TALLINN UNIVERSITY OF TECHNOLOGY
SCHOOL OF ENGINEERING
Department of Materials and Environmental Technology

# ASPEN PLYWOOD BONDING CHARACTERISTICS WITH PHENOLIC ADHESIVES

## HAAVAVINEERI LIIMÜHENDUSE UURIMINE FENOOL-LIIMIDE PUHUL

MASTER THESIS

| | |
|---|---|
| Student: | Silvi Treial |
| Student code: | 182546KVEM |
| Supervisor: | Triinu Poltimäe, senior lecturer |

Tallinn, 2020

## AUTHOR'S DECLARATION

Hereby I declare, that I have written this thesis independently.
No academic degree has been applied for based on this material. All works, major viewpoints and data of the other authors used in this thesis have been referenced.

"......." august 2020

Author: ..............................
*/signature /*

Thesis is in accordance with terms and requirements

"......." august 2020

Supervisor: ………………………..
*/signature/*

Accepted for defence

"......." august 2020

Chairman of theses defence commission: ...............................................
*/name and signature/*

**Department of Materials and Environmental Technology**

# THESIS TASK

**Student**: Silvi Treial, 182546KVEM

**Study programme:** KVEM12/18 Technology of Wood, Plastic and Textile; main speciality: Wood Technology

**Supervisor:** senior lecturer, Triinu Poltimäe, +372 620 2910

**Thesis topic**:

Aspen Plywood Bonding Characteristics with Phenolic Adhesives

Haavavineeri liimühenduse uurimine fenool-liimide puhul

**Thesis main objectives**:

1. Investigate the use of aspen for plywood manufacturing when using veneers from different log-soaking temperatures.
2. Testing of aspen plywood bonding characteristics and comparison with a control group - birch plywood - when both are manufactured at the same controlled laboratory conditions using the same phenolic adhesives.

**Thesis tasks and time schedule:**

| No | Task description | Deadline |
|---|---|---|
| 1. | Carrying out literature research | 03.2020 |
| 2. | Veneer testing | 03.2020 |
| 3. | Manufacturing of plywood panels | 05.2020 |
| 4. | Plywood testing | 05.2020 |
| 5. | Conducting analysis and formulating results | 06.2020 |
| 6. | Writing and formatting master thesis | 08.2020 |

**Language:** English **Deadline for submission of thesis:** "……." august 2020 a

**Student:** Silvi Treial ………………… "……." august 2020 a
*/signature/*

**Supervisor:** Triinu Poltimäe…………………….. "……." august 2020 a
*/signature/*

**Head of study programme:** Jaan Kers ………………… "……." august 2020 a
*/signature/*

## CONTENTS

INTRODUCTION ........................................................................................ 6

1. LITERATURE OVERVIEW ..................................................................... 8

  1.1 Description of aspen .......................................................................... 8

  1.2 Plywood production ...........................................................................10

  1.3 Wood adhesion ...................................................................................12

    1.3.1 Wetteability and surface roughness of wood .....................................13

    1.3.2 Adhesion theories ..........................................................................15

  1.4 Phenol-formaldehyde resin .................................................................16

    1.4.1 Resole-type PF resin .......................................................................17

    1.4.2 Lignin-phenol-formaldehyde resin ....................................................18

2. MATERIALS AND METHODS ..............................................................20

  2.1 Materials ............................................................................................20

    2.1.1 Veneer ..........................................................................................20

    2.1.2 Adhesives ......................................................................................21

  2.2 Methods .............................................................................................22

    2.2.1 Veneer testing ...............................................................................23

    2.2.2 Making of plywood .........................................................................26

    2.2.3 Plywood testing .............................................................................27

3. RESULTS AND DISCUSSION ............................................................29

  3.1 Surface roughness ..............................................................................29

  3.2 Contact angle .....................................................................................34

  3.3 Thickness, density and glue consumption ...........................................37

  3.4 Bond shear strength ..........................................................................39

  3.5 Conclusions .......................................................................................43

SUMMARY ...........................................................................................45

KOKKUVÕTE ........................................................................................47
LIST OF REFERENCES .............................................................................49

# INTRODUCTION

Producing plywood is an excellent way to use the wood material to the fullest as very little goes to waste when the log is rotary peeled. In recent years the manufacturing of plywood has gained more importance in Estonia as there has been a substantial amount of investments made into new and existing factories. If all the relevant factories would produce veneer and plywood at full capacity in Estonia, the output would be around 290 000 $m^3$ a year. The increase in plywood manufacturing between years 2018 and 2019 was +26% with an annual production volume of 134 500 $m^3$ in 2019. [1] [2]

