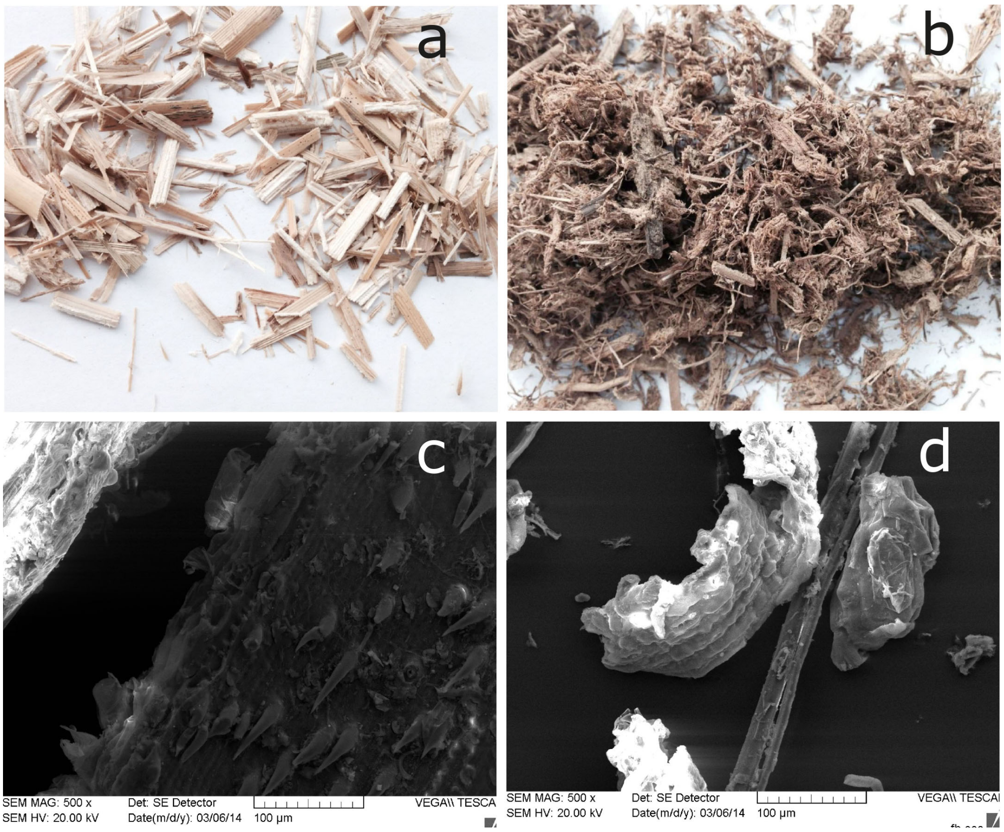

Figure 2.13: Raw materials of miscanthus with different treatments: a) chopped and b) steam pressurised at the macro level, respectively as well as c) chopped and d) steam exploded material at the micro level by analysing a scanning electron microscope, respectively (Schnabel et al. 2019d)

°C) and various processing times (e.g. 5, 10 and 20 min). After that the steam was quickly released and the lignocellulosic particles exploded at atmospheric pressure into fibres due to the weaker bonds a rupture of the biomass fibres rigid structure. The sudden pressure release of the wet particles defibrillates the cell complex of the lignocellulosic materials. Also, an incomplete disruption of lignin-carbohydrate-complex happens, whereas the hemicelluloses are easily soluble and the pulp remains (c.f. Figure 2.13 and Figure 2.14).

The results from this pilot studies showed that the size of the straw particles tend to be smaller at higher temperature and longer retention time as well as the dust (<250 μm particle size) was increased, respectively. However, the amount of fibres was found in the sample treated with higher temperature and longer retention time. Therefore, the materials used for the panel production and blowing insulation were steam pressurized at 200 °C temperature and 20 minutes retention time (Huber et al. 2015). The results of the cooking degree can be observed using the optical characteristics of the different treated raw materials. The steam explosion influences of the material on fibre and fibrils levels were shown in Figure 2.13. Neverthe-

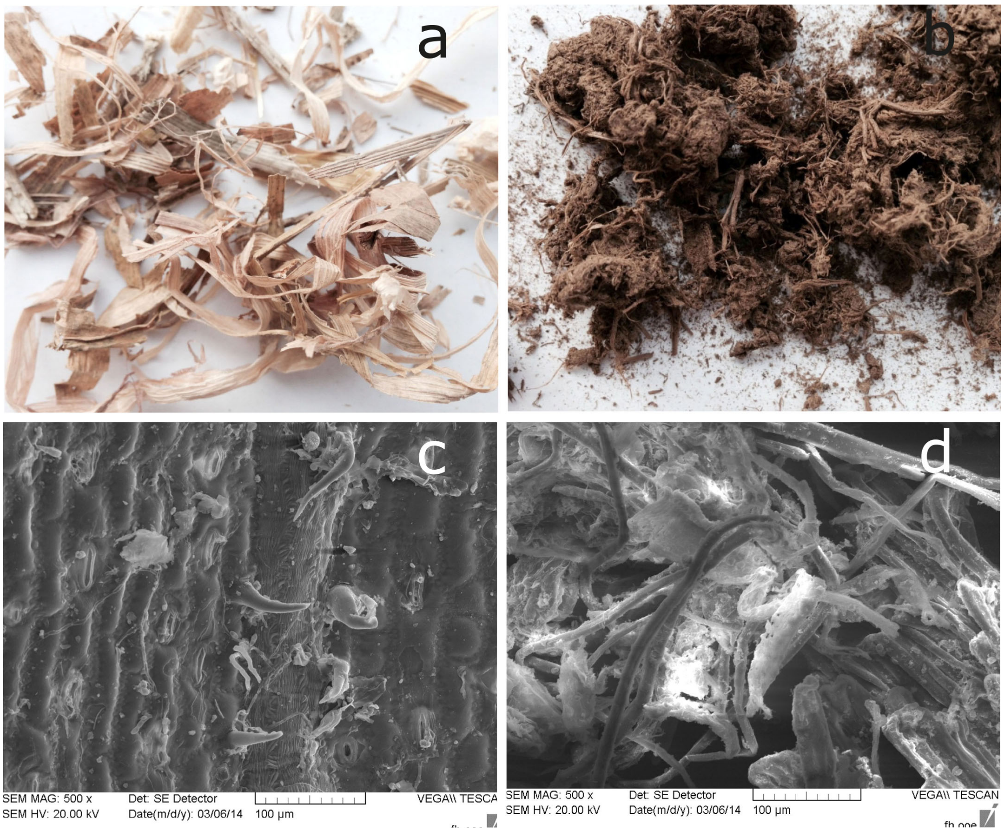

Figure 2.14: Raw materials of maize with different treatments: a) chopped and b) steam pressurised at the macro level, respectively as well as c) chopped and d) steam exploded material at the micro level by analysing a scanning electron microscope, respectively (Schnabel et al. 2019d)

less, the wax surface of the various straw species was also destroyed due to the steam explosion process. These observations can be also seen for the steam pressurized plant materials of maize in Figure 2.14. However, the analysis at the macro level depicted that the particle size distributions between the natural and untreated materials are different. Due to the treatment the size of the particle shifts to smaller dimensions compared to the raw material (Nagl et al. 2015c; Krenn et al. 2017a). The amount of material fractions below 250 μm were almost zero for the chopped raw materials. By contrast the treated materials show an amount of 8 % for the same fraction (below 250 μm). Most of this fraction is dust and cannot be used for the development of insulating materials as well as this material is lost for the insulation material. However, this fraction has a high impact on the bulk density. In the range between 250 μm and below 1.0 mm the amount of particles was also lower for the chopped samples than for the steam pressurised samples (Figure 2.15).

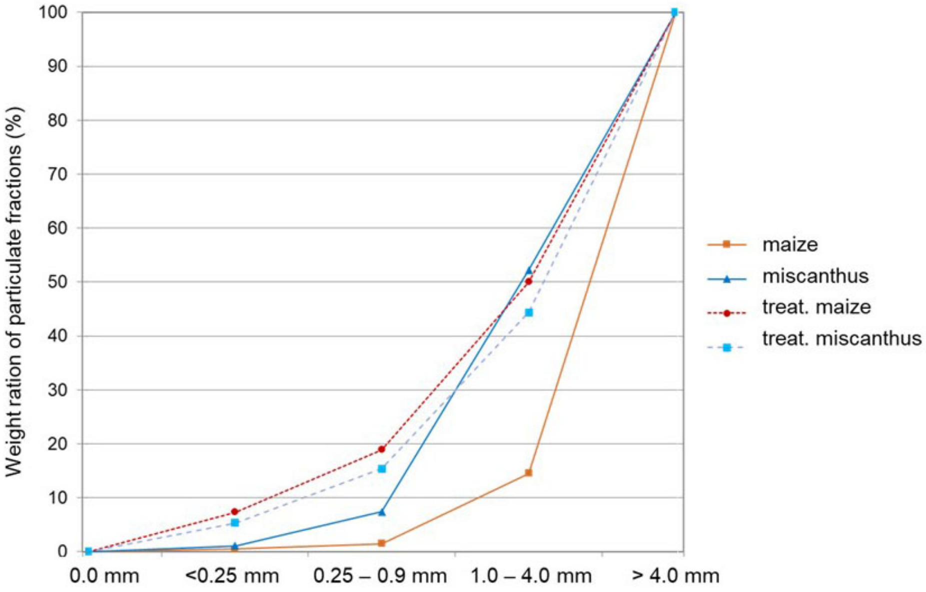

Figure 2.15: Particle size distribution regarding the different raw materials and treatments (Schnabel et al. 2019d)

The sieving results for the particle fraction between 1.0 and 4.0 mm depict that the relative amount of copped miscanthus, steam treated maize and miscanthus have a range of 29 to 45 %. Only the chopped maize samples have very low amount in this class, which laid around 13 %. This behaviour can be explained by the structure of the chopped maize stalks. The samples from the maize had a higher portion of leaves than stem material, whereas the miscanthus samples presented a high amount of stem material (cf. Figure 2.13 and Figure 2.14).

The relative amounts of the sieve fraction larger than 4 mm particle size showed different results. The values from the copped maize material showed the highest amount about 86 %, which is followed by the steam pressurised miscanthus samples with 56 % and treated maize with 50 %. The lowest values presented the chopped miscanthus with 48 % of the relative portion. Therefore, some amount of the material is lost for the production of insulations. Particle size distribution was determined using a shaking- or vibrating procedure (Nagl et al. 2015a). The particles were graded in decreasing size classes in the automatic sieve AS 200 (Retsch) within 15 minutes shaking time and an amplitude of 50.

2.3.2 Changes in chemical properties

Each different temperature and retention time of the steam pressured processes result in different values. The higher the temperature the higher is the digestion of the plant materials. Also, the retention time plays an important role the changes in the chemistry and structure of the different materials. Table 2.1 showed the changes in chemical composition based on the steam explosion process.

Table 2.1: Results of the chemical characterisation of different raw particles (Huber et al. 2015; Schnabel et al. 2019d)

	Tp. (°C)	Retention time (min.)	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Rest (%)
Miscanthus	0	0	63	20	12	5
Miscanthus	180	10	58	15	13	14
Miscanthus	200	10	50	7	16	27
Maize	0	0	39	31	12	18
Maize	180	10	34	18	15	33
Maize	200	10	44	5	9	42
Wheat	0	0	45	25	19	11
Wheat	160	20	46	20	17	17
Wheat	180	10	40	20	10	30
Wheat	180	20	43	20	12	25
Wheat	200	20	54	2	12	32
Hemp	0	0	40	34	14	12
Hemp	200	20	46	1	11	42

The chemical changes of the different materials due to the stream explosion process were analysed by using FT-IR spectroscopy (Figure 2.16). The chemical changes of the plant components due to the steam explosion occurred mainly in holocellulose and in lignin and this was also the first impression of the chemical changes caused by steamed pressurized treatments (Schnabel et al. 2019d). A difference between the IR spectra of treated and untreated miscanthus and maize samples were observed at the wavenumbers in the area about 2910 cm^{-1}. The peaks obtained at 2930 cm^{-1} and 2890 cm^{-1} are an indication of stretching vibration form CH, CH_2 and CH_3 (Pretsch et al. 2010). These functional groups can correspond with the wax on the raw materials. Miscanthus samples showed a clear form of the two bands. After the steam pressurised treatments, the heights of the flanks were decreased and clear form of the peaks cannot be observed anymore. The peak at around 1730 cm^{-1} is an initiation of carbonyl groups or COOH-groups, which may correspond with the hemicellulose and fatty acids of the natural wax (Schnabel et al. 2019c) and has an influence on the wettability of the material (Huber et al. 2019). At around 1510 cm^{-1} a peak can be observed, which are corresponding to the aromatic molecules (Schnabel and Huber 2014; Schnabel et al. 2014). This peak can be increased due to the treatment based on the exploration process of the

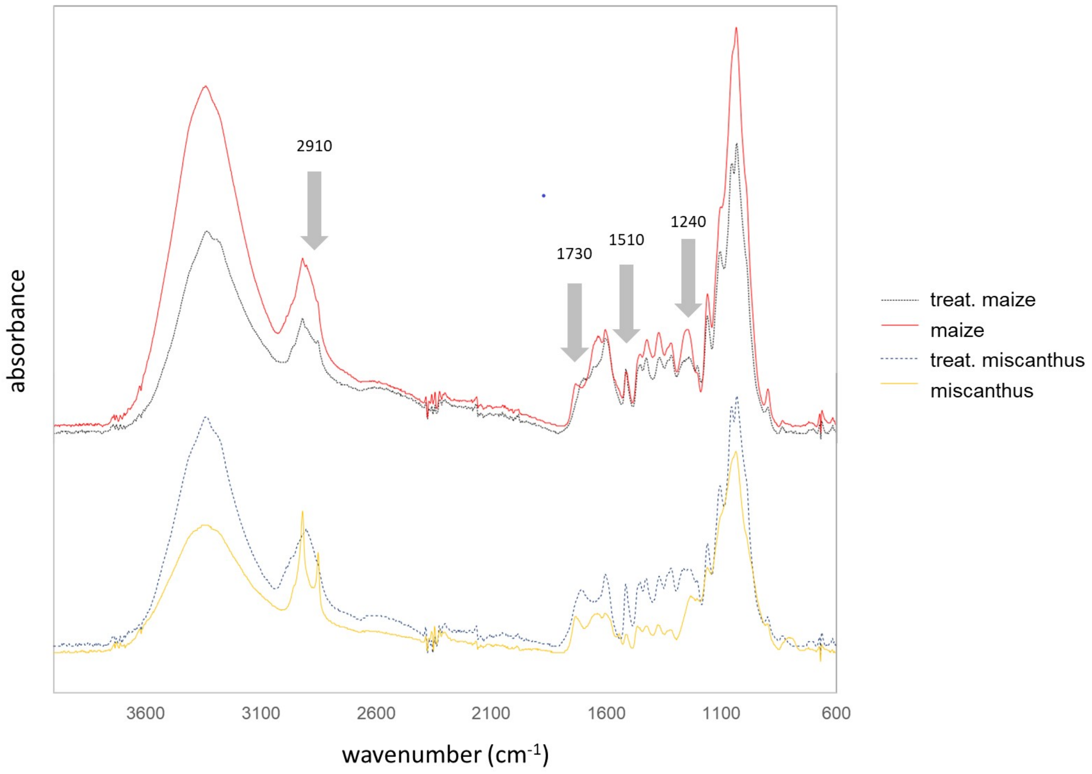

Figure 2.16: FT-IR spectra of treated and untreated maize as well as miscanthus, respectively (Schnabel et al. 2019d)

miscanthus materials. The band around 1240 cm^{-1} was confirmed to the stretching vibration of C=O and COOH groups of aromatic compounds and hemicellulose (Ebner et al. 2014; Petutschnigg et al. 2013). Based on these results, it can be assumed that the hemicellulose was degraded due to the steam explosion process. Besides the chemical modification the changes in structure are important for the application for the use as insulating materials (Schnabel et al. 2019a).

2.3.3 Changes in physical materials properties

Table 2.2 shows that the bulk densities of the references and stream pressurised lignocellulosic materials are different. Due to the treatment, the bulk density of the maize samples increased. This phenomenon results due to a relative high amount of dust in the materials compared to the untreated maize material (c.f. Figure 2.15). For the miscanthus material a contrary change can be observed. The bulk density decreases due to the effect of the steam explosion process through the increase of material fraction in the range between 1.0 to 4.0 mm.

Table 2.2: Overview of different material properties of chopped and stream pressurised materials at 200 °C (Schnabel et al. 2019d)

	Tp. (°C)	Retention time (min.)	Bulk density (kg m^{-3})	Moisture content (%)
Maize	0	0	48.6	9.5
Maize	200	10	82.7	6.1
Miscanthus	0	0	137.6	10.1
Miscanthus	200	10	93.4	5.7

Measurements of the thermal conductivity at 10 °C of chopped and loose stalk treated maize showed that there is only a small difference between both materials, as bulk densities are very similar (Table 2.3). Separating the raw materials into different fractions show different results of the thermal conductivity. The larger the particle size the higher are the values of the thermal conductivity. The measurement area can compactly fill in with small particles. This phenomenon can be seen if the bulk density in the measurement area is compared (Schnabel et al. 2019d). Higher densities represented higher amount of material and less air inside the measurement field.

Table 2.3: Results from the thermal conductivity measurements of loose maize samples at 10 °C temperature (Schnabel et al. 2019d)

	Tp. (°C)	Retention time (min.)	Particle size (mm)	Bulk density (kg m^{-3})	Thermal conductivity (W mK^{-1})
Maize	0	0	org.	98.0	0.04600
Maize	200	20	org.	100.8	0.04573
Maize	200	20	>4	80.0	0.04626
Maize	200	20	<4 - 2	86.2	0.04661
Maize	200	20	<2 - 1	94.1	0.04362
Maize	200	20	<1 - 0.25	106.6	0.04277

Table 2.4 shows the thermal conductivity at 10 °C of the loose miscanthus samples. However, the high bulk density in the measurement area of chopped miscanthus material of 151.9 kg m^{-3}, which could not obtain with the steam pressurised materials. The results show that treated material samples have a lower thermal conductivity compared to the chopped miscanthus material. At a bulk density of 100.3 kg m^{-3} a thermal conductivity value of 0.04546 (W mK^{-1}) was determined and this value was lower than the measured value from the treated miscanthus materials with 120.0 kg m^{-3} bulk density. The low values in thermal conductivity and bulk densities are affected from the low moisture content of the steam pressurised material samples (Schnabel et al. 2019d).

Table 2.4: Results from the thermal conductivity measurements of loose miscanthus samples at 10 °C temperature (Schnabel et al. 2019d)

	Tp. (°C)	Retention time (min.)	Particle size (mm)	Bulk density (kg m^{-3})	Thermal conductivity (W mK^{-1})
Miscanthus	0	0	org.	151.9	0.05147
Miscanthus	200	20	org.	120.0	0.04726
Miscanthus	200	20	org.	100.3	0.046546
Miscanthus	200	20	<4 - 2	98.1	0.047251
Miscanthus	200	20	<2 - 1	79.1	0.04484
Miscanthus	200	20	<1 - 0.25	96.0	0.04286

Measurements of the thermal conductivity of the fractionated treated materials showed a clear trend. With decreasing particle size of the material, the values of thermal conductivity are getting lower to 0.04286 (W mK^{-1}) (Schnabel et al. 2019d). However, this behaviour is not related with the bulk density as measuring the thermal conductivity with the maize material. Thermal conductivity measurements with larger particle size than 4 mm could not be conducted due to the insufficient coverage of the test area.

Furthermore, the treated lignocellulosic materials were used for the production of fibre boards with UF glue and the thermal conductivity was measured again. The production of the insulation boards took place without difficulties and the particles from all screen fractions were used. However, not all insulation boards could be produced with the same density, as this not only depends on the individual properties of the fibres (e.g. fibre geometry or fineness), but is also influenced by the respective material bulk densities. All materials show a dependence of thermal conductivity on board density over the investigated density range (Figure 2.17). It was found that the panel densities with the treated materials had higher values than with the reference materials. This was due to the increase in the fine fraction caused by the treatment.

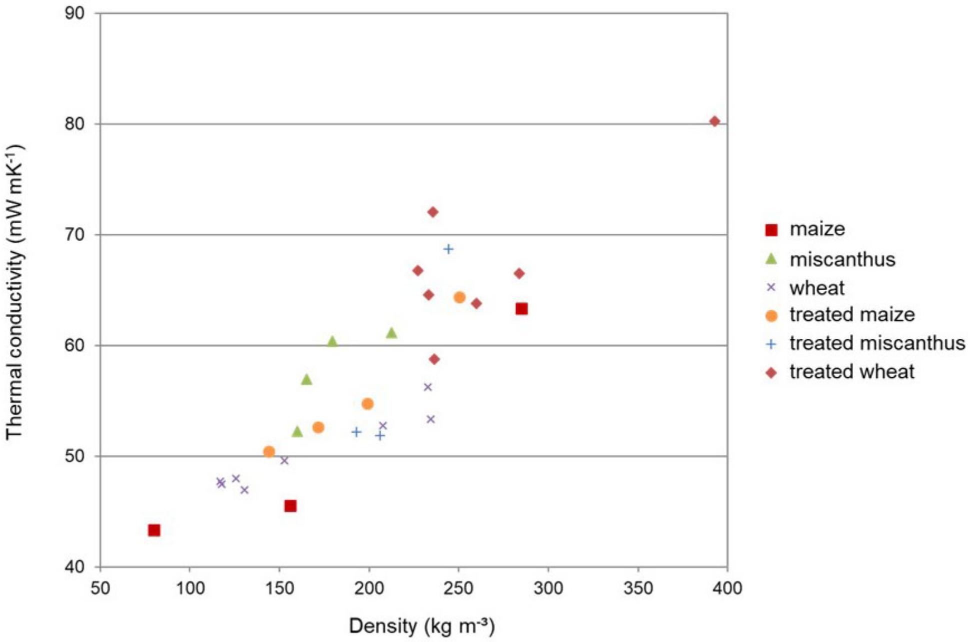

Figure 2.17: Thermal conductivity of the investigated insulation boards as a function of the density at a measuring temperature of 10 °C and a temperature difference of the boards of 15 K

2.4 Product development and applications

Based on the results from the previous sections, new fields of application for the different types of stalks and the pre-treated materials were identified. The analysed materials should be used for timber frame constructions (e.g. intermediate wall as sound insulation and exterior wall as heat insulation). Systemically, these are lightweight components with inhomogeneous, multi-layered construction.