As most plywood in Northern Europe is produced from either birch or different softwoods, or a combination of them, there might be interest to research other wood species for plywood manufacturing. One suitable species might be aspen, for it’s ease of processing, light colour and reasonable amount of raw material and having a lower price tag than birch for example. In 2019 the average price for a birch log intended for the veneer/plywood industry was was 107.91 EUR whereas a regular aspen log was priced at 43.75 EUR. [1] [3]

Not much research has been carried out to investigate the suitability, quality and specific properties of aspen veneer when considering it as a raw material for plywood. When manufacturing plywood, many steps and parameters need to be monitored in order to achieve the best quality in the final product. These include the soaking conditions, peeling, drying, gluing and pressing. Perhaps the most important to consider is the strength of the adhesive bond in the final product of a plywood panel and factors affecting it during production.

The aim of this thesis is to investigate the bonding characteristics of aspen plywood with phenolic adhesives. The main objectives are:

1. Investigate the use of aspen for plywood manufacturing when using veneers from different log-soaking temperatures.

2. Testing of aspen plywood bonding characteristics and comparison with a control group - birch plywood - when both are manufactured at the same controlled laboratory conditions using the same phenolic adhesives.

The properties when investigating aspen plywood bonding characteristics in this thesis are: veneer moisture content, veneer surface roughness, veneer surface contact angle, plywood thickness, plywood density and plywood bond shear strength. For the adhesives

used, in addition to the most common phenol formaldehyde adhesive, a lignin phenol formaldehyde adhesive is also tested to see how well it compares to the phenol formaldehyde adhesive.

The thesis is divided into three main chapters. The first chapter gives an overview of the literature where it briefly describes aspen wood, the plywood production process, wood adhesion and two types of adhesives: phenol formaldehyde and lignin phenol formaldehyde. The second chapter describes the materials and methods. The third and final chapter presents the results and a discussion of the results.

Keywords: aspen, plywood, bond shear strength, phenolic adhesives, master thesis

# 1. LITERATURE OVERVIEW

## 1.1 Description of aspen

Aspen (*Populus tremula*) is one of the most common hardwood species in Estonia's forests and also in the world [4]. It is a biologically adaptable species that is capable of growing in many different conditions, but it does require a lot of light and good soil conditions [4]. Estonia is situated in the optimal growth area of aspen [4]. Figure 1.1 demonstrates aspen species' growth area in the world [5].

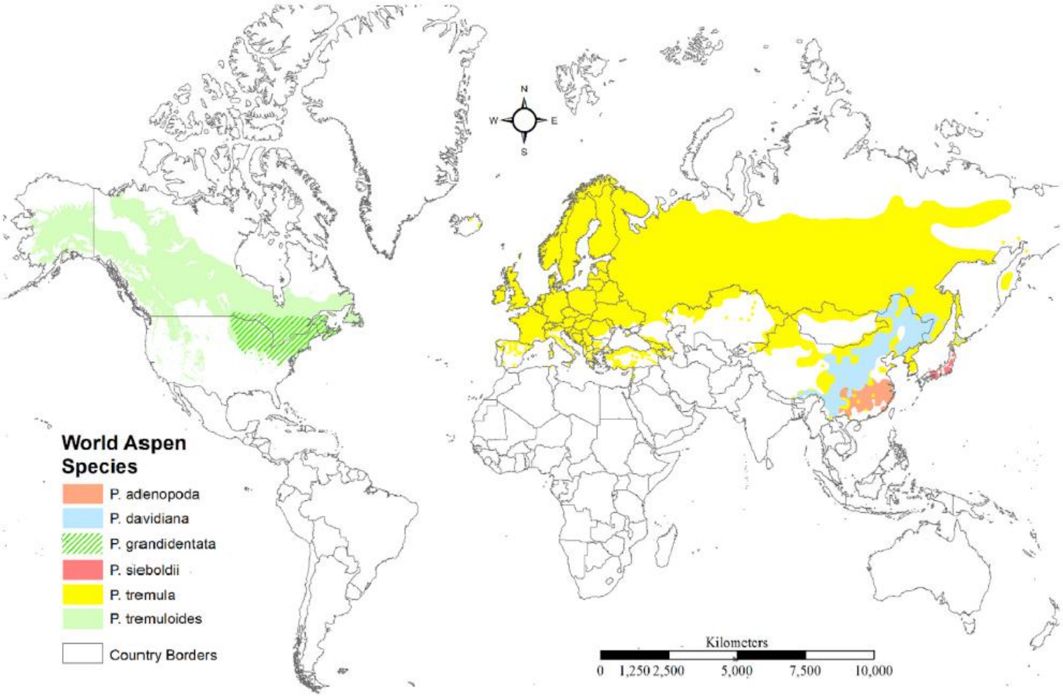

Figure 1.1 Aspen's growth area in the world [5]