The two different products (panel insulation and blow-in material) were also taken into account in the study from Schnabel et al. (2019a). In order to ensure a simple application of the insulation, material development was oriented towards existing products, and so it was possible to achieve through appropriate properties that the panel insulation and the blow-in insulation could be easily integrated into the existing concepts of wall structures (cf. www.dataholz.at).

Various stalks particles of different plants were glued with a urea-formaldehyde resin, a sodium water glass and a tannin-hexamine binder and pressed to light panels (density <300 kg m^{-3}) using a lab equipment. In addition to the thermal conductivity of the boards, the particle distribution, the water up-take or release as well as the fire behaviour of the different starting materials as well as of the resulting their panels/plates were examined. The measured thermal conductivity of all panels was in the range of other natural insulation materials on the market although there were no additives included to improve certain insulation properties. The influence of the different adhesive systems plays only a subordinate role in the water up-take. However, differences in adhesives can be determined by the fire behaviour test. Here, the tannin-hexamine binder shows advantages over the two other systems.

Steam explosion treatment were used to refine the straw materials of maize and miscanthus. These particles and fibres were analysed for the applicability as insulating material (e.g. blow-in insulation). The particle size of the steam pressurised samples decreases compared to the reference samples due to treatment of the natural fibre. Another positive effect is the lower moisture content of the treated fibres compared to the raw material. Furthermore, the use of steam pressurised materials has beneficial effects on the bulk densities and thermal conductivity by using fractionated materials.

2.4.1 Developed materials

Due to the different treatments and their influence on the properties, different materials can be used for further product development (Figure 2.18). These findings provide a basis for help in transfer from laboratory to industrial conditions for consumer applications. As the treatment intensity increases, the digestion of the plant fibres or particles also increases the application possibilities of the material. For example, the untreated stalks of different plants with the lowest mechanical processing in the field cannot be used as fibre raw material for insulation boards or blow-in insulation without further processing. However, it is also possible to use thermo-mechanically defibred material (e.g. steam explosion) for the production of fibreboard and/or blow-in insulation. The research also showed that the higher the treatment intensity of the analysed samples was the higher the fine fraction and the resulting material loss. In most cases, the fine fraction must be reduced by suitable methods in order to manufacture products or materials, which is one additional process with extra process costs. However, the dust may use for energy generation. In general, straw can be used to manufacture a wide range of products and materials for the use of different insulation materials. Current research suggests that straw can be used to insulate buildings.

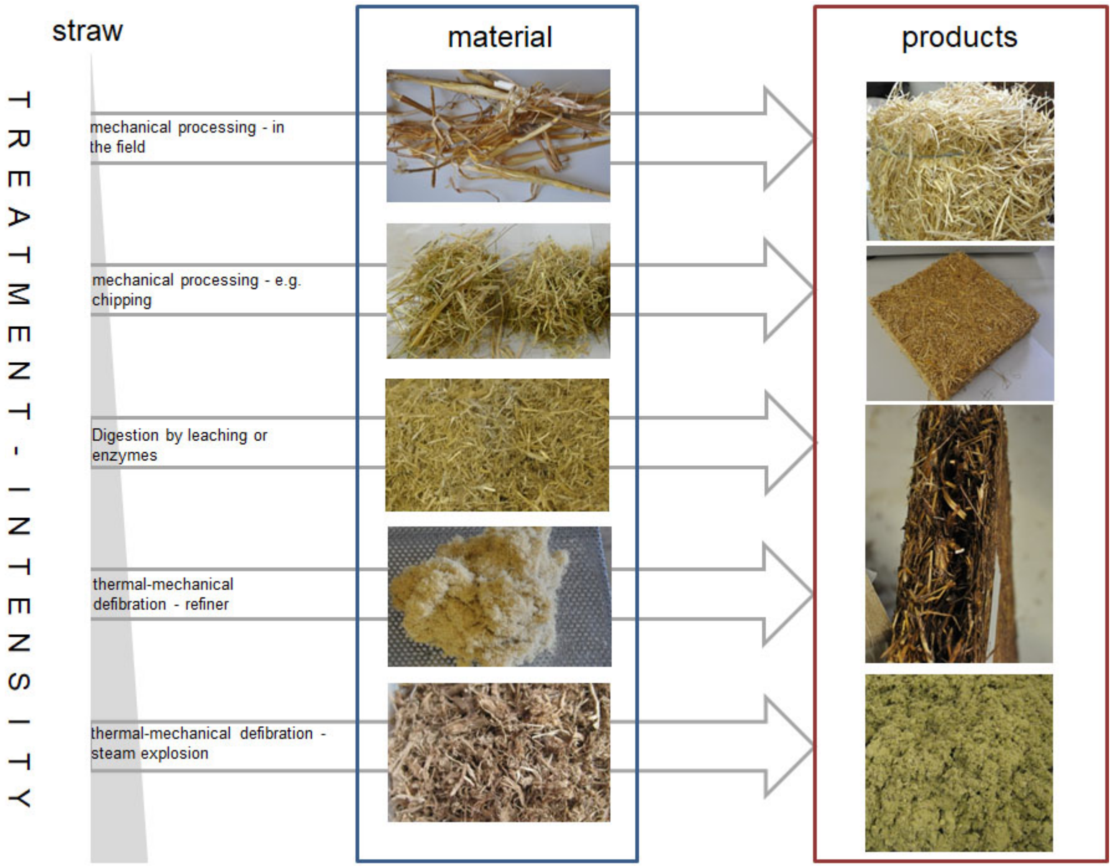

Figure 2.18: Overview of the feedstock and their possible products made of lignocellulosic materials (Schnabel et al. 2020b)

2.4.2 Presentation of different products and applications

Different material properties can be achieved depending on the raw material (e.g. wheat, rye, and miscanthus), pre-treatment (e.g. mulched, and chopped), adhesive (e.g. tannin, UF, and water glass) and bulk density of the boards (Schnabel et al. 2019a). These influence the product concepts for outdoor or indoor use. Due to the achieved material strength, the boards can be fastened to the façade with commercially available screws and/or plugs for insulating materials (Figure 2.19).

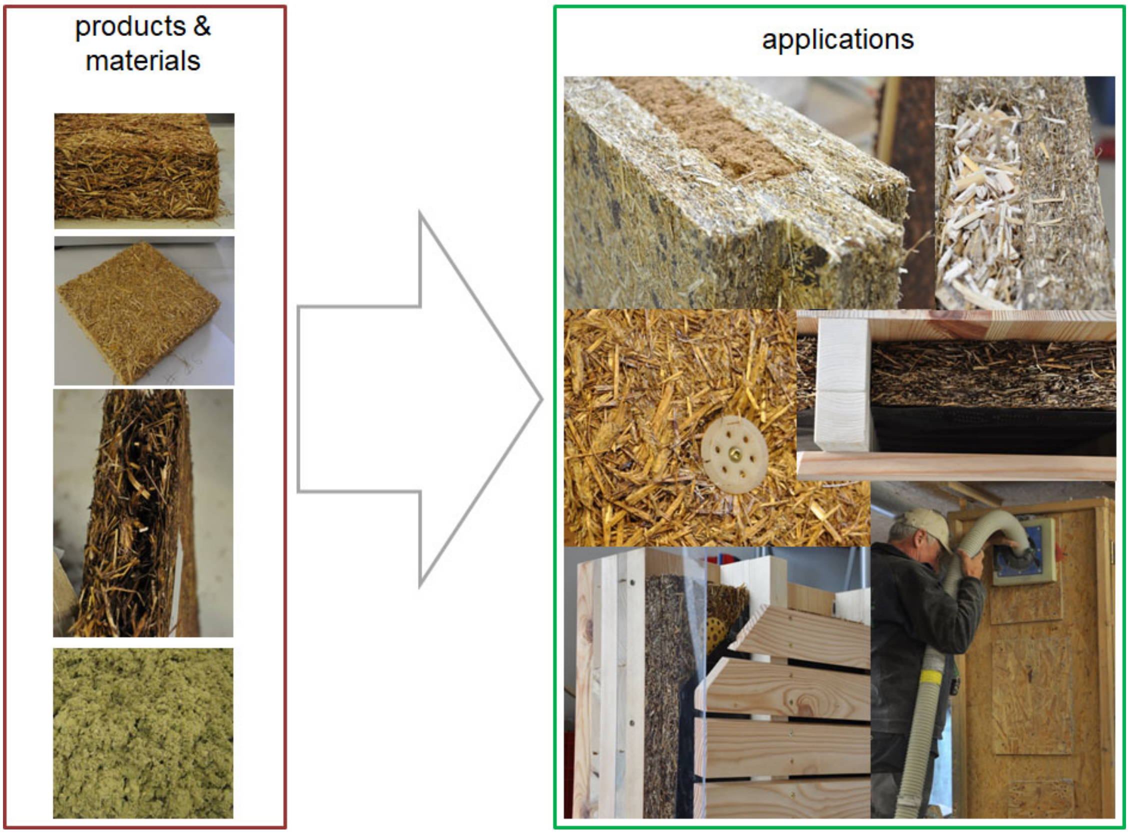

Figure 2.19: Overview of possible feedstock and products made of lignocellulosic materials as well as their applications (Schnabel et al. 2020b)

The first blowing-in tests showed very good technological material values of the defibred straw with regard to density, thermal conductivity and injection behaviour. Therefore, the existing injection systems and wall structures can be used for the most part for the straw material. The raw density values achieved in the blow-in tests with the defibred straw showed comparable values as with cellulose or wood fibre blow-in insulation and should therefore also provide the necessary settlement safety. However, long-term investigations on the settlement safety of the material still had to be carried out. A further research approach is that the density of blow-in insulation is higher than 120 kg m^{-3} to guarantee the settlement safety of the straw bulk insulation, which is a guideline of tradespeople (Schnabel et al. 2019a). However, further tests are needed to show the real settlement of straw bulk insulation materials.

The individual material and product developments were evaluated with regard to the technical and economic areas and examined for industrial feasibility. The collection of the process data for the individual pre-treatments was carried out on the basis of the available literature. Since the pre-treatments in this project were carried out with laboratory equipment, these data are not representative and cannot be used alone for an objective consideration. Depending on the material pre-treatment, different insulating materials (e.g. boards or blow-in insulation) with different property values can be produced (Schnabel et al. 2019b). Nevertheless, also other parameters have to be considered. The higher the mechanical and thermomechanical process of the straw material pre-treatment is the higher the consumption of energy. Furthermore, natural adhesive systems (e.g. tannin, a natural ingredient from e.g. tree bark) with very good bonding properties were successfully used in the production of the insulating materials and thus include an important environmental aspect in the production and disposal of boards.

3 Up-cycling of residuals for leather-wood composite materials[*]

The up-cycling of alleged residues from industrial production processes may provide a sustainable way to raw material procurement. A look at the resources of other different industries is sometimes rewarding and is one main process to establish a new value chain based on the bio-economy approach. This is achieved by underlined a number of different examples. According to the current market situation in Europe and an increasing trend of using energy that is gain by burning any kind of biomass (Barbu et al. 2014a;b), a wealth of investigations according to possible new material resources had been conducted. Most of these investigations were dealing with an up- or recycling of by-products (Winandy et al. 2007). A variety of different board types and panel systems with specific properties and different features are established to the market so far (Dunky and Niemz 2002). To obtain a new and innovative range of products for the future, unusual ways need to be considered to achieve this goal with sustainable methods (Barbu et al. 2014b; Paulitsch and Barbu 2015). One of the possible options is to develop a new source of raw material and combine its positive properties with those of wood (Ashby 1999).

In general, two processes can be identified and distinguished for the development and/or optimization of wood-based panels. On the one hand, the use of lightweight construction or the introduction of structures into the material is possible for the enhancement of the properties (Ashby 1999). On the other hand, unused bio-based resources can substitute previous materials. The so-called "up-cycling" of residual materials has a long tradition in wood-based materials research, has continued to grow in recent years and should continue to be pushed forward.

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[*] The author managed and did the research in the development and characterisation of wood-leather fibreboards between 2010 and 2015 within three research projects with four national and international partners. These research results were presented in 5 scientific articles and in 8 international conferences. The author contributed to the funding acquisition, design and implementation of the research, to the analysis of the results and to the writing of the manuscript of these articles.

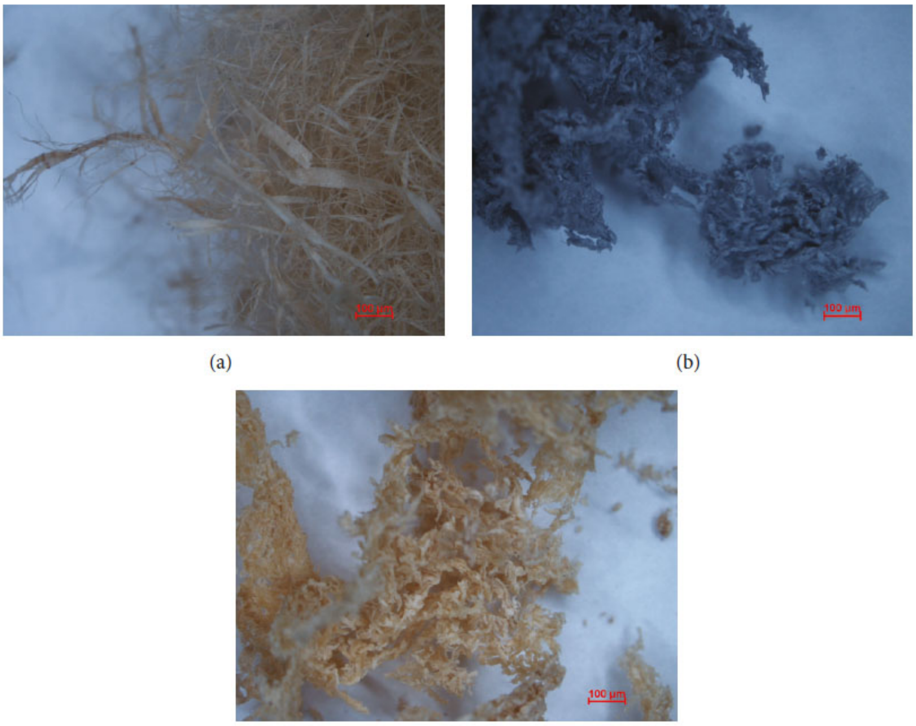
3.1 Structural description of the leather particles and wood fibres

Leather-wood fibreboards are new composite materials (Wieland et al. 2010b) and this combination of both materials allows the improvement of the performances of the final product (Schnabel et al. 2019c). The combination of wood and leather changes the properties of both different bio-based resources, resulting in improved composite material, which was shown by the results of the two research projects entitled 3d-LeFaShape and FLAME (Rindler et al. 2015; Schnabel et al. 2019c). Leather-wood fibreboards are innovative composite materials (Wieland et al. 2010b), which combine together the high mechanical properties of wood (Grünewald et al. 2013; Rindler 2014; Rindler et al. 2015; Solt 2014; Solt et al. 2015) and the superior fire behaviour properties of leather (Schnabel et al. 2019c). This may have a direct impact on the forest products industries and further applications. With new material properties the number of wood-based panels can increase and the innovative boards can broaden the applications. The up-cycling of potential residues from industrial production cycles allows sustainable methods of raw materials procurement to be developed according to the bio-economy approach.

The leather and tannery industry produces a high amount of waste materials, e.g. 200,000 tons per year in Europe (Grünewald et al. 2013; Joseph and Nithya 2008). The majority of these by-products will be currently used for thermal purposes in Central Europe and the USA or deposited in landfills (Cabeza et al. 1998; Wieland et al. 2010a). In Figure 3.1 is shown the material from the leather tannery process (wet white (ww) and wet blue (wb)). When leather gets produced, hides have to run through different production steps (Rindler et al. 2015). After the withdrawal of the skin a preservation process has to be done to protect the freshly peeled skins against the influence of microorganisms. The next step, the tanning process, is used to protect the skin against enzymatic degradation and increase its resilience. Only after this production step the skins are called leather. These particles used are leather fold chips and are produced by levelling the leather before it is further processed. The leather shavings used here have similar technological properties

Figure 3.1: Resource of the composite materials (Solt et al. 2015)

to the end product leather (Solt et al. 2015). In this investigation were used two types of leather, namely wet blue and wet white, were used (Figure 3.2). The wood fibres have structural differences compared to wet-white and wet-blue leathers, on the one hand the wood fibres are longer lighter and are sporadically present. On the other hand, the leather particles are more compact and form a loop similar to a ball of wool. Therefore, the two types of leather have a higher bulk density compared to the wood fibres and this has drawbacks by the gluing of pure leather particle without any amount of wood fibres, as the air flow does not transport the particles further and they clog the whole system as a result.



(a) (b)

(c)

Figure 3.2: Resources to produce the composite materials: a) wood fibres, b) leather shavings wet blue, and c) leather shavings wet white (Wagner et al. 2015)

3.2 Raw material characterization

The haptic and sensory complaints to these particles cannot lead to a classification of these products as durable. These smart composite materials combine two different raw materials (e.g., wood and leather). Spectroscopic methods gain increasing interest in the last decade not only for the analysis of wood-based panels, but also for the on-line manufacturing control (Tondi et al. 2015; Wagner et al. 2015). In order to be able to build meaningful models for online control, a good understanding of data distribution and the interaction of the respective constituents has to be developed in the lab beforehand.

3.2.1 FT-IR spectroscopy

The fingerprint region (1800 - 800 cm^{-1}) of the FT-IR spectra is reported in Figure 3.3. The analyse of the IR signals show high vibrational absorbance differences between the two materials of wood and leather shavings (wet-blue particles). In particular the bands at 1630 (C=O stretching - amide I), 1540 (N-H bending - amide II), and 1448 cm^{-1} (C-N. C-C, C-H amide II) can be easily used for highlighting the chemical differences between the components (Tondi et al. 2015).

This information of these three bands at the various wavenumbers are important for allowing the transformation of the two-dimensional mapping of the wood-leather fibre from optical surface image into its chemical image (cf. Figure 3.4). The two-dimensional mapping (Figure 3.4) presents a good agreement between optical and chemical information. It can be seen that the blue colour spots of leather in the visible image are represented in the chemical image as red colour spots with different intensities according to the portion of leather and fibre in every punctual scan with the ATR unit of the microscope (Tondi et al.

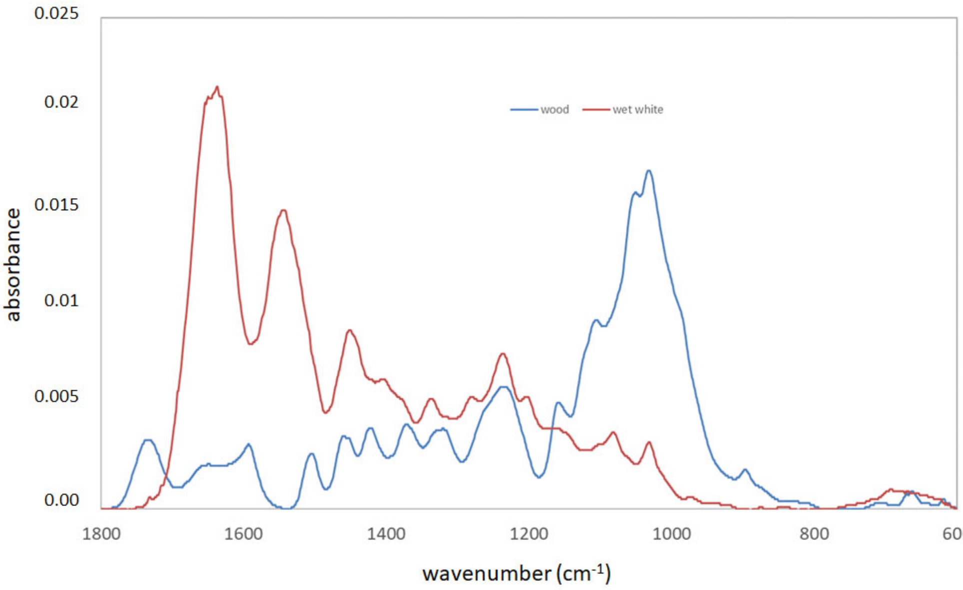


Figure 3.3: ATR FT-IR spectra of wood fibre and leather shavings of wet white

2015). The chemical mapping image shows a gradient of the different material boarders while the optical image appears clear ones. Based on the fact that the information is from punctual measurements, which can contain both constituents in one scan area, and the signal intensity gives a mean from both materials. Although the chemical information obtained demonstrates that it is possible to recognize the components in the composite materials. It is worth noting that identification of the constituents was obtained with only 16 scans, which promotes this technique as an interesting quality control system for the surface properties of the leather fibre panels (Tondi et al. 2015). This method shows great potential for chemical investigation of the surface of composite materials.

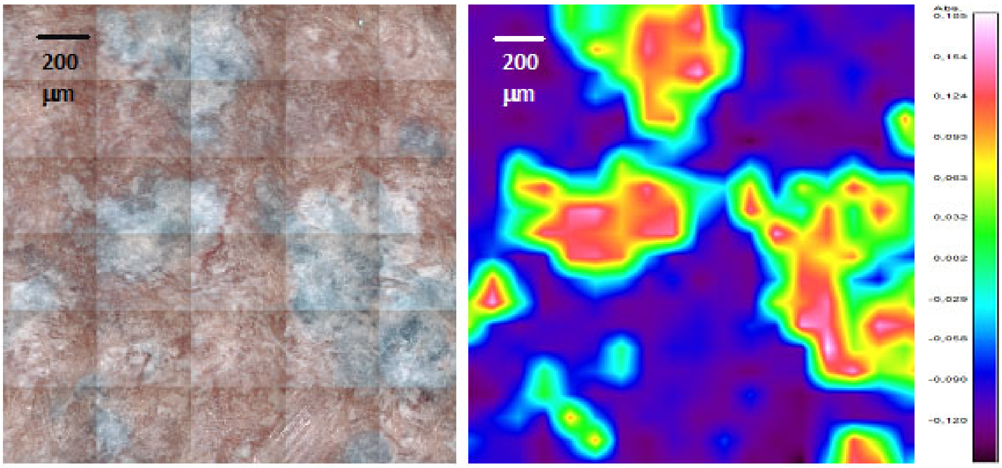


Figure 3.4: Images from the Perkin-Elmer microscope: a) optical image, b) ATR FT-IR chemical mapping (Tondi et al. 2015)

3.2.2 FT-NIR spectroscopy

Consequently, the results of FT-NIR spectroscopy showed also the potential for speeding up the industrial quality control (Wagner et al. 2015). The chemical information relating to the two different milled leather powders and the milled spruce wood powder was obtained by using the FT-NIR spectroscopy equipped with a fibre probe (Wagner et al. 2015). Figure 3.5 shows the spectra in the region between the wavenumber range 9000 - 4000 cm^{-1} of the wood fibres and the wet white (ww) and the wet blue (wb) leather powders. The differences between the wood fibres spectrum and the two different leather particles spectra can be seen. The bands around the wavenumbers 6660, 4886, and 4587 cm^{-1} corresponded with the structure of proteins, here especially the first overtone of N-H stretching, the single or combination of amide I or amide II, and the second overtone of N-H bending vibrations (Shenk et al. 2001). Also, the second overtone of O-H

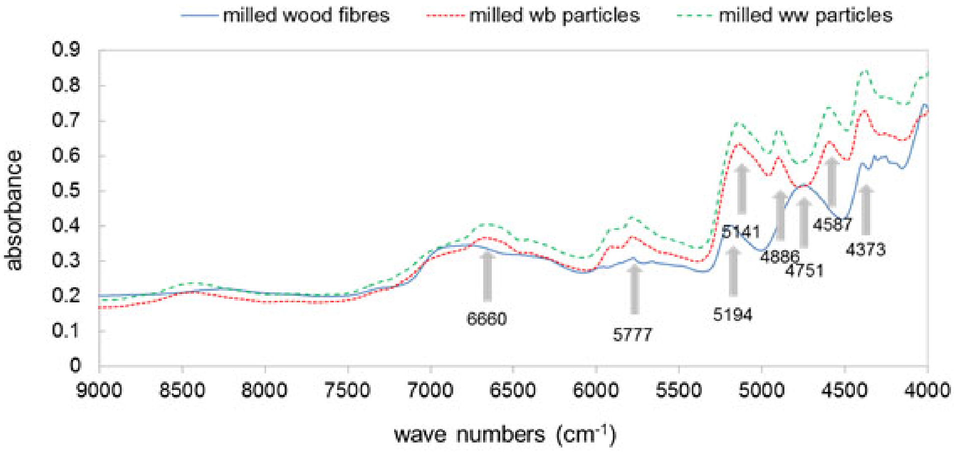


Figure 3.5: FT-NIR spectra of these 3 types of raw materials in the wavenumber range of 9000 - 4000 cm^{-1} (Wagner et al. 2015)

bending vibrations at band around 5141 cm^{-1} (Shenk et al. 2001) is different compared to the wood fibres spectrum as this spectrum shows the first overtone O-H stretching vibrations at the wave number 5192 cm^{-1} (Schwanninger et al. 2011). Furthermore, the spectrum of the wood fibres shows a significant difference at the band around 4751 cm^{-1}, which corresponded to the third overtone of the asymmetric C-O-O stretching and the O-H bending as well as the C-O stretching vibrations (Shenk et al. 2001; Schwanninger et al. 2011). The bands at around 5777 and 4373 cm^{-1} corresponded to the first overtone of C-H stretching and the second overtone of C-H bending as well as CH_2 deformation vibrations (Shenk et al. 2001). These results show that the NIR spectroscopy is suitable for characterizing different materials of wood and leather, which was also depicted by the FT-IR and Raman Spectroscopy (Grünewald et al. 2013; Tondi et al. 2015; Wagner et al. 2015). However, the two types of wet white and wet blue leather materials cannot be distinguished by the analysis of the two NIR spectra.

3.3 Properties of the composite panels

Fibre composite panels made of wood and different wet white and wet blue leather particles with a dimension of 450 x 450 x 4.5 mm^3 and densities ranging from 700 to 1200 kg cm^{-3} were produced and tested according to different international standards.

3.3.1 Mechanical properties

The composite mixing ratio of the two leather fold chips and the wood fibres was varied for this study and the ratio of leather to wood can be listed as follows: i) 0 %, ii) 15 %, (iii) 25 %, (iv) 35 % and (v) 50 %. The glue amount of 10 % urea-formaldehyde resin (UF) and the addition of 2 % hardener were left constant for all materials. The simultaneous gluing of both materials (wood fibres and leather particles) was carried out in a plough blade mixer type WAM WBH 75 and a spray nozzle (slurry) with a diameter of 2.3 mm and a pressure of 2 bar. The pressing temperature was determined by preliminary investigations in the temperature range from 60 °C to 180 °C by Rindler et al. (2015) and Solt et al. (2015), which largely fulfilled the requirements on the part of the leather particles, since already in a preliminary stage during the shrinking process at low temperatures (below 60 °C) possible fibre damage can occur depending on the type of leather (Loewe 1959). The pressing temperature of 80 °C and the pressing cycle were kept constant for all manufacturing trials.

After the pressing process the samples were stored in a standard climate (20 °C and 65 % relative air humidity (r. H.)). The sample preparation and the mechanical testing procedure for the modulus of rupture (MOR) and elasticity (MOE) were according to Solt et al. (2015). To obtain meaningful results, each of the mechanical tests had an amount of 5 samples. Figure 3.6 shows the transverse tensile strength as a function of the bulk density. The achieved transverse tensile strengths of the wood fibre reference boards lie between 0.77 N mm^{-2} and 1.51 N mm^{-2} and are comparable with the majority of composite boards with different leather contents (Solt et al. 2015). The transverse tensile strength of the composite panels with

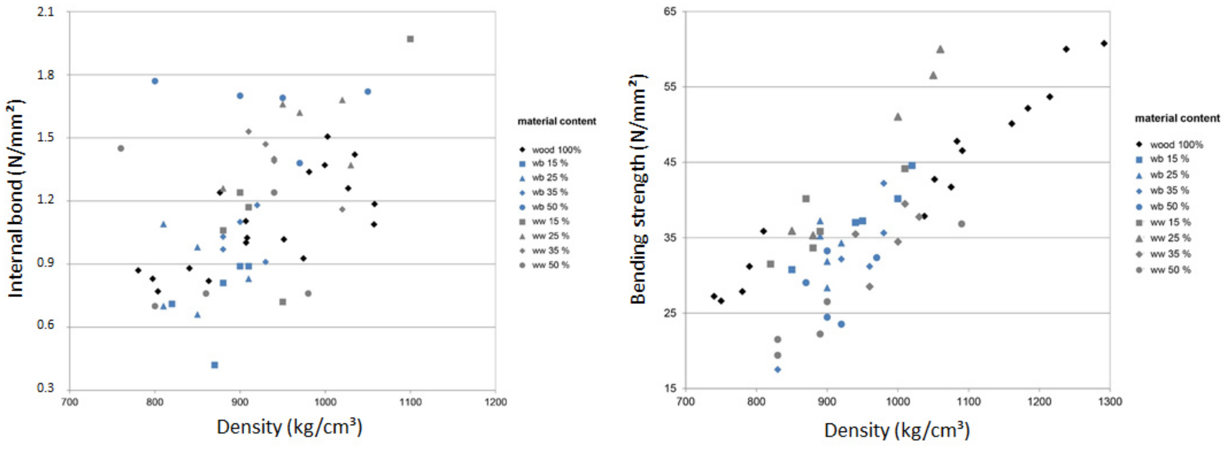


Figure 3.6: Mechanical properties of high-density composite panel made from wood, wet white (ww) and wet blue (wb) leather particles as a function of the density, left) internal bond and right) bending strength (Solt et al. 2015)

50 % wet blue particles tends to be higher. However, no clear differences in transverse tensile strength can be found between the individual types of leather (Wieland et al. 2013).

The bending strength is influenced by the different leather proportions (Figure 3.6). The strengths of the reference samples are higher than those of the wood-based composite boards, only the boards with wet white leather proportions of 15 % and 25 % can show a similar ratio of flexural strength to bulk density as the reference boards. A similar behaviour can be observed in the results of the modulus of elasticity (Solt et al. 2015).

The mechanical properties of fibreboards (MDF range around 800 kg m^{-3}) show similarities compared to the different leather materials. The ratio of different leathers proportions to the wood fibres and the various thickness of fibreboard samples were selected from the results of various previous mechanical studies by Solt et al. (2015) and Rindler et al. (2015). The values of the physical and mechanical properties of various wood leather fibreboards are shown in Table 3.1. With increasing of the leather amount the values of mechanical properties of the wood leather fibreboards were decreased. This phenomenon can be determined for various thicknesses of the wood leather fibreboards. All these results are consistent with the detailed analysis of mechanical properties by Grünewald et al. (2013); Solt et al. (2015); Rindler et al. (2015).

Table 3.1: Estimated mean and standard deviation (SD) of the physical and mechanical properties of the wood leather fibreboards with different leather types (ww and wb) and various ratios of wood fibre and leather (Wagner et al. 2015)

composition of panel			physical properties			
wood (%)	ww leather (%)	wb leather (%)	thickness (mm)	Density (kg m^{-3})	MOE (N mm^{-2})	MOR (N mm^{-2})
66.6	33.3	0.0	8.0	807.0 (49.9)	1997.53 (148.30)	23.32 (1.66)
66.6	33.3	0.0	12.0	854.0 (31.4)	1779.72 (120.77)	17.11 (1.79)
66.6	33.3	0.0	16.0	767.0 (39.1)	1721.00 (156.89)	17.14 (2.03)
66.6	33.3	0.0	20.0	764.0 (50.5)	1627.89 (195.89)	15.47 (2.35)
33.3	66.6	0.0	8.0	829.0 (27.7)	1178.70 (95.74)	13.00 (1.10)
33.3	66.6	0.0	12.0	890.0 (29.3)	1151.01 (83.25)	13.33 (0.89)
33.3	66.6	0.0	16.0	835.0 (34.0)	1278.56 (127.11)	13.80 (1.35)
0.0	100.0	0.0	8.0	722 (24.3)	539.00 (88.67)	6.35 (0.94)
0.0	100.0	0.0	12.0	935.0 (59.1)	1099.31 (237.85)	9.35 (1.76)
0.0	100.0	0.0	16.0	956.0 (63.7)	908.67 (160.08)	10.67 (1.93)
0.0	100.0	0.0	20.0	951.0 (59.0)	816.53 (156.04)	10.30 (1.98)
66.6	0.0	33.3	12.0	772.0 (90.0)	1513.00 (349.01)	14.57 (3.42)
66.6	0.0	33.3	16.0	703.0 (60.5)	1577.00 (98.86)	11.80 (0.87)
66.6	0.0	33.3	20.0	694.0 (77.7)	1488.00 (189.81)	15.15 (1.82)
33.3	0.0	66.6	12.0	828.0 (30.4)	1142.00 (277.49)	12.59 (2.86)
33.3	0.0	66.6	16.0	777.0 (79.6)	1122.00 (251.38)	11.80 (9.72)
33.3	0.0	66.6	20.0	725.0 (34.5)	938.00 (208.01)	9.72 (2.01)

The variation of the measured density values can be attributed to the manual small-scale scattering. On the other hand, spring back effects after the pressing process in the fibre boards (only wood) and shrinkage effect in ww and wb leather particles cannot be neglected. Likewise, the density differences may occur due to the fact that leather absorbs higher moisture than wood (Rindler et al. 2015). For the comparison of the further investigations, the samples were stored at 20 °C and 65 % relative air humidity up to constant weight. Table 3.1 shows the bulk density, bending strength and modulus of elasticity for the testing samples with wood, wet blue and wet with and its combinations. The bending strength is influenced by the different leather proportions. The achieved strengths of the wood fibre boards (HDF) are lower compared to the majority of composite boards with different leather contents. The bending strength of the composite panels with 66.6 % ww or wb particles tends to be higher (Solt et al. 2015). However, no clear differences in strength can be found between the individual types of leather. This behaviour cannot be clearly interpreted and depends on many factors. On one hand, particle distribution and different structural properties of wood fibres and leather particles as well as the expression of chemical bonds effects the properties of the composites (Paulitsch and Barbu 2015). On the other hand, wet white and wet blue leather particles differ in the tanning agents used. This also changes the individual material properties, for example the drying temperature in mineral tanning (100 °C to 120 °C) is higher than in synthetic tanning (75 °C to 85 °C) (Pauligk and Hagen 1987). Therefore, the chemical properties of the leather are also influenced by the tanning agents and this could affect the bonding of the composite material (Wagner et al. 2015). Vegetable and synthetic substances form bonds via hydrogen bonds or dipole forces between the tanning agent and the leather (protein), while tanning with mineral salts (e.g. chromium or aluminium) is of a coordinative nature and has a higher bond strength than synthetic tanning (Loewe 1959). This effects the chemical bonds between the adhesive system (e.g. UF resin with ammonium sulphate hardener) and the leather. Also, the pH value plays an important role because not only the curing of UF adhesives, but also the tanning processes are controlled with this value (Pauligk and Hagen 1987).

3.3.2 Multivariate analysis of mechanical properties

Based on their mechanical and technical properties the wood leather fibre composites are one of the most interesting wood engineered materials developing at Salzburg University of Applied Sciences - Campus Kuchl since 2010. These composite materials are highly sustainable because they can be produced by coupling wood fibres with industrial waste of tannery plants (Wieland et al. 2010b).

The analysis of the wood leather fibreboard with the near infrared spectroscopy (NIRS) can provide a basis for further efforts in the up-scaling from the laboratory to industrial conditions for consumer application concerning this tool for the development of quality assurance control system (Wagner et al. 2015). Also, the NIR spectra of the various wood leather panels show differences in IR bands regarding the wavenumbers of the bands (Figure 3.7). The significant bands are around the wavenumbers 4886 and 4587 cm^{-1} for the leather particles and the wave number 4751 cm^{-1} for the wood fibres. With the increase of the amount of ww leather shavings the changes in these wavenumbers can be also seen. The wood and leather materials can be easily distinguished, while a difference between ww and wb particles could not be determined. Therefore, both types of leather shavings (ww and wb) were together analysed. For this reason, the data of the NIRS were used for the data reduction via principal components analysis (PCA). Figure 3.8 states the results of the data reduction of the NIR information of the wood leather panels. The variance of the data explained by the PC is reported in parentheses. The two principal components of the score plot

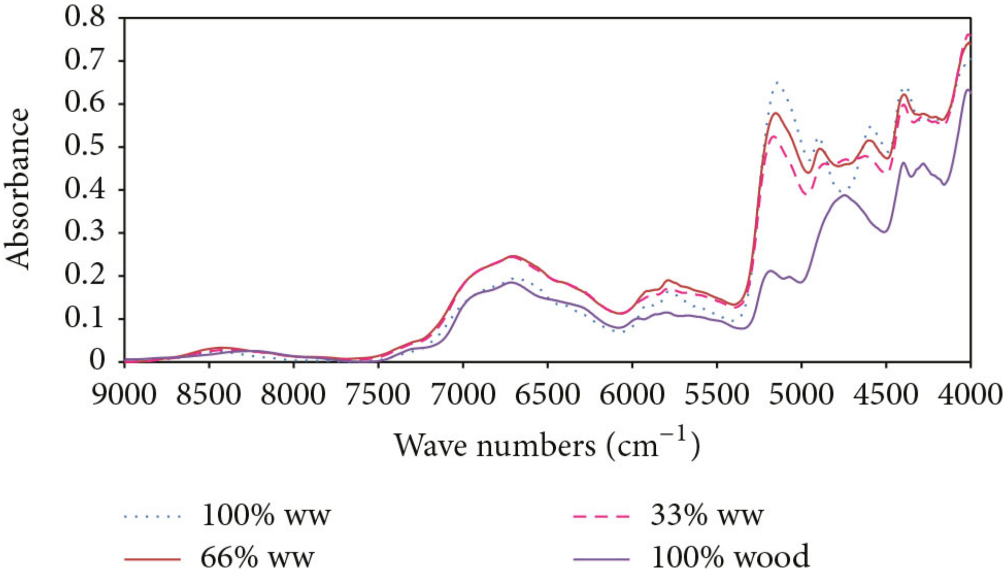


Figure 3.7: FT-NIR spectra of wood leather fibreboards with various concentrations of wood and wet white (ww) leather particles in the wave number range between 9000 and 4000 cm^{-1} (Wagner et al. 2015)

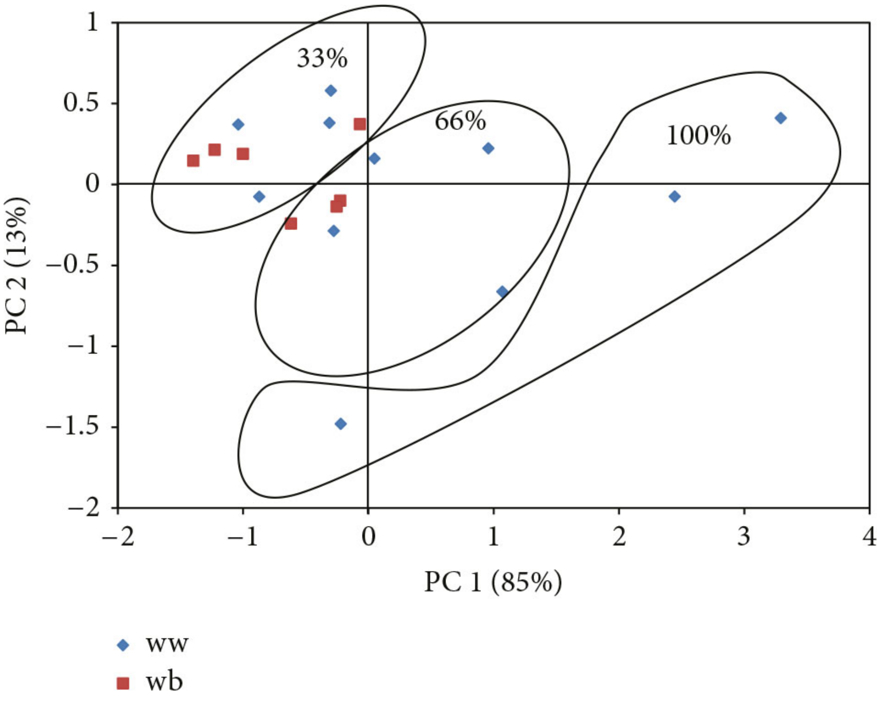


Figure 3.8: Principal component (PC) analysis score plot of near infrared spectra of various wood fibre leather panels (Wagner et al. 2015)

allow the representation of the amount of leather shavings in the various panels. Even though the PC 1 explained 85 % of the variance, whereas the PC 2 explains only 13 % of the variance, the combination of the two components describes the two most important parameters i) wood fibre and ii) leather contents (Wagner et al. 2015). The loadings of the PCs provide relationship between each of wavenumbers and the corresponding score plot of the principle components (Figure 3.9). The loadings of the PC 1 show high positive values for the wave numbers around 5110 and 4562 cm^{-1}, which represent protein vibrations. In the loadings for PC 2, contributions of the wood fibres (e.g. cellulose) derived bands can be observed at band around the wave number 4747 cm^{-1}, which has the highest positive values of the loadings. The results of the physical and mechanical properties of various wood leather fibreboards are shown in Table 3.1. With increasing of the leather amount the values of mechanical properties of the wood leather boards were decreased (Rindler et al. 2015; Solt et al. 2015; Wagner et al. 2015; Wieland et al. 2010b). This phenomenon can be determined for various thicknesses of the wood leather fibreboards. All these results are consistent with the detailed analysis of mechanical properties from Grünewald et al. (2013); Solt et al. (2015).