In Estonia aspen makes up of 8.6% of volume on forest land and it is surpassed by birch (20.7%), spruce (24.8%) and pine (30.2%). The growing stock ($m^3$) and felling volume ($m^3$) of aspen is 7.5% and 5.6%, respectively. In each case the same wood species surpass aspen, with pine and spruce being the top two in statistics, followed by birch. It could be concluded that aspen is the 4th most important wood species in Estonia. [6]

Aspen's wood is uniformly light, sometimes with a slight greenish tint. The cellular make up is 55% libriform fibers, 34% vessels and 11% rays. The chemical description of average aspen wood consists of 0.32% ash, 18.24% lignin, 24.47% pentosans, 47.11% cellulose. Aspen is a diffuse porous hardwood species and its microscopic structure can be seen in Figure 1.2 where there are tangential, transverse and radial views. [4] [7]

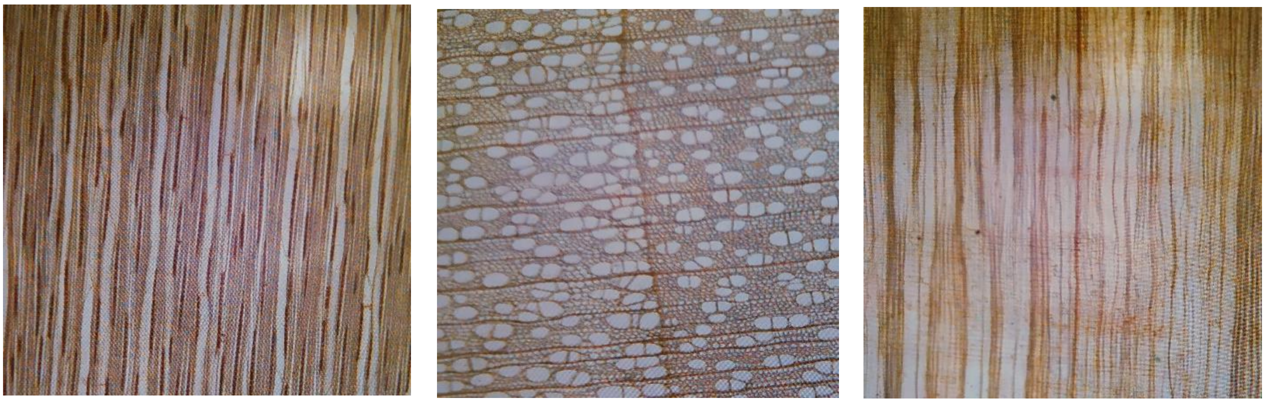

Figure 1.2 From left to right: aspen's microsopic structure in tangential, transverse and radial view [7]

The general properties of aspen wood are presented in Table 1.1 where they are compared to properties of birch wood, as this thesis will later deal with the comparison of aspen and birch plywood [7]. It can be seen that for each property, birch has a higher value, meaning aspen is softer, lighter and mechanically weaker.

Table 1.1 Comparison of properties between aspen (*Populus tremula*) and birch (*Betula pendula*) [7]

| **Property** | **Aspen** | **Birch** |
|---|---|---|
| Density, kg/m$^3$ | 490-540 | 630-670 |
| Shrinking, % | 13.5-14 | 14.2 |
| Tensile strength, MPa | 110 | 137 |
| Compression strength, MPa | 42-47 | 54-60 |
| Bending strength, MPa | 75-82 | 107-123 |
| Modulus of elasticity, MPa | 11 000 - 13 000 | 13 000 - 15 000 |
| Hardness (transverse/radial), Janka | 320-360/250-270 | 460/420 |

Historically, aspen has been used for various purposes: tool handles, skis, dishes, packaging and roof shingles as it is a very easy wood to process. Aspen has been long used for matchmaking because it has no resinous substances and it has good impregnation qualities. It has found use in the interior parts of saunas and it is also used for firewood. [4]

Other main area of use is the cellulose industry, where it is used in combination with other wood species to produce superior quality paper [4]. It has been reported that every other aspen tree that is felled in Estonia is utilized by the company Estonian Cell, that produces wood pulp [8].