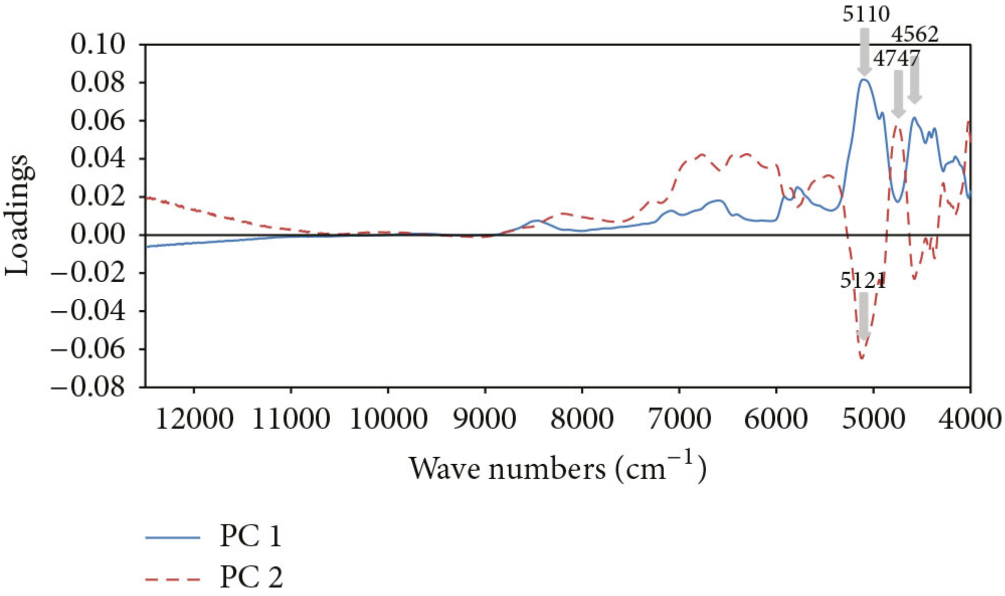


Figure 3.9: Loadings of the first two principal components (PCs) of near infrared spectra of various wood fibre leather panels (Wagner et al. 2015)

Based on the NIR results multivariate statistic methods were used for the estimation of the mechanical material properties. For the PLSR models the NIR data were pre-treated by using the second derivative (15 smoothing points). A full cross-validation was performed for every sample. For wood leather fibreboard samples (n = 18), the coefficient of determination (R^2) was 77.35 % and the RMSECV was 50.3 % with two principle components (PCs). PLSR models for the bending strength (MOR) of the fibreboard were also calculated with 18 samples, resulting in R^2 = 93.55 % and RMSECV = 1.7 % with two PCs (Wagner et al. 2015). Figure 3.10 shows the measured versus the predicted values of bending strength of various wood leather fibreboards. With these results it seems that the FT-NIR spectroscopy is able to estimate the physical

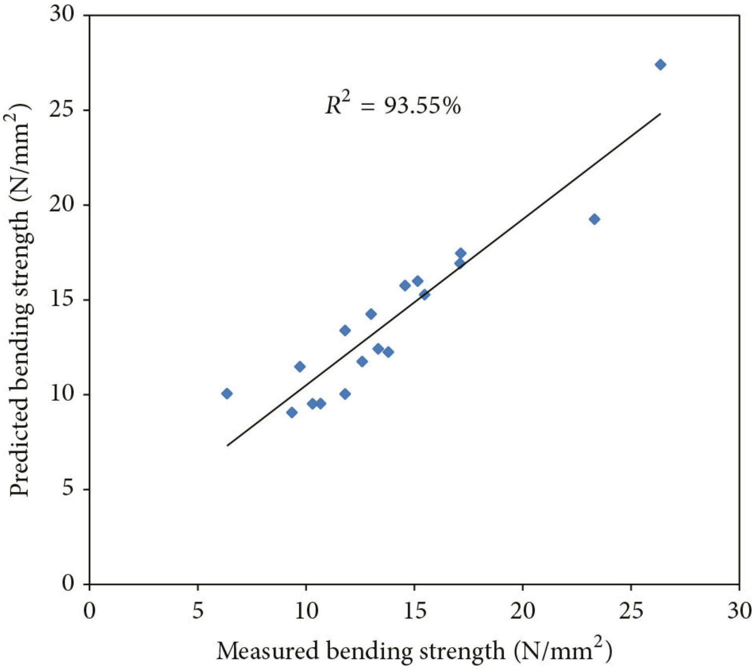


Figure 3.10: Predicted versus measured values of the PLSR model to estimate the bending strength with the FT-NIR data (Wagner et al. 2015)

and mechanical properties. The relationship between the mechanical properties and the FT-NIR spectra demands further consideration. The physical and mechanical features of the wood fibre leather composite samples depend on the leather content. The geometric form of leather particles is not comparable with wood fibres. Therefore, the distribution of the leather particles is not homogeneous (Rindler et al. 2015; Solt 2014). There are some areas with bigger leather accumulations (Tondi et al. 2015). Simultaneously, the leather particles can fill in the void of the fibreboard (Wagner et al. 2015). With changing leather contents, the material properties of the fibreboards composite are also modified. Therefore, the leather particles are not only an additive of the wood based boards and can be used for new material resources for wood based panel (Wagner et al. 2015) with additional material properties (Schnabel et al. 2019c).

3.4 Fire resistance properties of leather-wood fibreboard

The combination of wood and leather in a composite material showed enhanced fire resistance properties (Wieland and Schnabel 2012) . The reasons of this behaviour and the chemical reactions involved are unexplained (Rindler et al. 2015). Schnabel et al. (2019c) studied the reasons why wood-leather fibreboards showed highly enhanced fire resistance. A comparison between the combustion of wood fibreboard and wood-leather fibreboard is shown in Figure 3.11. After the same time of flame impingement the wood-leather fibreboard sample displays less degradation than the wood fibreboard sample (MDF) and even the wood fibreboard is burning. The combustion resistance of wood-leather fibreboards was observed also with the hot-gun perforation test (Schnabel et al. 2019c). In this case, wood (90 %) fibreboard (MDF), mixed wood (45 %) - leather (45 %) boards, and pure leather (90 %) boards underwent the hot air (530 °C) and the time until the 4.5 mm thickness of the boards was burned through were registered according to the method from (Rindler et al. 2015).

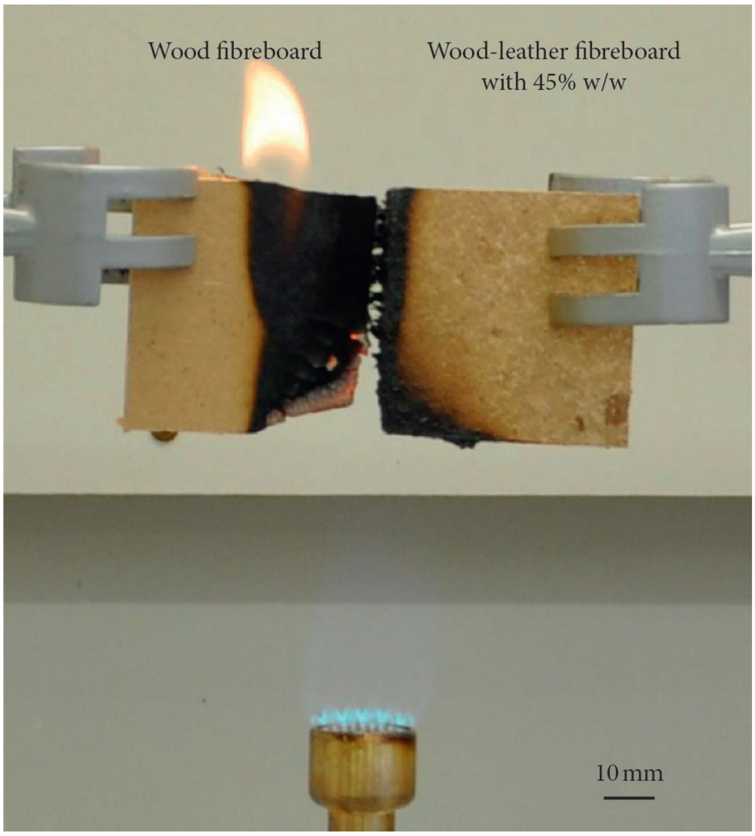


Figure 3.11: Combustion test of wood (left) and wood-leather (right) fibreboards by using a Bunsen burner (Schnabel et al. 2019c)

3.4.1 Influence on fire resistance

In Figures 3.12, the results of the hot-gun perforation test of wood and ww leather wood composite materials are reported. The wood fibreboard samples presented the lower penetration time, and according to the specific density, they needed less than 100 sec to be perforated by using the high temperature of 530 °C. The mixed boards with wood and leather presented a set of results which were always much improved from the pure wood product (Figure 3.12a). Finally, the pure leather panel could not be destroyed by the hot-gun temperature and energy after 25 minutes, time at which the test was stopped. These data showed that leather is the important player, and it is responsible for the improved fire resistance of the panels (Rindler et al. 2015). The formation of a black, smooth, and plastic-like surface and simultaneous swelling/foaming phenomenon was observed (Figure 3.12), which may act as a heat barrier and protect the inner layers for degradation of the high temperature in this test. The surface of the heated area looked shiner an less rough than the unaffected part and the latter seemed to be very tight, and it does not allow the heat to go through this layer(Schnabel et al. 2019c). Furthermore, the swelling of the first layers of leather fibreboard material (cf. Figure 3.12c), due to the release of gases (e.g., water, ammonia, nitrogen and carbon oxides), produce a foamy structure, which hinders/slows down the heat transfer to the product core offering a very effective physical protection against degradation by fire.

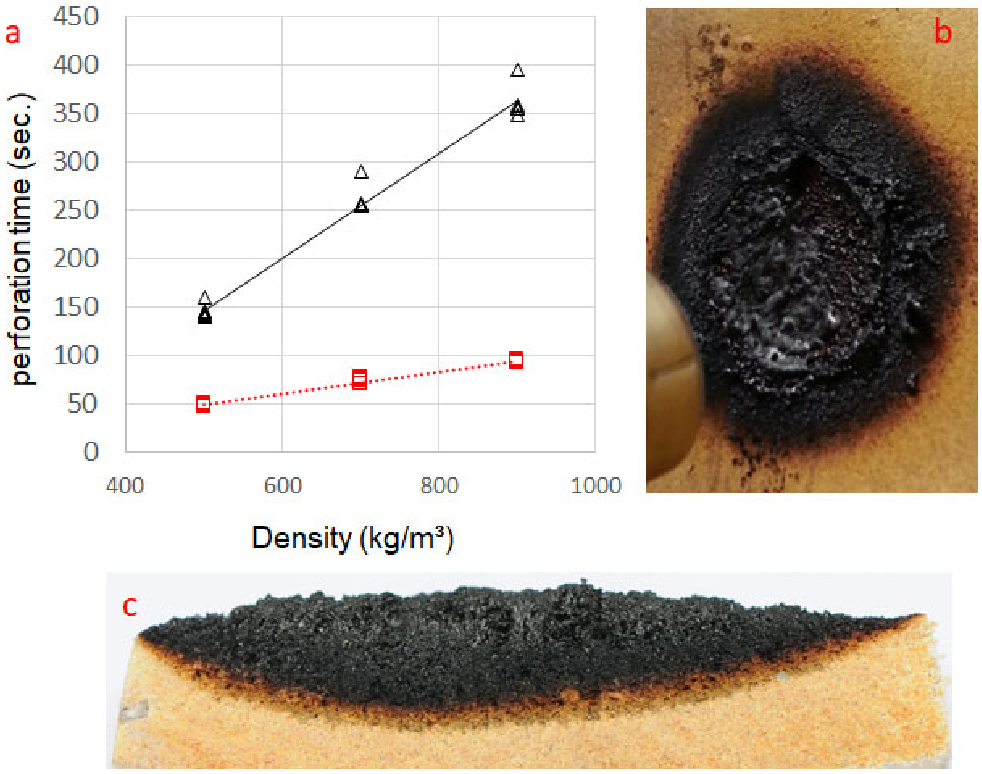


Figure 3.12: Results from the hot-gun perforation test of wood, wood-leather, and ww leather fibreboards. a) Perforation time vs density; b) Appearance of the leather surface after 25 minutes of hot-gun exposure; c) transverval session of leather (90 %) board after 25 minutes of hot-gun exposure (Schnabel et al. 2019c)

3.4.2 Chemical changes due to high temperature

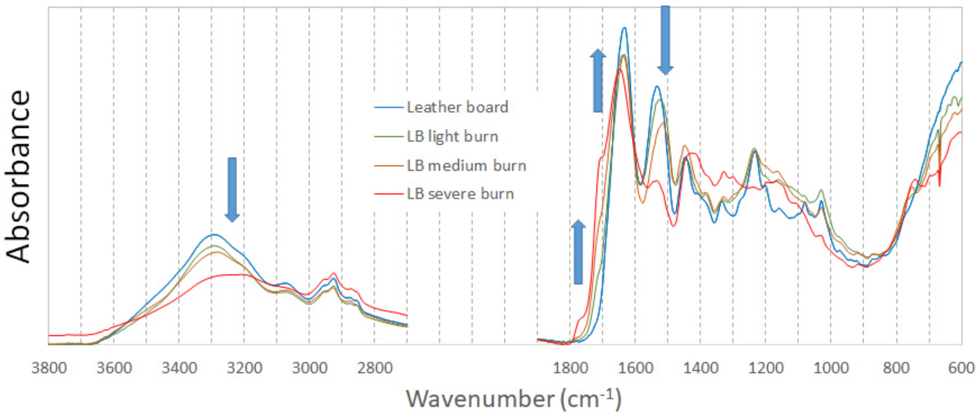


Figure 3.13: FT-IR spectra of a wet-white leather sample after hot-gun test of four areas with different treatment intensities (distance from the temperature source) on the surface (Schnabel et al. 2019d)

The IR signals from the FT-IR spectroscopy of the leather board after the hot-gun exposure were also collected on different surface areas (Figure 3.13) unaffected, slightly burned (yellowish region), and severe burning (black region). A defined trend can be seen, and the major changes observed are the following: the broad band between 3600 and 3100 cm^{-1} as well as the band between 1580 and 1480 cm^{-1} decrease significantly, while two new band appear at 1770 (only in the severe burning region) and 1720 cm^{-1}. The band between 3600 and 3100 cm^{-1} is typical for N-H and O-H stretching vibrations (Tondi et al. 2015), its decrease is due to two chemical reactions: (i) the primary and secondary amines evolve to more substituted forms and (ii) alcohols evolve to more oxidized products (e.g., aldehydes and carboxylic acid). The band around 1540 cm^{-1} is corresponding with out-of-plane vibrations of C-N stretching coupled with N-H bending vibrations (amide II) (Pretsch et al. 2010). This band is affected by establishing of secondary forces in the secondary structure of the proteins, and its decrease can be attributed to the rupture of this arrangement with formation of random conformations (Schnabel et al. 2019c). The new signal at 1770 cm^{-1} is probably due to the formation of anhydrides after severe burning, while the increasing signal at 1720 cm^{-1} can be attributed to acids. This means that during the thermal degradation process, carboxylic groups are formed. The signal at 1720 cm^{-1} band can also be partially attributed to the presence of 2,5-diketopiperazine (DKP) that can be a possible rearrangement for dipeptides (Schnabel et al. 2019c). In the schema of Figure 3.14, a possible rearrangement for a proline-glycine moiety is proposed. Other milder trends that can be observed are as follows: (i) the deceased of the IR signal at 1640 cm^{-1} attributed to the C=O stretching of the amide (amide I), which is the logical consequence of the degradation process because of the amidic bond rupture evolving to more oxidized species (e.g., carboxylic acids and hydroxylamines) and (ii) the band at 1440

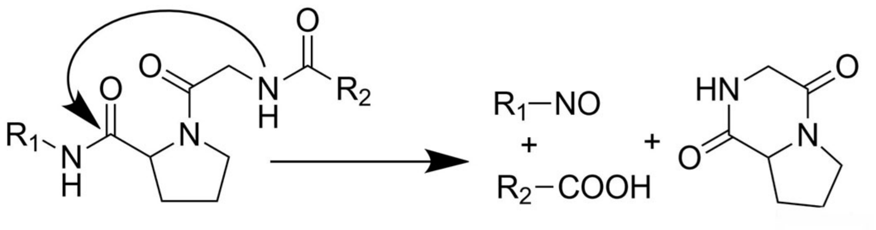


Figure 3.14: Possible reaction schema for the molecular rearrangement of wet-white leather undergoing thermal degradation (Schnabel et al. 2019c)

cm^{-1} that could be attributed to the presence of higher amount amino-aromatic moieties like pyrazine and pyrrole (Schnabel et al. 2019c). These rearrangements begin to be significant when leather is exposed to temperature higher than 250 °C for 5 minutes, confirming the findings of the study by the fact that the temperature development inside the fibreboard with leather particles is reduced compared to the wood fibreboard samples (Figure 3.15). The thermal degradation of wet-white leather particles was also observed

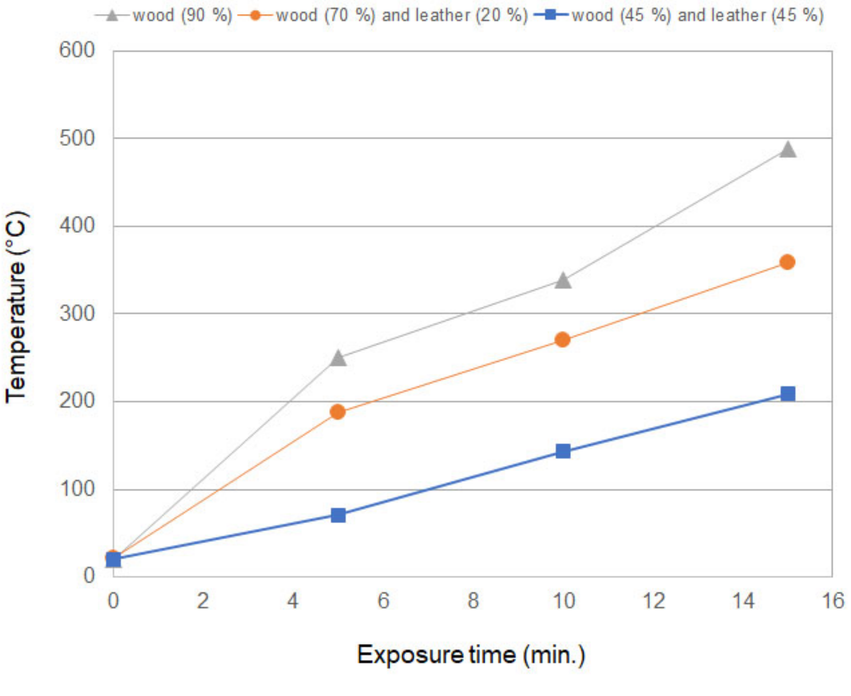


Figure 3.15: Temperature curve of different wood-leather fibreboards at 1 mm under the surface, which exposures with 50 kW m^{-1} (Schnabel 2015)

by using a proylosis-GC/MS method at the temperature of 330 °C, and the list of the compounds registered is shown in Table 3.2. Several fractions were separated during the gas chromatography, and they can be categorised in two families of compounds: i) nitrogen-containing aromatics, derived by the rearrangement of proteins, and ii) phenolics, derived from the synthetic tannin used for the tanning of the leather. The great majority of the molecules released during the GC-MS study were nitrogen-containing aromatics (around 75 %). These molecules may be produced by the intermolecular cyclization/aromatization of amino acid pairs and, in particular, in the majority of the cases the degradation occurred via simple intra-molecular cyclization of two amino acids units in which one of them was proline. Due to its exceptional conformational rigidity, proline cannot participate to alpha-helix formation, and therefore, it constrains the protein chain to assume a folded structure, which facilitates the dimeric intermolecular arrangement (Schnabel et al. 2019c). The pathway proposed in Figure 3.14 shows a proline-containing protein that cyclizes. this mechanism can occur for many dimers with different branches (Pro-Glx principally, but also Pro-Ala and pro-Leu, which are the three more frequent aminoacids in leather).

Table 3.2: Pyrolysis-GC/MS analysis at 330 °C of wet-white leather shavings (Schnabel et al. 2019c)

Retention time (min)	Assignments	Possible source	Relative amount
20.199	4-Cloro-3-methylphenol	Synthetic tannin	12
23.172	Pyrorole-2-carboxaminde	Proline	27
25.871	Xanthine	Glycine	10
26.805	2-Hydroxybiphenyl	Synthetic tannin	29
32.864	Phenol[2,3-e]pyrimidine/1,3,4 H)2-one	Tyrosine	21
34.433/ 35.117	Hexahydropyrrolo[1,2-a]pyrazine-1,4-dione 2-methyl	Proline + alanine	15/ 18
36.396	Hexahydropyrrolo[1,2-a]pyrazine-1,4-dinoe*	Proline + glycine	100
39.065	5,10-Diethoxy-2,3,7,8,tetrahydro-1H,6H-dipyrrolo[1,2-a;1',2'-d]pyrazine	Two prolines	32
42.984	Chlorohydroxyl biphenyl	Synthetic tannin	8
44.506	Hexahydro pyrrolo[1,2-a]pyrazine 2-isobutyl-1,4-dione	Proline + leucine and/or isoleucine	21

So, it results that the degradation of leather is a process that involves the production of several amino-aromatic compounds due to the intramolecular arrangement of the aminoacids under oxidative conditions. It can be stated that the surface of the leather after thermal degradation results to be i) richer in acid groups and/or diketopyperazine (DKP) and ii) poorer of amidic signals and intra-molecular H-bonds due to less organized arrangement. Consistently, the major products of the Py-GC/MS are diketo-nitrogen-containing aromatics.

3.4.3 Possible material reaction

The findings related to the thermal degradation of wet-white leather shavings boards can be summarized and interpreted in the next sentences. During burning, the surface of leather rearranges in an overlay coating-like heat barrier that slows down the penetration of oxygen in the core. Simultaneously, the gas developed during burning remains partially blocked into the structure, which develops into a foamy structure that delays the heat transmission to the core. The leather modifies its structure and rearranges by producing acid moieties and these moieties can further react with amino groups of other peptide chains, by producing a networked polymer before undergoing to char. This would be the chemical origin of the plastic-like barrier observed. DKP are produced during the degradation process. These gases do not burn, and they contribute to the outstanding results observed by (Rindler et al. 2015; Schnabel et al. 2019c). A possible mechanism of proteins rearrangement is shown in Figure 3.16.

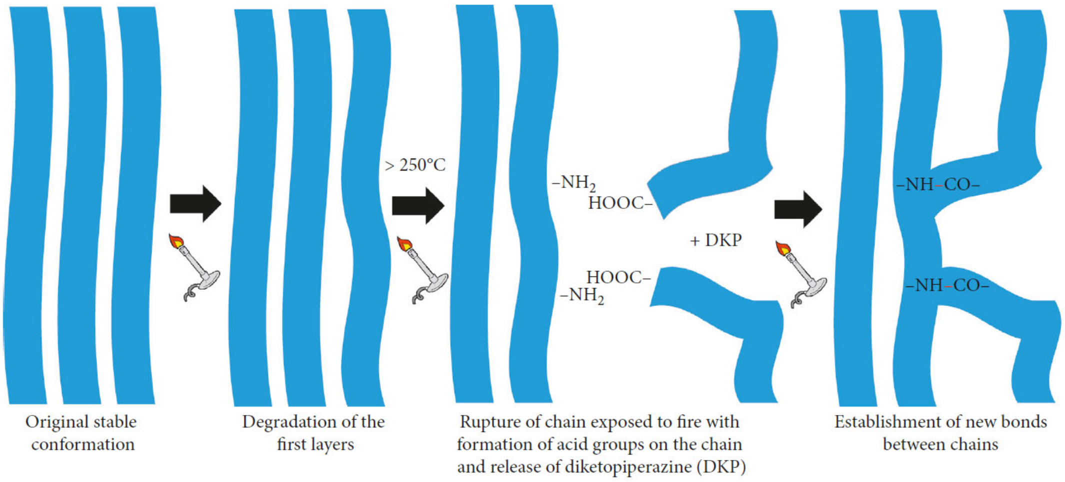


Figure 3.16: Scheme of the possible mechanism for the high resistance of a wet-white leather shavings board against the hot-gun perforation test (Schnabel et al. 2019c)

Initially, the proteins have a stable conformation, then, heat goes in contact and the first layers change their arrangement. Longer exposure and/or higher temperature causes the rupture of the chain by producing DKP and acid-containing moieties. The latter may react with free amino groups of other peptides, building a networked polymer impenetrable for gases. Simultaneously, small molecules such as water, ammonia, NO_X, CO, and CO_2 but also DKPs are produced and they expand inside this networked polymer producing a foam-like structure. This mechanism could be applied also to mixed wet-white leather shavings in wood fibreboards. The acid moieties produced by heating of the leather could also esterify the hydroxyl groups of wood with the similar result of tightening the structure. In this case, Maillard reaction between carbohydrates and proteins could also be considered (Schnabel et al. 2019c). However, the establishment of overall impenetrable network, and a minor plastic-like effect was observed; consequently, the results were less performing than the ones observed for pure leather fibreboards.

These sustainable bio-composites can be considered for several applications where natural and fire-resistant products are needed. Automotive, nautical, and green building are some of the most interesting sectors, where leather-based fibreboards can be suited. All these findings provide a basis for further efforts in the upscale form laboratory to industrial conditions for consumer applications (Wagner et al. 2015).

4 Valorisation of bio-based resources to high added-value products and applications[*]

Based on the consideration of the bio-economy that all raw material should be used was established an exploratory focus in this field (Atena and Schnabel 2018; Thorenz et al. 2018). This includes a new collaborations in other scientific areas (e.g. medical sciences) and industries (e.g. natural cosmetic or plastic) (Morandini et al. 2018; Schnabel 2016; Wagner et al. 2017; Tondi et al. 2019).

The term biomass is used broadly for this sub chapter and includes all plant products of different tree or shrub species, such as wood, knots, bark as well as energy wood (e.g. stump and root wood). On the one hand side, the lignified resources are used as raw material for various applications, but also for energetic energy production (Zabaniotou 2018). A target large-scale used of the tree extractive is currently not performed, so large amounts of these substances simple released into the environment or thermally destroyed. On the other hand, plant extracts have always been used as raw material sources for valuable products in various fields such as nutritional supplementation, pharmacognosy, cosmetics and many more. For example, it is not surprising that more than 50 % of FDA-approved (US Food and Drug Administration) medicines include parts of plants (Newman and Cragg 2016). But not only drugs are made from the extracts, but also additives for food, cosmetics and healthy products (Schönwälder et al. 2000; Tondi and Schnabel 2020). In addition, substances from wood-based sustainable materials can be used with little or no further chemical synthesis for pharmaceutical and cosmetic additives as well as nutritional supplements (Schnabel 2016).

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[*] The author managed and did the research in the characterisation of bioactive extractives properties of different tree tissues between 2013 and 2020 in the frame of five research projects with 14 national and international partners. These research results were presented in 5 scientific articles and in 6 international conference. A doctoral thesis has also been co-supervised in this field of research since 2018. The author contributed to the funding acquisition, design and implementation of the research, to the analysis of the results and to the writing of the manuscript for these articles.

4.1 Circumstances

Regarding the bio-economy and bio-refinery approach, not all substances in the wood are currently used (Bowyer et al. 2003). New thinking and techniques are helpful to exploit the huge potential of bio-based products for innovative applications from the laboratory to industry. Different gaps exist and bridges are necessary to industrially use the by-products (e.g. bark) (Ding et al. 2017) for new applications (e.g. bio-based polymers) and not only for energy generation (Atena and Schnabel 2018; Wagner et al. 2018a). On the one hand, the lumber and other bio-based materials must be cleaned and dried for immediately stored and/or processed in order to make the raw materials usable for products and applications (Correal-Mòdol et al. 2014; Schnabel et al. 2007; Schnabel 2016). These different processing water is not used for further application and it is only waste materials at the current stage. For example, the approach is shown in Figure (4.1) to use the condensate of different wood processing processes (Wagner et al. 2016). These results may show that new value chains of the wood based industry in the field of bio-economy can be developed (Atena and Schnabel 2018; Sepperer et al. 2019; Wagner et al. 2018a). Other examples for establishing a new timber value chain exist (cf. chapter 1), however, further research is needed to foster the knowledge of possible applications of different materials (Jarre et al. 2020; Zabaniotou 2018; Schnabel et al. 2020a).

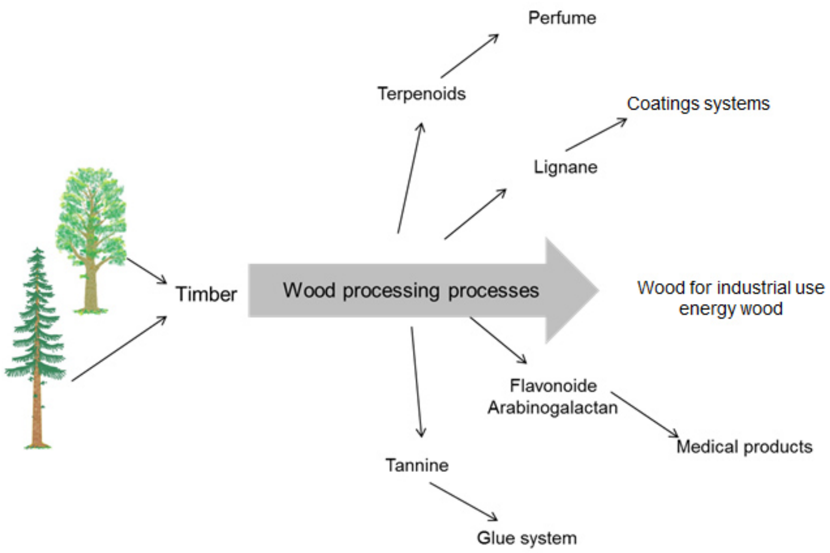

Figure 4.1: Holistic approach of the biomass usage (Schnabel 2016)

On the other hand, the remaining bark with bast and cambium is economically neglected compared to other parts from tree, which accounts for an average of 10 % of a tree and the bark has a much more amount of extractives compared to wood (Wagner et al. 2018a). Decades ago, land-filling of the bark as a waste product was a frequently used way of disposal (Ding et al. 2017). Currently, the amount of bark used is mainly used for thermal energy production within the wood industry or heating plants close to forested areas, but the high ash content and combustion characteristics than wood present difficulties (Kain 2016). Also large amounts of forest biomass are used for the energy production for urban areas (Atena and Schnabel 2018; Chirat 2017; Piotrowski et al. 2016b; Pieratti et al. 2020). The calorific value of the biomass is heavily dependent on water content and degree of impurities (Bowyer et al. 2003). As a significant loss of heat energy for the evaporation of the water contained in the combustion lost. However, a high amount of water content not only effects the calorific value but also the furnace and exhaust gas temperature. It can lead to the increased formation of unburned gases, wood tar or different acid (e.g. acetic acid), other toxicological potential of particles (Orasche et al. 2012) and increased flying ashes and blue haze due to the impurities attached during harvesting (Paulitsch and Barbu 2015). Furthermore, the gas during the combustion of wood does not present the volatile constituents of wood and these evaporate out even at low temperatures without chemical reactions changes and thus also do not contribute any energy. This can also apply to the burning of bark (Schnabel 2016). Therefore, in addition to worse combustion properties, the unused portion of volatile substances is also added, which should be no option for today.

In the sense of a holistic use of resources (e.g. cascade use), it should be the energetic used at the end of the residues (Schnabel et al. 2020a). The aim of this research is to develop new option for the transformation of waste materials to useful products and/or to increase the potential of the sustainable resource wood for different applications (e.g. in hygiene sensitive areas) (Kavian-Jahromi et al. 2015). The condensate from the steam process of wood is a possible example from the wood industry, which can be used for further applications. The total extractive contents of the condensate samples are lesser then in hot water extraction of larch or spruce. However, the total phenolic content is between 8.07 and 66.95 μ GAE ml^{-1} (GAE means Gallic Acid Equivalent) and showed antimicrobial effects for different bacteria strains (Ming et al. 2017; Vainio-Kaila et al. 2015; Wagner et al. 2018a). Based on the findings of the analysis of wood extractives in aqueous solutions from different wood species and wood processing techniques the potential of antimicrobial activities can be shown.

4.2 Differences in tree tissues

This section deals with the analysis of potential tree extractives as high added-value products, which can be used as preserving agent for natural cosmetic products. Therefore, for the first step, there were focuses on understanding of the antimicrobial effect of wood and the antimicrobial properties of wood.

4.2.1 Wood and photoirradiated wood

Wood is a well-accepted material for appearance products in furniture and building applications (Schnabel et al. 2009). Indeed the wood is a smart materials (Schnabel et al. 2017) and it is important for the wood industry to add functionality to this material for new applications and markets (Kaestner et al. 2016). Besides the main components cellulose, hemicellulose and lignin, the plant extracts play an important role for the recovery of valuable bioactives (Schnabel et al. 2016a; Wagner et al. 2020a). However, wood and its compounds undergoes various changes in properties during the life time (Schnabel et al. 2009) and also the functionality of the material may alter.

An interesting belongings of wood is the antimicrobial effect against microbe (Laireiter et al. 2014). The hygienic properties of wood have been discussed over the last decades since it might be important for various applications (e.g. cutting boards and interior material).

Table 4.1: Logarithmic mean (standard deviation) of CFUs (Colony Forming Units) of MRSA and *K. pneumoniae* recovered from references and different materials of larch after various incubation periods (Kavian-Jahromi et al. 2015)

Materials	Bacterial strain	Time (h)		
		0	3	24
References				
	MRSA	8.115 (0.000)	8.01 (0.000)	8.00 (0.000)
	K. pneumoniae	7.54 (0.000)	7.71 (0.000)	7.98 (0.000)
Cube				
Heartwood	MRSA	6.91 (0.043)	5.91 (0.158)	1.76 (2.590)
	K. pneumoniae	6.41 (0.192)	5.09 (1.435)	0.33 (1.291)
Sapwood	MRSA	6.11 (0.115)	4.27 (2.227)	0.33 (1.291)
	K. pneumoniae	5.63 (0.299)	1.00 (2.070)	0.00 (0.000)
Shaving				
Heartwood	MRSA	3.04 (1.032)	2.25 (1.488)	0.00 (0.000)
	K. pneumoniae	2.77 (1.922)	2.18 (2.089)	1.35 (1.982)
Sapwood	MRSA	2.40 (1.242)	0.00 (0.000)	0.27 (0.405)
	K. pneumoniae	0.80 (1.373)	0.46 (1.127)	1.00 (1.464)

Hygroscopicity of wood causes dehydration of bacteria and is the physical effect, whereas the inhibition of the growth of bacteria due to antibacterial substances in wood can be considered as the chemical effect (Kavian-Jahromi et al. 2015). Both forces are important for utilization of wood for hygienically sensitive reasons. This antimicrobial effect could be divided into passive and active effects, while these terms have not commonly differentiated in many publications (Laireiter et al. 2014). However, not every wood specie has the same hygienic property (Kavian-Jahromi et al. 2015; Wagner et al. 2017), whereas the wood has normally high hygroscopic properties, so that the material can easily absorb the necessary water for surviving of the bacteria strains. Kavian-Jahromi et al. (2015) were concluded that larch sapwood has a greater antibacterial effect than larch heartwood (Table 4.1). This can be ascribed to the fact that sapwood has larger pores and therefore any liquid can be absorbed into the wood more quickly (Niemz 1993; Skaar 1972). Thus the surface of the wood is sooner released from bacteria. The slighter hydrophilicity of cell walls in heartwood (Song et al. 2014) can explain the reduced bactericidal effect of heartwood. There exists differences in behaviours on sapwood and heartwood in the same board of a tree (Kavian-Jahromi et al. 2015; Laireiter et al. 2014). The total wood extractives amounts in larch by using a 6 hours hot water extraction method (e.g. Soxhlet apparatus) are 3.8 % (37.9 mg g^{-1}) of sapwood and 13,4 % (134.3 mg g^{-1}) of heartwood of dry wood as well as 12.50 % (125.0 mg g^{-1}) of dry bark (Wagner et al. 2017). If a simple maceration process performed for 24 hours at room temperature (22 - 23 °C). The total extraction yields of larch bark with the maceration method depicted differences and ranged from 50.1 mg g^{-1} of oven dry bark with methanol extraction to 22.0 mg g^{-1} with water extraction (Wagner et al. 2019). If methanol is used for the extraction, then the amount of wood extractives obtained values of 1.3 % for sapwood and 2.3 % for heartwood (Wagner et al. 2019).

Based on these results from Laireiter et al. (2014) it was shown that not only the water absorption of wood has an influence on the antimicrobial activity of wood but also the wood extractives plays a significant role for this material property. Nevertheless, it can be assumed that not all substance of the wood extractives shows the same antimicrobial activities and/or have significant influence on this activity. By using different solvents for the extraction process the influence on different components (e.g., resin acid, sterol or lignans) of the antimicrobial property of tree parts was shown that only a small amount of different substances are important for this activity (Wagner et al. 2017; 2018b; 2019).

The results of the experiments from Laireiter et al. (2014) show that the sample size of the wood plays and important role for a successful extraction of anti-microbial active substances. Due to the large freely available surface of sawdust, anti-microbial substances were dissolved more easily. Extracts of pine heartwood also had an active anti-microbial effect on *E. faecium* and *B. subtilis* (Figure 4.2). No statistically significant differences between sawdust- and wood disc-extracts were found for both germs. The data showed that

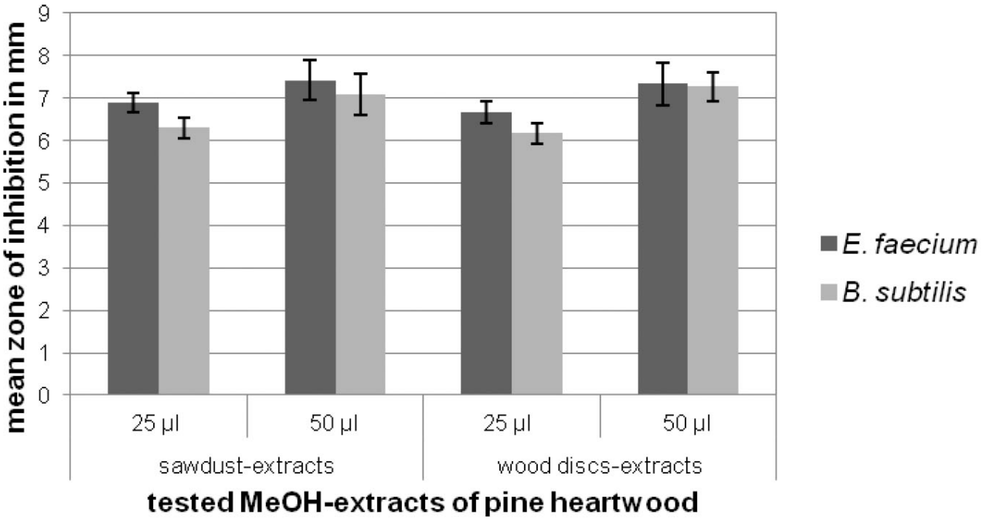

Figure 4.2: Results (mean ± SD) for the analysis of inhibitory zones when testing *Enterococcus faecium* and *Bacillus subtilis* with MeOH-extracts of pine heartwood (Laireiter et al. 2014)

the anti-microbial effects of the wood extracts were concentration dependent in all cases. The heartwood of Scots pine has an active anti-microbial effect against the chosen gram-positive bacteria *E. faecium, B. subtilis,* and *S. aureus*, whereas the sapwood did not show significant active anti-microbial effects on the bacteria investigated.

For this reason, the focus of one of the author's study is on larch sapwood and heartwood (*Larix decidua* [Mill]), because they are used widely in the alpine region and their microbiological properties in regards to bacterial on surfaces have still not been fully understood.

Due to the influence of the environmental conditions (i.e., light) the surface properties change (Fengel and Wegener 2003). Chemical bonds break and the lignin content of wood decreases as a consequence of photodegradation (Schnabel and Huber 2014; Huber and Schnabel 2015). New chromophores form as exposure time is increased. Furthermore, the colour of wood varies rapidly when it is exposed to light (Schnabel et al. 2009; 2015). These changes are a superficial phenomenon because of the small penetration of ultraviolet and visible light into wood (Schnabel and Huber 2014). However, this process may also influence the antimicrobial effect on wood (Wagner et al. 2019). After a certain time, the antimicrobial activity of wood can reduce or increase in hygiene sensitive areas.

Table 4.2: Mean of CFUs of *K. pneumoniae* and MRSA recovered from different materials without and after UV-light irradiation (Wagner et al. 2020b)

Bacterial strain	samples	Time (min)						
		30	120	240	360	480	1440	2880
K. pneumoniae	control	4650	4455	4130	3098	6135	10000	10000
	larch sapwood	27	9	4	4	3	1	0
	larch sapwood UV-exposed	3	1	1	0	0	0	0
	larch heartwood	34	2	2	2	1	0	0
	larch haartwood UV-exposed	341	9	5	2	3	0	0
MRSA	control	2463	4611	7113	6050	6960	4608	8640
	larch sapwood	115	62	85	68	77	21	34
	larch sapwood UV-exposed	28	11	12	18	8	3	5
	larch heartwood	118	112	112	122	132	23	49
	larch heartwood UV-exposed	560	42	14	9	6	5	1

For both bacterial strains, the microbial growth on larch heart- and sapwood samples decreases significantly after the various incubation times, which are in consistent with the results from Kavian-Jahromi et al. (2015). However, after the UV-light irradiation of the samples, the reduction of the CFU values for K. pneumoniae is higher for larch sapwood than the samples without UV-light exposure. Table 4.2 lists the plate count of bacteria strains for all samples, which were incubated on larch samples before and after 20 h of UV-light irradiation. This behaviour guaranteed that the bacteria were not destroyed through the UV-irradiation, but the changes in material properties of the UV-exposed wood surfaces. In addition to this, after the UV-light irradiation of the samples and the new inoculation of the aged samples the reduction of the CFU values for *K. pneumoniae* is higher for larch sapwood than the samples without UV-light exposure. In addition to this, the heartwood samples which were measured showed another behaviour of the CFU values, because higher values of the 20 h UV-irradiated samples were determined compared to the samples without UV-light exposure. Moreover, the differences between the sap- and heartwood without irradiation and after the ageing process were also determined for the MRSA bacteria. However, after the first 30 min time of the inoculation of the wood samples the CFU values of aged larch heartwood samples decreased dramatically compared to the unirradiated larch wood samples. The results show that the antimicrobial effect of wood was positively influenced by the ageing process on larch sapwood for *K. pneumoniae* and MRSA and the larch hardwood for the MRSA only. It can be assumed that the chemical changes of wood due to the UV-light irradiation, which occur in short period of time, were sufficient to increase the antimicrobial activity of larch sapwood Through the UV-light irradiation the chemical compounds of the wood

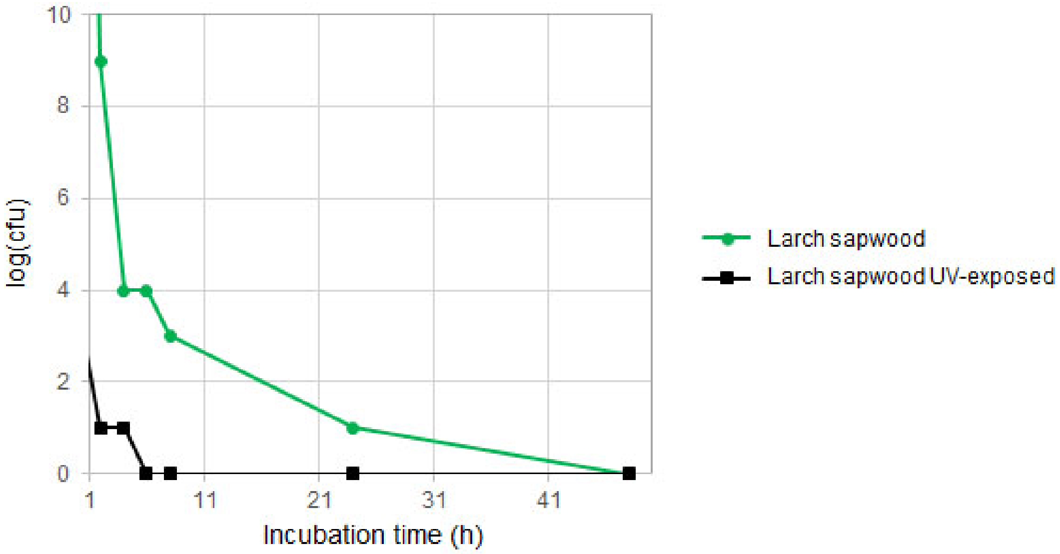

Figure 4.3: Mean of CFUs of *K. pneumoniae* from larch sapwood materials without and after UV-light irradiation (modified from Wagner et al. (2020b))

are alter. The various lignin degradation products and key precursor are more soluble and can be leach out by using water (Hon and Chang 1984; Tondi et al. 2013).

This approach was analysed in the following steps by FT-IR spectroscopy, followed by a HPLC method and determination of the total phenolic content. The FT-IR spectra from larch heartwood and sapwood samples before and after the UV-light irradiation are presented in Figure 4.4. The peaks obtained at 2930 cm^{-1} and 2890 cm^{-1} are an indication of stretching vibration form CH, CH_2 and CH_3 (Pretsch et al. 2010). These functional groups can correspond with the long chain values of fatty acids on the wood extractives. The IR signal of the carbonyl band at 1730 cm^{-1} increased due to light irradiation (Rosu et al. 2010). This observation is due to the formation of new carbonyl groups during photo-oxidation of the surfaces (Schnabel and Huber 2014). The band at 1600 cm^{-1} corresponds to aromatic skeletal breathing with CO stretching vibrations, which is increased based on the chemical changes in the lignin’s aromatic ring structure. This observation is also seen at the band 1510 cm^{-1}, the photodegradation of the lignin advances during the UV-exposure and carbonyl/carboxyl groups arise (Schnabel and Huber 2014). The band at 1270 cm^{-1} for CO stretching vibrations in lignin (guaiacyl) behaves in a very similar way to the 1600 cm^{-1} band (Schwanninger et al. 2004). This mentioned behaviour can also be seen in Figure 4.4. The absorbance height of the band for skeletal vibration of lignin (1510 cm^{-1}) is decreased, while the carbonyl or/and carboxyl groups of the wood surfaces are increased (Faix 1991). Due to the photodegradation of lignin the relative amount of cellulose increase and can be seen at the signal area from 1363 cm^{-1} to 1378 cm^{-1} which are the deformation vibration of CH groups assigned to carbohydrates (Pretsch et al. 2010). The band around 1030 cm^{-1}

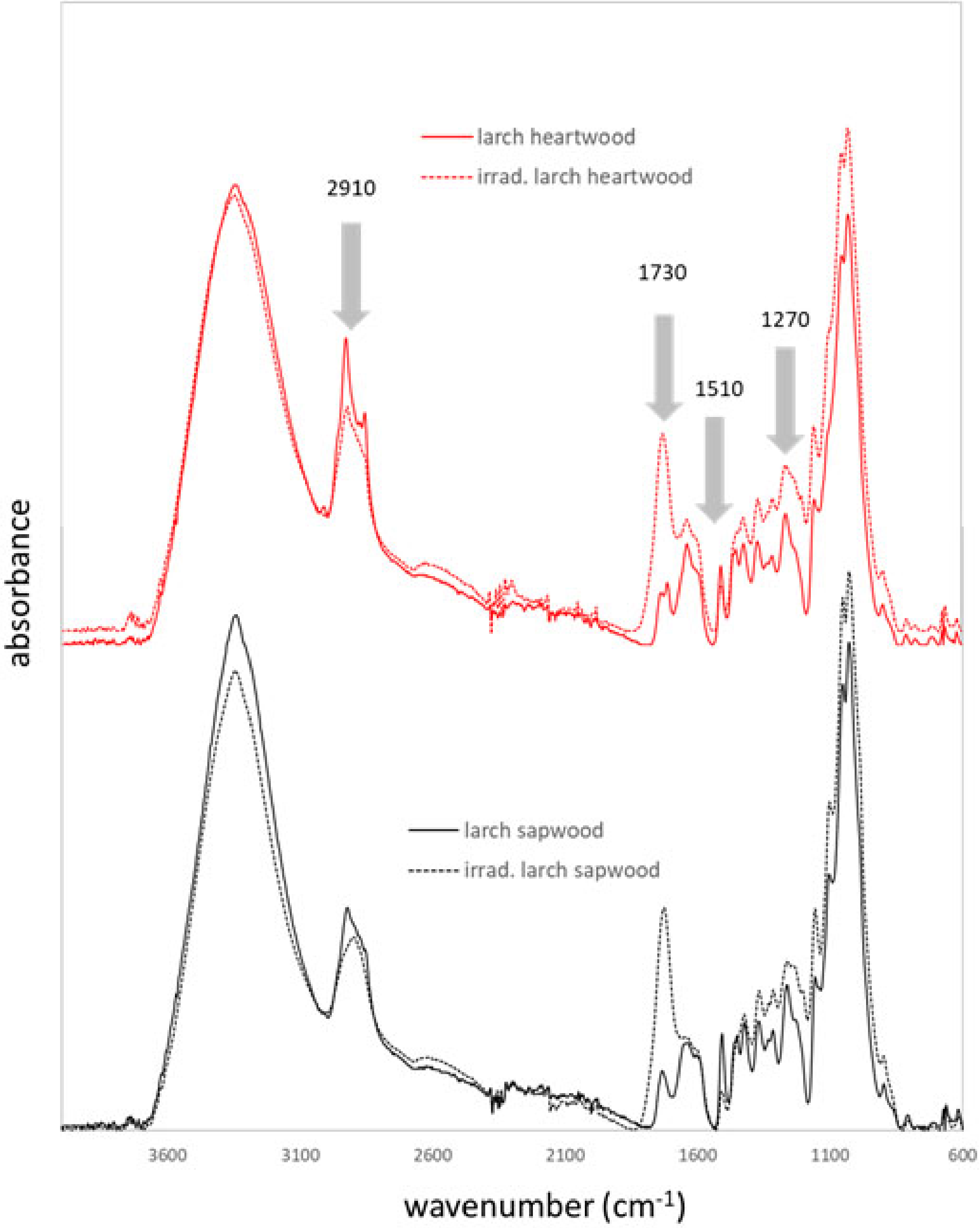

Figure 4.4: FT-IR spectra of unirradiated and 20 h UV-light irradiated of larch sapwood and heartwood obtained in the wavenumber range 600 cm^{-1} and 4000 cm^{-1} (Wagner et al. 2020b)

corresponds to the aromatic CH in plane deformation and CO deformation in primary alcohols as well as CO stretching vibration in lignin, cellulose and hemicellulose (Morgan and Orsler 1968; Schwanninger et al. 2004) and show an increase in absorbance in this area resulting in a better wettability of water on the wood surface.

Table 4.3 shows the relative ratios of various IR bands, which corresponds to the relative crystallinity index (H1370/2900) of cellulose (Schnabel et al. 2015), the OH/CH_2 ratio (H3500/2900) and C=O/CH_2 ratio (H1730/2900) of wood (Schnabel and Huber 2014) before and after the UV-light irradiation. The degree of crystallinity of cellulose changed due to the UV-irradiation. The hydroxyl (-OH) and carbonyl (-C=O) groups are hydrophilic, while the hydrocarbon (-CH_2-) chains are hydrophobic groups. The changes of these functional groups can impact the wettability of the wood.

Table 4.3: Relative mean ratios of IR bands of relative crystallinity of cellulose and different functional groups (Wagner et al. 2020b)

		Relative ratios of IR bands		
sample	UV-exposure (h)	1370/2900	3500/2900	1730/2900
larch heartwood	0	0.43	1.02	0.22
larch heartwood	20	0.67	1.20	0.96
larch sapwood	0	0.53	1.24	0.28
larch sapwood	20	0.72	1.23	1.15

The material surface was changed to more hydrophobic properties after the UV-light exposure. Therefore, the wettability of the wood surfaces is different. Moreover, the wettability depends on the chemical and the structural properties of the surfaces (Tondi et al. 2013; Huber et al. 2019). However, it can be assumed that the 20 hours of UV-light irradiation does not structurally change the surface strongly enough to result in a change in the wettability of the material (Kleinert 1970).

The increased hydrophilicity of UV-light exposed cell walls can explain the better antimicrobial effect of heartwood and sapwood compared to unirradiated wood samples. As the bacterial strains do not have more liquid for survival on irradiated wood surfaces. However, based on the results from Table 4.3, it should be assumed that the aged larch heartwood samples show the highest antibacterial activity, which is partly in line with the findings in Table 4.2, and conforms only with the CFUs of MSRA bacteria strain. Therefore, the determining factor for the better antibacterial effects of both aged wood samples may the changes in wood chemistry. New bonds and molecules were generated due to the UV-light exposure (Schnabel et al. 2009; Schnabel and Huber 2014). Figure 4.5 presents the HPLC chromatograms of the solid-liquid extraction of unirradiated and 20 h UV-light irradiated sapwood of larch. In Figure 4.5 the peaks height of the wood extractives from the irradiated wood samples increase, which are corresponding with a higher concentration of chemical substances of absorption at 280 nm (e.g. phenolic acids). Normally, the total extraction yield of polyphenols by using water is a very small amount compared to other extraction solvents

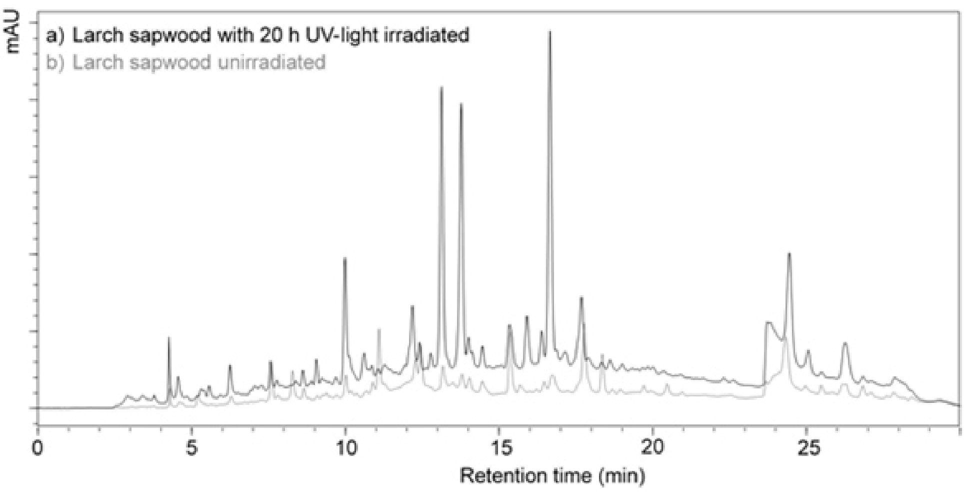

Figure 4.5: HPLC chromatograms (λabs = 280 nm) of unirradiated and 20 h UV-light irradiated of larch sapwood (Wagner et al. 2020b)

(e.g. methanol) (Wagner et al. 2019).

Only the hydrophilic wood extractives were soluble in the unirradiated wood by using the solid-liquid extraction method, therefore, the peaks height of extracts from non-irradiated samples were smaller than the UV-light irradiated samples. Through the UV-light irradiation the chemical compounds of the wood are alter, and photodegraded wood constitutes (e.g. low molecular weight substance of lignin) can be leached out by using water (Hon and Chang 1984; Kleinert 1970).

Not every peak from the irradiated samples are in accordance with the analytic from the untreated samples. There are some peaks, which were only found in the solution of the natural samples. On the other hand, some substances can be only detected in the irradiated samples, which indicates the photodegraded low molecular weight substances. Especially, the peaks of retention time at around 10, 13.1, 13.8, and 16.8 min are quite different compared to the peaks of non-irradiated wood samples. The reference substances of 3,4-Dihydroxybenzoate, 4-Hydroxybenozoate, and 4-Hydroxy-3-methoxybenzaldehyde (vanillin) show peaks at similar retention time at around 10, 13.1, and 16.8 min, respectively. Small differences in chemical structure result in different retention time and UV-absorbance. Therefore, a characterisation with reference substances are difficult because the degradation process occurs randomly and the pathways can vary so that the resulting degradation products are different. However, all reference substances own one or more phenolic hydroxyl groups in their chemical structure. This phenomenon is one assumption that photodegraded wood substances due to UV-light exposure were leachable by using water.

This leads to the interpretation as before that there was an alteration in the chemical composition of the wood samples and in further consequence the water-soluble wood extractives content was increased with possible degradation products after the UV-light exposure.

The degradation of wood and lignin is well documented in the literature (Fengel and Wegener 2003). Nevertheless, the whole process and differences in wood species have not been completely understood in detail yet. Therefore, the HPLC analyses and comparison with reference materials are difficult, because of the wide range of chemical differences of the substances. It is planned for the further research in this topic.

Table 4.4: The total phenolic content larch sap- and heartwood before and after UV-light irradiation (Wagner et al. 2020b)

Sample	Total phenolic content (mg GAE l^{-1})
larch sapwood without UV	3.99
larch sapwood with UV-irradiation	96.58
larch heartwood without UV	41.23
larch heartwood with UV-irradiation	129.46

Table 4.4 presents the result of the total phenolic content of unirradiated and 20 h aged larch sap- and heartwood after solid-liquid extraction with water. The findings show that the untreated samples have a lower concentration of phenolic contents as the samples which were treated for 20 hours under UV-light. Only the hydrophilic wood extractives were soluble in the unirradiated wood by using the solid-liquid extraction method, therefore, the amount of total phenolic content is low. On the other hand, after the UV-light exposure the total phenolic contents is highly increased. Thought the UV-light irradiation the chemical compounds of the wood are alter. The lignin of wood is not classified as extractives and these macromolecules are linked to cellulose and hemicelluloses by forming lignin-carbohydrate complexes (LCC) (Tribot et al. 2019) cannot be extracted by using only water with room temperatures (Fengel and Wegener 2003). However, photodegraded wood constitutes can be leached out by using water (Hon and Chang 1984; Kleinert 1970). The total phenolic content of the aged wood increased for sapwood samples around 24 times, whereas the increase for heartwood samples is around 3 times and showed significant difference between both sample groups.

The effect of photodegraded lignin on antibacterial activities of larch wood seems very realistic as different kind of pre-treated lignin polymers were used for increasing the antioxidant and antimicrobial properties of different bio-based films (Domenek et al. 2013; Yang et al. 2016a;b). One of them analysed the potential of kraft lignin for the capacity to inhibit the bacterial growth. The characterisation of softwood kraft lignin presents two distinct fractions, whereas the acetone soluble fraction is a less polymeric material with various chemical structure (Crestini et al. 2017). A similar fraction of water-soluble lignin degradation products may also result a better antimicrobial activities of aged larch wood samples. However, the influence of the new compounds of the antimicrobial activity of wood has not been analysed yet and were not proven before this study. The results show that the proposed assumption, that the wettability and the chemical compounds cause the higher antimicrobial activity of the irradiated wood surfaces, could be verified.

During the services life time of products made out of wood the chemical compositions is changing due to environmental conditions (e.g. light). However, the antibacterial activities were stable or/and improved due to the simulated ageing process of larch wood. Especially, the photodegraded substances in wood result in this effect mainly. These results could be used to functionalised the wood surfaces for more antimicrobial activities as an innovative nature inspired material property. It is important to understand how the wood compounds interact with different light sources (e.g. sun light) and the new established molecules different bacterial strains, for this more research is needed. A better understanding for the antimicrobial resistance is essential for the application of wood in hospitals and school, where the products are highly stressed and influencing variables may change.

4.2.2 Knotwood

This study deals with the analysis of wood chips from an industrial Austrian sawmill for the potential uses of wood extractives. The European larch (*Larix decidua* Mill.) is a commercially important species in the European Alpine areas and larch wood chips are normally produced as one of the by-products during the sawing process of wood (Atena and Schnabel 2018; Wagner et al. 2020a).

However, the outer part (mostly of it is sapwood) of the timber round wood cannot be used for the sawn wood and it is chopped to wood chips and used as thermal energy resources in larger part. As larch wood is not a preferred pulpwood species, since the amounts of resin and other extractives are large (Schnabel 2016). Furthermore, there is a non-negligible number of knots in the fraction of these wood chips, which are sorted out during the pulp and paper process. However, knots contain exceptionally large amounts of secondary plant metabolites (e.g., phenolic compounds) (Atena and Schnabel 2018). The amount of knot wood extracts is often higher than in the stemwood for many tree species, and the hydrophilic compounds exhibit strong bioactivity against oxidation, bacteria and fungi exploitable for various new applications (e.g., dietary supplements and cosmetic products) (Välimaa et al. 2007; Wagner et al. 2019).

A knot (knotwood) is indicated as the part of the branch which starts to grow from the pith and is hidden

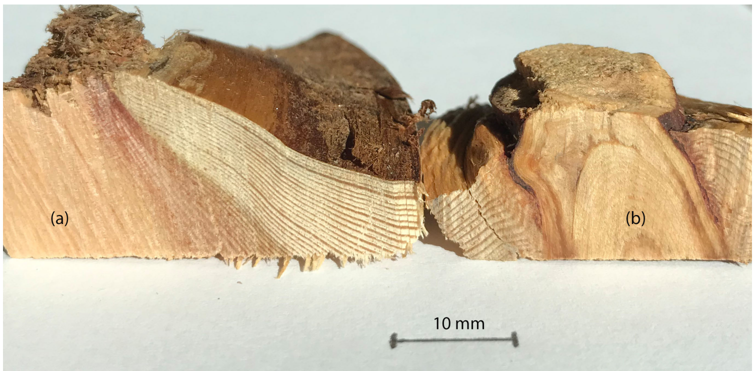

Figure 4.6: (a) One example of a larch wood chip with a sound knot and the intact connection between knot and stem wood; (b) a larch wood chip with a dead knot and the inclusion of bark as well as oxidized resin (Wagner et al. 2020a)

inside the stem. Two different types of knots can be distinguished, based on their function (Figure 4.6). Sound knots are connected with the stem wood and intact branches of the tree (Sjöström 1993). If the lifetime of the branches is over then the tree loses the branches, however, the knot will be protected against bacteria and fungi by the wood extractives. This knot is called a dead knot. As the stem grows further, it will gradually grow over the stump of lost branches, and finally the knot will be completely embedded in the stem wood (cf., Figure 4.6 (b)).

Table 4.5: Main component groups in acetone/water extracts of different larch wood materials by GC-MS (Wagner et al. 2020a)

Component groups	Larch sap- and heartwood (mg/g)	Sound knotwood (mg/g)	Dead knotwood (mg/g)
Carboxylic acids	0.026	0.139	1.011
Phenolic acids	0.116	1.666	0.081
Sugar	0.206	0.472	0.045
Fatty acids	0.140	0.483	0.047
Resin acids	0.079	0.186	0.014
Polyphenols	2.726	23.766	1.305
Sterols	0.016	0.235	0.011

Table 4.5 shows the total amounts of characterized component groups in acetone/water extracts in mg/g of freeze-dried powder of larch wood mixture (heart wood and sapwood), sound knots and dead knots. The amount of total extraction gravimetric yields of the acetone/water solution differed in a range from 3.38

mg g^{-1} of dried mixture wood (sapwood and heart wood), 27.13 mg g^{-1} of dried sound knotwood and 1.53 mg g^{-1} of dried dead knotwood (Wagner et al. 2020a). The hydrophilic extracts of the different wood parts contained various component groups. Based on results of the qualitative analysis all component groups (e.g., carboxylic acids) were found in each material. Nevertheless, there exist high differences in the quantitative results for the group of polyphenols.

The taxifolin and kaempferol amounts in acetone/water extracts of the sound knotwood (10.02 and 2.76 mg g^{-1}) were higher compared to the larch wood (1.46 and 0.73 mg g^{-1}) and dead knotwood (0.49 and 0.12 mg g^{-1}) (Wagner et al. 2019). Both compounds have an antioxidative potency and/or antimicrobial activities. Compared to the results of other studies, the determined amount of taxifolin and kaemperol were lesser in this study. The used wood chips for this study were from the outer part of the logs and presented dominantly sapwood. Nevertheless, the samples of the sound knotwood showed the largest amount of taxifolin and kaempferol compared with the larch wood.

The results of the hexane extract of the different tree sections showed differences compared to the acetone/water extracts. The amount of total extraction yields from the dead knotwood was the highest for 7.06 mg g^{-1} of dried material, followed by the sound knotwood with 5.05 mg g^{-1} and larch wood with an amount of 2.46 mg g^{-1} of dried materials (Wagner et al. 2019). Larixol and resin acids (e.g. isopimaric acid) were the main compounds of dead knot and sound knotwood.

Sound knotwood can provide more extraction yield amount than dead knotwood and larch wood, mainly sapwood with a small amount of heartwood, from outer part of the logs. While resin acids and larixol can be extracted more from dead knotwood samples compared to the sound knotwood and larch wood samples. Therefore, the results of chemical analysis of different parts of the trees are interesting for discovering wood tissues with a large extraction yield. The difference between both knot types was not focused upon in various studies of extractives and material properties. GC-MS analyses are often applied to determine the chemical composition of wood extractives (Laireiter et al. 2014; Wagner et al. 2019; 2020a). This method gives reliable results and is used for the detection of single components of natural extracts. However, in some processes less time-consuming analytical methods are important for the identification of different materials. Vibrational spectroscopy methods have much potentials for the sensitive and selective determination of organic compounds in wood and wood extractives (Schnabel et al. 2014). These methods are also relatively inexpensive to implement and easy to apply; furthermore, they are non-destructive techniques (Wagner et al. 2015). New insight into the different extract contents will help in the current efforts to use more environmentally friendly raw materials for innovative applications. Based on the chemical composition, three lead compounds were defined for the classification of the different wood raw materials. Furthermore, FT-NIR-RAMAN, FT-IR and FT-NIR vibrational spectroscopy methods were applied for development of classification between sound and dead knots, which is also needed for the transformation of the results from the laboratory to the industry (Schnabel et al. 2015). The connection between raw materials and extraction yields of the target values is important for the utilization of different current unused wood tissues (Schnabel 2016).

The wood powder was also analysed by an FT-IR spectroscopy method. Based on the results from the GC-MS analysis, small differences for the identification of larch wood, sound and dead knotwood know wood should be characterized. The different larch wood types as well as taxifolin and kaempferol as references were analysed for the chemical differences (Figure 4.7). The spectrum of larch wood differs significantly in some peak profiles from the knotwood spectra. This tendency is detected for all peaks at 2919, 2880, 1734, 1694, 1630, 1506, 1451, 1316 and 1264 cm^{-1}, although the differences cannot be explained in all cases by the consideration of the spectra of taxifolin and kaemperol compounds. The bands around 1736 and 1694 cm^{-1} did not show any IR signal of the references used and can correspond to carbonyl groups in different molecule arrangements (Pretsch et al. 2010). Furthermore, larixol shows vibrational infrared activities in

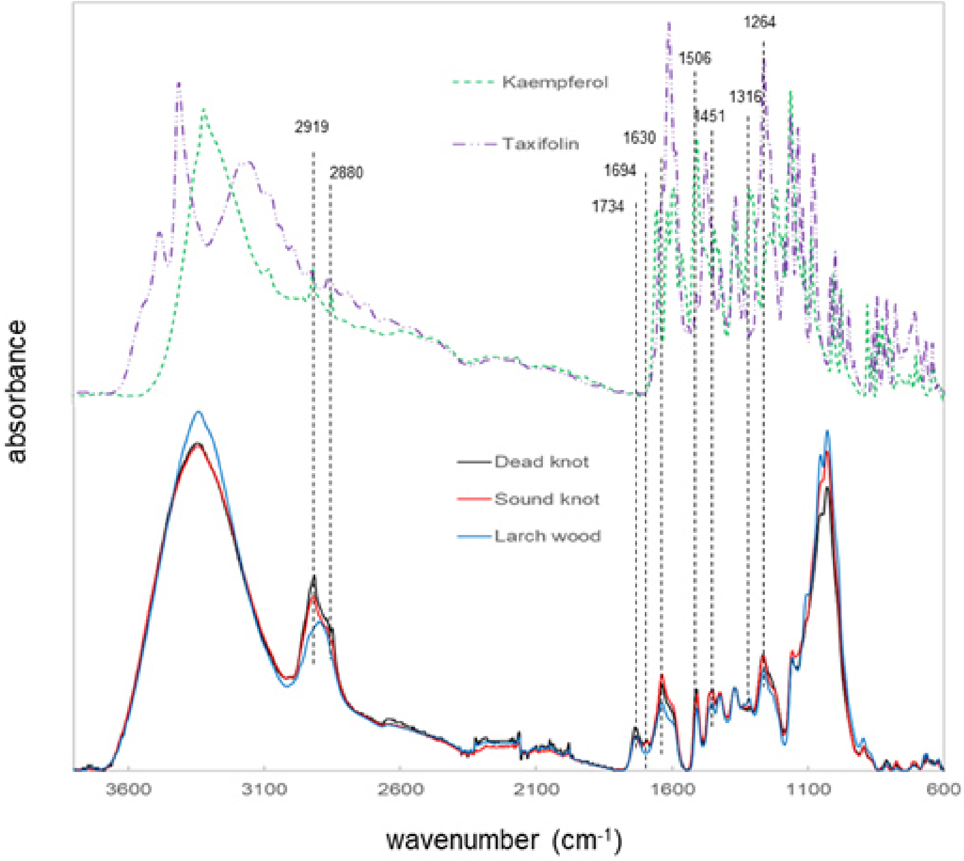

Figure 4.7: ATR FT-IR spectra of larch wood, sound and dead knotwood as well as kaempferol and taxifolin (Wagner et al. 2020a)

the range of wavenumber between 1750 and 1660 cm^{-1} (Wienhaus et al. 1960). Larixol and resin acids were predominantly observed in the different knotwood samples and can correspond to the observed bands in the spectra. The IR absorbance bands at 2919 and 2880 cm^{-1} changed significantly to each other, which correspond to differences in the amount of CH groups (e.g., CH2 and CH3) (Pretsch et al. 2010).
This fact could be assigned to higher number of bio-polymers in knotwood samples. The infrared spectra depicted that there is a possible differentiation between the larch wood samples and the knotwood samples. The direct measurements on the various wood samples gave more detailed information about the chemical differences compared to the wood powder, which was used for the RAMAN spectroscopy. With respect to these results, a method for classification of different wood samples with vibrational spectroscopy can be further developed. FT-NIR spectroscopy is very often used for this process (Schnabel et al. 2014; Wagner et al. 2015). The chemical information relating to three various larch wood types was obtained by using the FT-NIR spectroscopy (NIRS). Figure 4.8 shows the original spectra in the region between the wavenumber range 12500 - 4000 cm^{-1} of the larch wood, sound knotwood and dead knotwood samples. The original spectra present differences in the wavenumber range between 6000 and 5300 cm^{-1}, which are associated with wood extractives mainly from the polyphenols (e.g., taxifolin). The band at around 5970 cm^{-1} is corresponded to the aromatic groups in lignin and wood extractives (Schnabel et al. 2014) (Figure 4.9). Furthermore, a difference in the band at around 4640 cm^{-1} can be observed and could be associated with the combination of Caryl-H stretching and C=C stretching (Wagner et al. 2020a). For the C-H stretching vibration the NIR spectroscopy presents tree principal bands at around 4370 cm^{-1} the C-H stretch combination, around 5760 cm^{-1} the C-H stretch first overtone, and around 8350 cm^{-1} the C-H

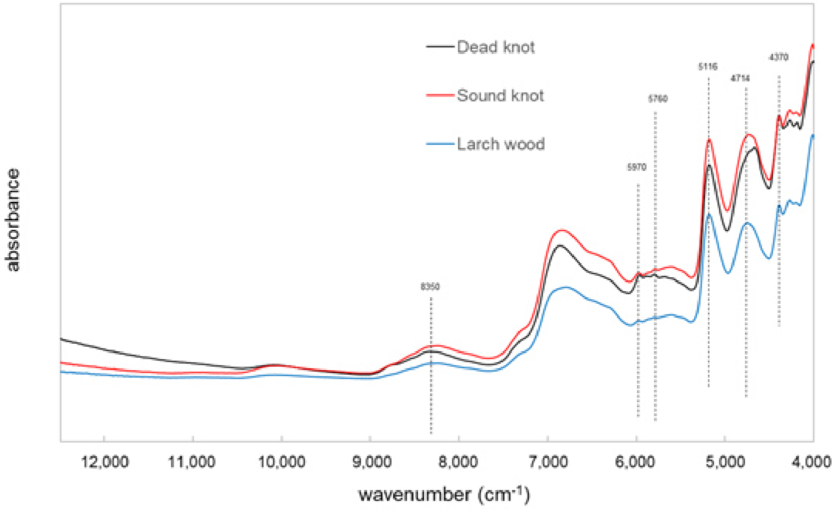

Figure 4.8: NIR spectra of larch wood, sound and dead knotwood (Wagner et al. 2020a)

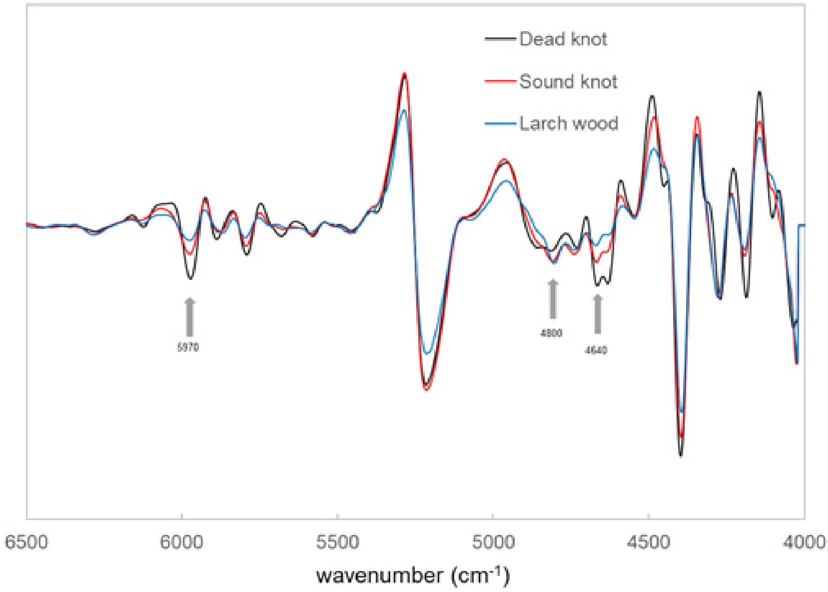

Figure 4.9: Second derivate FT-NIR spectra of larch wood, sound and dead knotwood (Wagner et al. 2020a)

stretch second overtone, which have significant correlations for the major C-H stretching bands at 2919 and 2854 cm^{-1} of mid-infrared spectroscopy of lipophilic extractives (Barton II et al. 1992). The results of the NIR measurement showed the higher absorbance of these bands for the sound and dead knotwood samples. Therefore, it seems that the two types of knots may be distinguished from the larch wood by the univariate analysis of the NIR spectra. Further analysis should be done to show the potential of NIRS for the classification of the various types of wood materials.

The data of the NIRS were used for the classification via principal components analysis (PCA). Figure 4.10 shows the distribution of two significant factors of the NIRS data. The two principal components explain the highest value of variance within these data and have a great impact on the differentiation of the various wood types used. Even though the PC 1 explained 93 % of the variance, whereas the PC 2 describes only 3 % of the variance, the combination of these two components was influenced by the two most important chemical substance groups: polyphenols and lipophilic extractives. The mixture wood has a lower number of polyphenols and fatty acids compared to the different knotwood types. The measured samples are clearly separated from the other materials (Figure 4.9). The sound knotwood samples presented a higher amount of taxifolin and kaempferol, whereas a lower amount of larixol than in the dead knotwood samples can be observed. Therefore, the score plot of the PCA shows also differences between sound and dead knotwood samples. Nevertheless, there are some areas without direct assignment of the samples (e.g. dead knotwood samples). The NIR measurements were performed on different surfaces from various selected samples of the larch wood, sound and dead knotwood. It can be assumed that the wood extractives distribution is

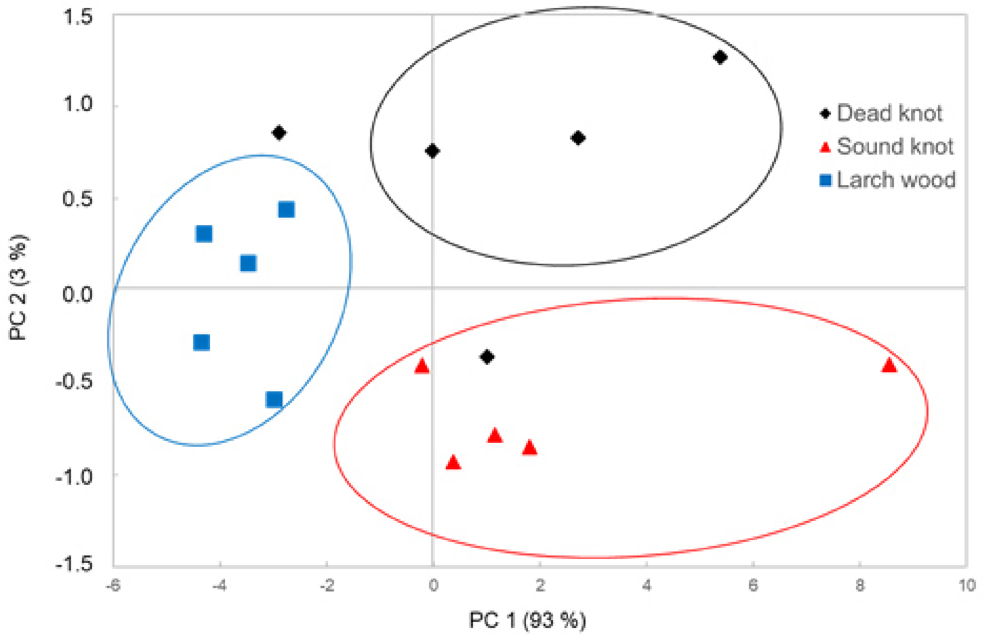

Figure 4.10: Principal component (PC) analysis score plot of untreated NIR spectra of various larch wood types (e.g. mixture wood, sound and dead knotwood) (Wagner et al. 2020a)

not homogeneous. Therefore, some areas of the same tree tissues have a high amount of wood extractives, whereas some areas have a low amount (Fengel and Wegener 2003).

The loadings of the PC 1 state high positive values for the wavenumbers 6870, 5970, 4640 and 4370 cm^{-1}, which are mainly corresponded to the phenolic compounds in wood extractives (Figure 4.11). The results of principal component analysis showed that the loadings of the PC 2 are contrary to the loadings of the PC 1 at the same NIR band at around 5116 cm^{-1} (water band of NIR signal), while the loadings of the PC 2 contribute to other wood extractives (e.g., lipophilic compounds) with lower moisture content within the samples. The results from the GC-MS were used to analyse three various spectroscopy methods for the potential of the characterization of wood extractives and identification of the three wood types. With all vibrational spectroscopy used, chemical differences between the wood and knotwood were observed and can be used to fulfil various research tasks. Moreover, the methods of multivariate statistics were applied to analyse materials by using the score plot and loadings of the PC analysis (Schnabel et al. 2014). These results demonstrate that a classification of various wood tissues based on the different chemical components (e.g., polyphenols and lipophilic substances) is possible with fast, non-destructive measurement methods. The current unused wood chips for material use can be separated into different fractions, where the extraction yields of the target substance (e.g., taxifolin) are higher compared to the other fractions. This method may serve as basis to establish guidelines for quality assurance control systems of this new approach for

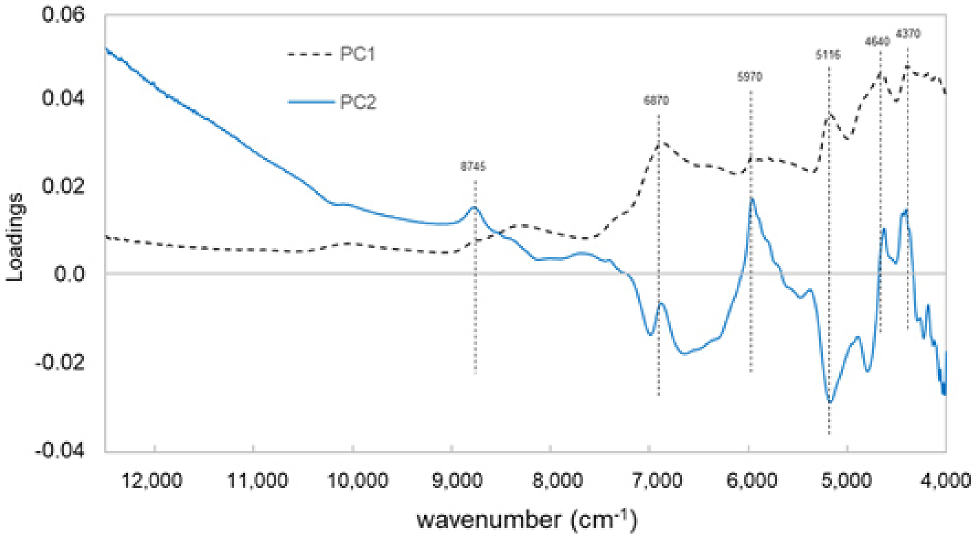

Figure 4.11: Loadings of the first two principal components (PCs) of near infrared spectra of various wood tissues (Wagner et al. 2020a)

material use to obtain the huge potential of bio-based products for innovative applications and for further efforts in the upscale from laboratory to industrial conditions (Atena and Schnabel 2018).

4.2.3 Bark

Bark contains unique compounds that are not present in or that vary from those in wood (Meints et al. 2016; Ravber et al. 2015; Wagner et al. 2019). For example, tannin from bark can be extracted (Wagner et al. 2018b; Tondi et al. 2012a) for their property as an adhesive and raw materials for foam as well as a substitute for crude oil products (Tondi et al. 2019; Sepperer et al. 2019; Tondi et al. 2013; Schnabel 2016). Certain bark materials contain large amount of phenolic substances, such as lignans, flavonoids, and stilbenes (Wagner et al. 2017; 2018a; 2019). The antimicrobial properties of several wood species, including larch, were previously tested by Laireiter et al. (2014). Larch bark extract resulted in an inhibition zone of 7.00 to 8.39 mm for *S. aureus* strain. These preliminary results showed that the way the bark is processed plays an important role for successful extraction of anti-microbial active substances (Schnabel 2016; Wagner et al. 2018a; 2016; 2020a). Therefore, bark is a potentially valuable source of natural antioxidants that can be used for further applications. Thus far, the characterization of the molecular compounds of the bark substances has not been performed yet and therefore, responsible compounds, which may cause these antimicrobial effects, were not identified. The presence of defined antimicrobial properties of bark extracts would allow for applications in the pharmaceutical and cosmetic industry. Laireiter et al. (2014) previously showed that larch bark, as well as larch bark extracts with methanol as solvent, had an antimicrobial effect. Also, the study from Wagner et al. (2019) shows that methanol extracts from larch bark materials affected the growth of *Staphylococcus aureus*, whereas the cold-water extracts did not show any antimicrobial activity. *Staphylococcus aureus* (*S. aureus*) are gram-positive bacteria, which can cause human diseases, and this pathogen can easily colonize the surface and form biofilms (Ming et al. 2017).

In addition, the qualitative and quantitative compositions of the two larch bark extracts with water and with methanol as solvent were analysed, which has not been performed thus far (Wagner et al. 2019). Regarding the qualitative and quantitative analysis of the extracts, in a first step the amounts of solid material after drying were determined. The total extraction yields differed in the range from 50.1 mg g^{-1} oven dried larch bark with methanol extraction to 22.0 mg g^{-1} with cold water extraction of 24 hours. Dissimilarity between this study and the literature references can be found in methodological alterations such as the extraction temperature, pressure and iterations, as well as in natural differences caused by three variations. The drying and extraction methods used in the present study were chosen to be gentle in order to avoid a loss of volatile compounds by heating or ad destruction of the molecules present in the extracts. Both solvents used have a similar polarity and are used to extracts polyphenols from different plant materials, which were the target group (Wagner et al. 2019). Nevertheless, single sugar molecules, alcohols, and acids can also be found in methanol and water extract of larch barks (Table 4.6).

Table 4.6: Main component groups in different larch bark extracts analysed by GC-MS (Wagner et al. 2019)

Component groups	Extractives in different solvents (mg g^{-1})	
	Water	Methanol
Long chain alcohols	0.00	0.59
Carboxylic acids	1.83	1.80
Single sugar	6.85	6.86
Fatty acids	0.05	0.23
Resin acids	0.01	1.38
Terpenoids	0.01	0.65
Polyphenols	0.69	4.05
Lipophilic substances	0.07	1.82

Compared to the different substance groups, the non-phenolic constituents add up to a higher portion of the extractives found. Similarities in the amount of larch bark extract compounds obtained with different solvents were determined for carboxylic acids and single sugars, whereas differences between both extracts were found for the quantitative amount of aliphatic alcohols, saturated and unsaturated fatty acids (e.g. lignoceric acid), resin acids (e.g. isopimaric acid), terpenoids, and lipophilic compound groups. For the polyphenols, the largest difference between the water and methanol extract was determined. The focus was placed on these substances, since polyphenols were shown to have antimicrobial effects towards different bacteria, yeast, and fungi. For further considerations, the group of polyphenols was divided into subgroups of flavonoids, lignans, and stilbenoids (Table 4.7).

Table 4.7: Tree component groups of polyphenols different larch bark extracts analysed by GC-MS (Wagner et al. 2019)

Component groups	Extractives in different solvents (mg g^{-1})	
	Water	Methanol
Flavonoids	0.043	1.62
Lignans	0.24	0.96
Stilbenoids	0.02	1.49

The water extractives containded (+)-catechin (0.39 mg g^{-1}) as the main compound in the flavonoid substance class, as well as taxifolin. However, the taxifolin amount (0.036 mg g^{-1}) in the water extract was higher compared to the methanol extract (0.019 mg g^{-1}). The major substance of the flavonoid group in the methanol extract was (+)-catechin (1.532 (mg g^{-1}). A very small amount of kaempferol (0.057 mg g^{-1}) was found via GC-MS analysis. Both compounds were shown to have antimicrobial activities from the literature (Rauha et al. 2000). However, the results were not found by using the different water and methanol extracts. In addition, the (+)-catechin alone cannot explain the mean inhibition zone of 8.2 ± 0.44 mm for *Staphylococcus aureus* (Laireiter et al. 2014; Wagner et al. 2019). Therefore, it seems that the concentration of (+)-catechin only in the water and methanol extracts was not adequate to affect the observed results. Interestingly, kaempferol is known for an antimicrobial activity against *Staphylococcus aureus*, when used at a high concentration of 1 mg μL^{-1} of the pure phenolic compounds (Rauha et al. 2000). However, the amount of kaempferol in the methanol extract was low in this study. Compared to results from Rauha et al. (2000), the determined kaempferol concentration was too low to induce a clear inhibition zone for *Staphylococcus aureus*.

The water extracts from the group of lignans contained lower amount of several substances compared to the methanol extracts. The two major compounds of lignans were isocariciresinol and lariciresinol. In addition, peroresinol could be detected with the GS-MS method used. The results from literature showed that lignins from spruce species have an antimicrobial effect against *Staphylococcus aureus* (Wagner et al.

2019). However, since spruce wood and acetone were used for the extraction in the previous study; it can be concluded that most likely different substances were extracted with acetone, water and methanol. The water and methanol extracts contained mainly the same lignans, but in different quantities. However, the methanol extract showed antimicrobial activities and thus, the results from the antimicrobial analysis are quite different and it could be concluded that the lignans do not have an effect.

The stilbenoids are the final group of polyphenols determined in the two different extracts (Table 4.7). The results of GC-MS analysis showed that the methanol extracts contain only astringin (3-O-β-glucosyl-3',4',5-trihydroxystilbene). Furthermore, a small amount of astringin was also found in water extracts. However, this substance represents the main difference between methanol and water extracts. The concentration of astringin in the methanol extract was approximately 75 times higher than in the water extracts (Wagner et al. 2019). Therefore, it can be assumed that astringin is mainly responsible for the antimicrobial activity of the methanol extracts from larch bark against *Staphylococcus aureus*.

To summarize the results of the larch bark, the antimicrobial activities of flavonoids towards different bacteria were not determined (Laireiter et al. 2014; Wagner et al. 2019). Therefore, these data in combination with finding from previous studies (e.g. Rauha et al. (2000)) show that the compounds of kaemprerol and astringin of the flavonoids and stilbenoids are responsible for the antimicrobial effects of the larch bark. Compared to the methanol extract, the water extract did not exhibit an inhibition zone against the microbes (Laireiter et al. 2014; Wagner et al. 2019). Therefore, it can be assumed that the amount of both substance groups is too small to influence the microbial growth. Alternatively, the combined effect of several compounds in the extract acting together towards an antimicrobial effect, could have resulted in the observed results.

The methanol extracts show some potential in the pharmaceutical industry. Moreover, water extracts of the bark material can play an important role in the cosmetic industry as well as chemical industry (Wagner et al. 2018a). The residual material from larch bark can be used for the production of added-value products; this is advantageous for the bio-economy and for reducing the dependence on fossil fuel based raw materials (Atena and Schnabel 2018). Therefore, the development of successful production process of value-added products shows great potential (Morandini et al. 2018).

Part B

Professional achievements

This part presents the progress and career development plans from the professional, scientific and academic point of view. Professional and teaching experiences include lectures at the Salzburg University of Applied Sciences and mentoring activities for various actors (e.g. policy level, business support units, intermediates, innovation agencies) at different international projects (e.g. FORESDA, InCIMa and CriculAlps) in many countries (e.g. Germany, Hungary, Italy, Romania, Serbia and Slovenia). This scientific research experience is reflected by the number and type of national and international projects, which the author managed as project leader and supervised as an expert. The transnational and cross-sectoral collaborations with the Department of Forest Products Technology & Timber Constructions at Salzburg University of Applied Sciences should be strengthened. This includes further collaboration with the Faculty of Wood Engineering in Brasov of "Transilvania" University of Brasov and further research institutes in the frame of various international project activities (e.g. Erasmus projects and/or mentoring sessions).

5 Experience in scientific activities

The foundation stones for the development of scientific activities were the author's diploma thesis at the Salzburg University of Applied Sciences in Kuchl (Austria) and the doctoral thesis at the Technical University of Munich at the Institute ”Holzforschung München” (Germany). Subsequently, a large number of national and international research projects were successfully approved for grants. The function of project manager or/and expert was taken over these projects, which are presented below:

- **3d-LeFaShape:** 3-dimensional leather material surfaces made of regenerated leather for interior and furniture design, funded by the Austrian Research Promotion Agency (FFG), Sep. 2010 - Aug. 2012; The role in the project was technical project manager. The objective of the project was to develop materials for interior products based on by-products from the leather industry. The challenge in the development process of the material was to match the components used with the manufacturing process. The process and material developments for innovative product ideas and their analysis of market potential were focused of this project. The project was conducted on industrial level and four German and Austrian companies were involved in the manufacture of new materials and products. Based on these project results a new project entitled FLAME was developed.

- **FLAME:** Integrated (market) development of natural, flame-retardant materials from leather shavings for increased fire safety, funded by the Austrian Research Promotion Agency (FFG), Dez. 2012 - Nov. 2015; The role in the project was project manager. The aim of the cooperation and networking project "FLAME" was the (market-) development and industrial feasibility of new, fire-retardant 2D panel materials based on leather chips for increased fire safety. The approach was to use national and European resources currently available in leather production (leather shavings waste) to offer sustainable alternatives to ligno-cellulose raw materials, which are becoming scarce; other objective was to analyse the leather in fire-protective applications;

- **adTro-net:** Batch adsorption drying network Austria, funded by the Austrian Research Promotion Agency (FFG), Apr. 2013 - Mar. 2017; The role in project was expert and project coordinator for the partner Salzburg University of Applied Sciences; The aim of the project was to establish a competence network for adsorption drying in batch operation and to gather the leading research institutions in the field of industrial drying and strengthen the research in terms of the drying of food, agricultural products and wood. The energy consumption and increase of the quality of drying processes were the main objectives of the project.

- **SIHGA-Connector:** This project was funded by the Austrian Research Promotion Agency (FFG) in cooperation with an Austrian company, Oct. 2014 - Sept. 2015; The role in the project was the expert from the Salzburg University of Applied Sciences. The main objective of the project was to design a removal lifting system for a short and save transport of large-size cross laminated timber samples.

- **BioInsPa:** Up-cycling of straw - Biogenic insulation board - Development and possible applications, funded by the Austrian Research Promotion Agency (FFG) in cooperation with University of Applied Sciences Upper Austria in Wels and five national companies, Sep. 2012 - Aug. 2015; The role in this project was the project manager of the whole project consortia. The main objective of the project was to design new product concepts, prototypes and component systems for building insulation based on cereals straw as raw material. Pre-treatments of the plant fibres have potential to increase their homogeneity and to improve the material properties in order to increase the applications of these materials e.g. for insulation products.

- **BioSubTro:** Recovery of bioactive substances during wood drying, funded by the Austrian Research Promotion Agency (FFG) and three Austrian companies, Apr. 2016 - Mar. 2019; The role in this project was the project manager of the whole project consortia. The key element of this project is the possible use of natural substances and agents for high added-value products in the cosmetics industry. The wide utilization of recyclable components from condensates released during the drying process of biomass may be an appropriate response to the increasing scarcity of resources and may help to encourage the development of sustainable and natural products.

- **Splint:** This project was funded by the Austrian Research Promotion Agency (FFG) in cooperation with one national University of Applied Sciences and company, May 2016 - April 2017; The role in the project was the expert form the Salzburg University of Applied Sciences. The main objective of the project was to characterise the material properties of an innovative product after UV-light irradiation for simulation of the aging effects.

- **ILBitZ:** Innovative solutions through biomimetics in the transnational interaction of business and science, funded by the European Commission under Interreg Bavaria and Austria in cooperation with Deggendorf Institute of Technology (Germany) and two Austrian innovation and technology organisations (Innovation Service for Salzburg and Business Upper Austria), Sept. 2016 - Feb. 2020; The role in this project was the expert and project manager from the Salzburg University of Applied Sciences. The aim of this project is to foster the biomimetics as innovation and products development to small and medium enterprises. The knowledge exchange of biomimetics is focused on different topics and levels.

- **SMF:** Salzburg's multifunctional facade, funded by the Government of Salzburg and a national company, Aug. 2017 - Jan. 2019; The role in the project was the project manager from the Salzburg University of Applied Sciences. The aim of this project is to develop a building wall element with wooden parts for heating and cooling of a building.

- **FORESDA:** Forest-based cross-sectoral value chains fostering innovation and competitiveness in the Danube region, funded by the European Commission under the Danube Transnational Programme (DTP) in cooperation with Twenty organisations from thirteen countries[§], Jan. 2017 - Jun. 2019: The role in the project was the expert and project manager from the Salzburg University of Applied Sciences. The main focus was on the increased effectiveness of collaborative research and innovation activities on the forest-based industries in the Danube area. For leading to the emergence of new cross-sectoral value chains primarily in the areas smart and sustainable construction and furniture, innovative bio-based products and materials and energy efficiency were established.

- **InCIMa:** Smart characterization of smart materials, funded by the European Commission under the Interreg Italy - Austria programme in cooperation two different research institutes from Italy (Elettra Sincrotrone Trieste) and Austria (University of Salzburg), Apr. 2017 - Sep. 2019; The role in the project was the expert and project manager from the Salzburg University of Applied Sciences. The objective of this project is the creation of a cross-border de-localized infrastructure for the synthesis and characterization of intelligent functional materials at the nano-, micro- and macro-level using

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[§] www.interreg-danube.eu/approved-projects/foresda/partners state-of-the-art spectroscopic imaging and mapping techniques. These exploit the advantages of a wide range of both conventional radiation and synchrotron light from far infrared to hard X-rays.

- **CirculAlps:** Innovation to foster sustainability and circular economy in Alpine forestry value chain, funded by the European Commission under the Alpine Region Preparatory Action Fund (ARPAF) in cooperation with four research project partners from three countries[‖], Jan. 2018 - Dec. 2019; The role in this project was the project manager. This project aims at promoting circular and bio-economy in terms of research-based approaches in the Alpine timber section. New timber value chains of innovative products should be analysed.

- **InCIMa4:** Smart characterization of smart materials, funded by the European Commission under the Interreg Italy - Austria programme, Sep. 2019 - March 2022 in cooperation with six research partners from Italy and Austria[**]; The role in the project is the expert and project manager at the Salzburg University of Applied Sciences. Based on the formerly InCiMa project, the analysing methods and collaboration with SMEs should be continuously improved. The objective of this project is the creation of a cross-border delocalized infrastructure for the synthesis and characterization of intelligent functional materials at the nano-, micro- and macro-level using state-of-the-art spectroscopic imaging and mapping techniques. These exploit the advantages of a wide range of both conventional radiation and synchrotron light from far infrared to hard X-rays. The results will be used for the knowledge transfer to the industry. Further collaborations between the research institutes and the enterprises will be developed.

Many of the results in these research projects were presented in different scientific publications in journals and on conferences as well as in this habilitation treatise (see part A). All these studies supported the objective of new products innovations through transformation of raw materials to high added-value products in the frame of bio-economy. Furthermore, these projects show the further scientific development of the author and demonstrate the scientific area from strategic orientation and planning to project work and various analysis as well as interpretations to foresighted scenarios of the obtained results and innovations. Also, the possibility of international and cross-sectoral collaborations between various project partners at different levels (e.g. policy and business support units) could be seen, not only the work with other universities or research centres were shown, but also a close collaboration with companies were demonstrated within these projects.

[‖] www.alpine-region.eu/projects/circulalps

[**] www.incima4.eu/de/home/

6 Experience in education

Due to the academic studies and the acquired knowledge of the author in wood science and technology as well as in timber constructions and mathematical and statistical methods, a large number of different lectures and seminar with students can be proven and range from a mathematical/statistical basic lecture on current subject-specific topics of wood science.
This knowledge transfer from the research to the education is very important during the study time. The students need the right skills and competences to fulfil the current requirements within the industry. The author's academic activities started within the diploma study degree programme in the year 2004. After the Bologna process the diploma study degree programme (4 years) was reorganised by bachelor (3 years) and master degree course of 2 years. The description of various courses taught by the author at 3 study directions of forest products technology, furniture and interior design and timber constructions of SUAS at Campus Kuchl since 10 years follows:

- **Wood modification:** This lecture/course for diploma students (6th semester with 2 SWS) and bachelor students (4th semester with 1 SWS) deals with the state-of-the-art description of wood modification based on fundamental ideas, processes and applications of modified wood. After a short introduction of wood properties and problems of wood concerning on the chemical level, the important knowledge of the currently used wood modification processes (e.g. acetylation and furfurylation) or research-based processes (e.g. electron beam irradiation) is presented. Moreover, the properties of different products and application of the modified wood is discussed with the students. The sustainability and environmental impact of construction and interior materials were including in this lecture.

- **Funding and project acquisition:** This course for master students (3th semester with 2 SWS) give an insight about regional, national and international research funding programmes. The students are be taught to develop and write a research proposal concerning all required steps from the project idea generation to cost calculation as well as communication with internal and external project stakeholders. Moreover, each group of few students has to write a full research proposal for a national research funding programme.

- **Statistic and process analysis:** This basic course for bachelor (4^{th} semester with 2 SWS) and master (3^{th} semester with 2 SWS) students deals with basic knowledge and the application of descriptive and test statistic (e.g. t-test and ANOVA) for material research tasks and writing of their bachelor and master thesis. Different examples from the field of wood research and wood processing industry are analysed and discussed.

- **Research methods and design:** This lecture for graduate students (2^{th} semester with 2 SWS) give a detailed overview of different research design and comprehensive methods for data analysis (e.g. Principle Component Analysis) for the planning their own master thesis as well as best practice examples of successful project implementations. Different examples from the field of wood research and wood processing industry are analysed and discussed.

- **Bio-based materials:** Bio-based materials and products play an important role in the scientific and industry sectors. This course for bachelor students (4^{th} semester with 1.5 SWS) deals with the detailed description of bio-based raw materials (e.g. straw and leather), which can be used to produce new materials and/or products as well as building blocks for further applications. The discussion concerning the sustainability and environmental impact of construction and interior materials were included in this course.

- **Current topics in wood science:** The graduated students (2^{th} semester with 2 SWS) get a detailed overview about the current topics in the research at the institute. The current situation of the research work (e.g. challenges, research questions) and further development as well as the results of the research are discussed with the students. Therefore, the scientific exchange and discussion process for the students are promoted.

- **Projects:** In this category there are many different undergraduate ($1\text{-}6^{th}$ semester with 2 SWS) and graduate ($1\text{-}4^{th}$ semester with 2 SWS) student projects. The supervision of these projects is multivarious and ranges far from introduction for working in a chemistry laboratory to material, product

> and process development projects to specialisation projects for the bachelor, diploma and master thesis in cooperation with or without enterprises. The discussion concerning the sustainability and environmental impact of construction and interior materials were included in these seminars. The work is mostly done with a small team of students and has interdisciplinary character.

There was maintained a close connection between the academic and research activities by collaborating with students on different topics that are highly relevant in various research areas like wood modification, drying processes, biomedical analytic as well as methods for characterisation of material properties. Nevertheless, for early stage researchers a training session of the introduction of different characterisation methods of wood by using NIR spectroscopy was held in the frame of first COST Action FP1006 workshop - Basics for chemistry of wood surface modification in Kuchl on April, 24th to 27th 2012. Moreover, the author was a mentor for two Short Term Scientific Mission (STSM) of thermal modification of Spanish chestnut and the analysing of the interaction of coatings and surface of thermally modified wood in the year 2012 and 2018, respectively. Furthermore, numerous bachelor, diploma and master theses (number of theses are 9, 4 and 7, respectively) in a broad range of the utilization of natural resources for innovative applications and process optimization regarding the bio-economy were supervised and co-supervised at the Department Forest Products Technology & Timber Constructions of SUAS Campus Kuchl between 2004 an 2020. Also, one PhD student concerning the characterisation of wood extractives from the University of Salzburg has been actively co-supervised at the Department Chemistry and Physics of Materials of University Salzburg since 2018. The cooperation based on educational issues and teacher mobility with the University of Belgrade started with a funded Erasmus+ KA107 project in the year 2019.

7 Future research and academic activities

The transformation of sustainable raw materials to high added-value products in the frame of bio-economy is also important in the future. However, as fossil resources are still relatively cheap compared to bio-based resources, no external incentive exists to strengthen cooperation between producers of wood-based residues and eventual processors. In addition to sustainable innovations and breakthrough research and development, education is also in the focus of interest for people with the right skills and competencies for solving the problems in the future.

7.1 Record

The future of a circular-/bio-economy is strongly connected to the material use of natural resources and residues in local regions. Regions across the countries in EU are currently establishing their own development paths towards more sustainability or a comprehensive bio-economy strategy, or have done so already (Bezama 2016; Zabaniotou 2018). The potential of innovation of the material use of bio-based materials and residues thus hinges on the design and implementation of these strategies and how they could be connected, eventually (Morandini et al. 2018). Products, applications and research projects offer competition in a wide number of markets. When bio-based materials and residues (e.g. straw or wood) can be effectively and efficiently sourced (Thorenz et al. 2018), companies are able to provide alternatives to fossil-based products and materials. Markets, where competition from wood-based materials exists, include among others granules for injection moulding, fuels, landfill material made from paper mill residues, and/or carbon-rich soil additives (Schnabel et al. 2020a). Therefore, the use of residues from forestry, agriculture and wood processing potentially provides added value for foresters, farmers and the wood-working sector as a whole (Tondi et al. 2012b; Wagner et al. 2016; 2017).

Clearly, the market potential of bio-based residues lies in innovation beyond the classical value chain

(Atena and Schnabel 2018). What is therefore needed is the successful connection of primary biomass producers in remote areas with processors and producers of intermediate products and applications (Schnabel et al. 2020a). As of today, only wood that adheres to specific quality criteria can be processed into timber used for construction or carpentry, furniture and frames. Therefore, huge potential for innovation and marketisation lies in the valorisation of lower quality wood in bio-refineries and bio-product mills, for example (Atena and Schnabel 2018). To this end, a network of decentralised processing facilities across remote areas would be best to cater for the needs of biomass producers, biomass processors and manufacturers of bio-based products alike (Schnabel et al. 2016b; Atena and Schnabel 2018).

7.2 Education

The didactic advancement is a very important part of the author's career, which continues successful the international development of exchanges within different research institutes and universities by implementing the study of bio-economy for fostering the transformation of unused materials to high value added products in study degree programmes (e.g. Faculty of Wood Engineering (FIL) of the Transilvania University of Brasov (UTBv)), which attracts student exchanges from the EU through Erasmus(+) programmes. The author has not only a large and up-to-date network of professional relations with the EU and nonEU faculties, but also with external research and educational institutions that can provide support this special initiative.

In connection with a wider scientific content, the author would like to support a training centre for studies within the FIL of UTBv - Advanced in Wood Science and Technology, oriented to specialists with higher studies in the wood industry in Romania and in the neighbouring countries. In the last decades it has been observed that the wood processing industry is confronted with various new challenges like scarcity of resources, human potential (e.g. brain-drain) and environmental issues. Therefore, the effective utilisation of materials and energy resources is becoming an increasingly important challenge for the economy and society (Pieratti et al. 2020; Schnabel et al. 2020a). There are various approaches to solve this challenge. One option for the companies can be to react to this situation and to develop new materials and products from available and sustainable natural resources. Improvements in efficiency (cost effectiveness) as well as the modernisation of existing methods and technologies are important and will also be crucial for leading companies. Resource efficiency involves the consideration of all the stages of the life cycle of a product, including extraction of resources, production, the use of the product and recycling (D'Amato et al. 2017).

On the other hand, the academia can work on different levels to transfer the knowledge and technology into enterprises. Besides the classical basic research projects, they have carried out applied research projects in collaboration with companies (Lovric et al. 2020). Nevertheless, the knowledge transfer to the student is the most important goal of educational institutes, and this can support the development of innovative products and thinking based on circular economy (Zabaniotou 2018). Young people work in well-equipped laboratories during the study degree courses and can develop their own scientific skills, personality and experiences. Many of the students are multiplicators; they will work in the future for companies and they are enthusiastic for development of new bio-based and easy to recycle materials and products as well as their own business. It is not astonishing that start-ups were built up from students, as they have good knowledge, innovative ideas and access to technical equipment for the initial phase of establishment. However, there are also some studies in which the educational structure of academia does not meet the actual needs of the quick changeable economy and the implementation of important parts of the entrepreneurship (e.g. business plan model) especially for the forestry sector of various countries can be done in different study degree programmes (Schnabel et al. 2020a).

Another way is to support especially the transformation from students to the managers of their own business by using a business incubator lab for the wood industry, where the first idea for a start-up can be developed. Such a lab provides the needed resources and mentorship for different specific aspects (e.g. technology, business and law) for the development of start-ups and is related to the universities and/or research institutes.

Based on this approach the author would like to participate in the following actions of didactic activities:

- Modernization of course materials, reworking of didactic concepts for laboratory work and lectures, but also in e-learning technologies for long distance learning

- Support of the FIL - Advanced in Wood Science and Technology with expertise of material and process transformation related to bio-economy approach, both for laboratory work and seminars

- Fostering the entrepreneurial activities of students and/or academic members for the transformation of research results to industry

- Support the exchanges of EU students and teachers, but also from abroad, to the FIL study program, either through Erasmus(+), but also in the variant of some academic years

- Training of doctoral students from Austria and abroad in daily and distance learning programmes in European languages, related to current topics, not only in the field of wood science and technology, but also in materials from non-wood resources and bio-economy approaches

The activities will involve the elaboration the relevant core topics for developing a sustainable architecture/method for the knowledge and technology transfer of possible solutions of challenging issues (e.g. natural resources), products and common innovation process to the students. It is important that the students have an insight of different sectors, work interdisciplinary within the student projects and understand the other project members. This approach is supported by using small projects of developing a new business model typically for this economy sector to transfer these experiences to the involved students.

7.3 Scientific activities

An important way to continue the development of this kind of scientific research is the diversification of the field, modernisation of methods and constant gaining of knowledge in various fields within economy, ecology, science and technology. To achieve these objectives, it is important to continue the interdisciplinary forest research, transfer and bundling of different directions and dissemination of the research results. During the author's scientific work, the author built up a network with various higher education and research institutes, technology and innovation agencies was well as business support units in Austria, Germany, Italy, Slovenia, Croatia, Romania, Serbia, Sweden, Bosnia-Herzegovina, Bulgaria and Turkey. The author's connections can be used efficiently both for UTBv and for the creation of new kind of research activities. Also, in this sense, the author will claim some research grants within the cooperation of FIL and other university from EU member states as well as third countries in Eastern Europe and Near East, which would attract extraordinary attention and thus intensification of the exchange and strengthening of the role of the institute in the region.

The circular economy and bio-economy are very important for the future and huge research fields concerning to the upgrade of unused materials to valuable products for the consumer needs. To support this compilation a good collaboration between different industry sectors and research institutes are needed so that the understanding of various topics is guaranteed. The disciplinary approach to work together within different scientific areas is crucial for the solving new challenges concerning the various unused materials, the increasing of energy and resources consumptions. The addresses of new research topics and the choice of partnerships in material science, process technology, design and economy are curial for the future. In connection with a wider range of research, the author will continue to be concerned about:

- Increasing of the visibility of research activities of the FIL group of UTBv, whose regular publication included also the results of his one research in ProLigno journal (vol/year)
- Initialisation of international research projects in cooperation with the FIL to strengthen the exchanges of knowledge and technology from different countries
- Attraction of research funds by proposing and participating of the FIL for developing new directions of scientific field and for the formation of mixed teams with specialists from abroad
- Support young colleagues from the FIL in the formation of research teams that are oriented towards the requirements of the industry, with the necessary expertise and rapid response to ensure the continuation of the research themes with the results consistent and diverse
- Respond positively to requests to be the reviewer and/or editor of indexed journals and conferences or chapters in reference books of national and international publishers
- Support the development of research field of bio-economy and transformation for high added-value products by review activities for scientific journals

Proposals for further research are based on the subject of the transformation of bio-based raw materials to high added-value products related to materials for natural cosmetic, bio-based polymers and/or medical applications (Tondi et al. 2019; Tondi and Schnabel 2020; Wagner et al. 2019; 2020a). The old way to thinking and also classical scientific boarder have to burst and a break through should be possible with the future bio-economy approach. New thinking and innovative products as well as processes need modern methods for evaluating of different scenarios.

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