## 1.2 Plywood production

Plywood is an engineered wood material that consists of odd number of veneer layers that are placed at right angles and glued to each other. The use of odd number of layers provides similar properties in the different directions of the plywood panel, due to the anisotropic quality of the wood [9]. Plywood is symmetrical in regards to its core centreline [9]. Plywood has got various applications: transport, construction, concrete formwork, furniture, interior design, packaging – to list a few. [10] [11] [12]

Good quality plywood requires great quality veneers, which in turn require great quality raw material. As important are the processing steps beginning from soaking the logs and ending with lamination (finishing) of plywood. A general scheme for the manufacturing of plywood can be seen in Figure 1.3 and a selection of these steps will be briefly explained in this chapter.

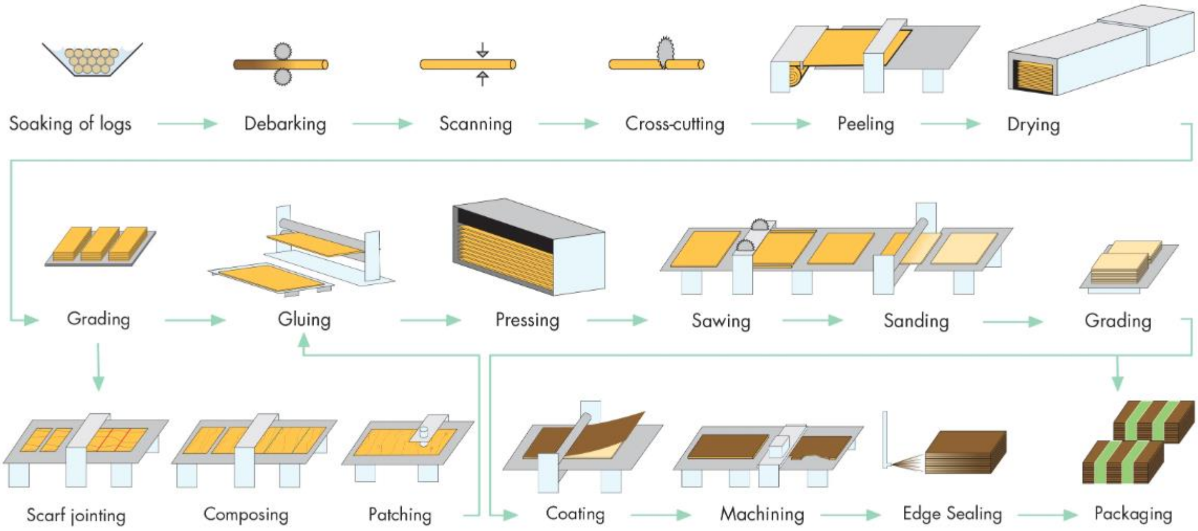

Figure 1.3 Plywood manufacturing process [13]

### *Soaking of logs*

When suitable logs have been selected, then they are usually pre-treated by heating them with steam or warm water. The purpose of the heating of the logs is to increase the softening of the wood so that it can be peeled better without creating extra mechanical damage (bigger lathe checks). Logs are usually soaked for 24 hours. [14]

### *Peeling of logs*

Peeling is a very important parameter when producing veneers. The peeling creates two sides on the surfaces of the veneer: loose side of the veneer (with lathe checks) and a tight side of the veneer (without lathe checks); see Figure 1.4. The importance of knowing the loose and tight side of the veneer will arise in the gluing and pressing part of the process. In order to minimize the lathe checks the cutting knife also has a nosebar to apply pressure when peeling. In many cases the thickness for peeling hardwoods is 1.5 mm and for softwoods 2 mm. [14] [9]

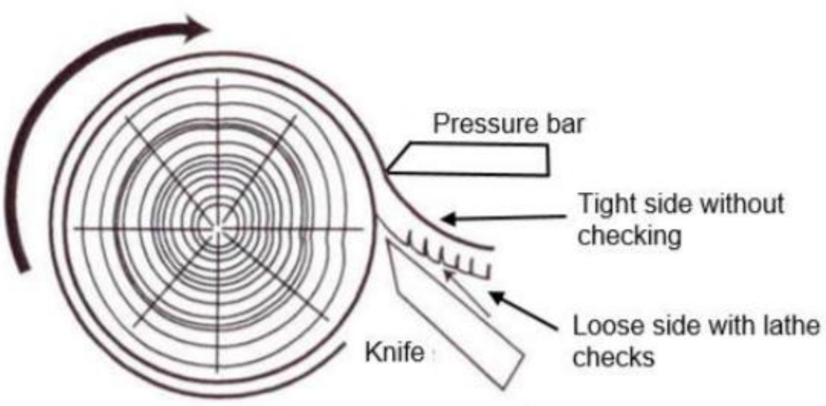

Figure 1.4 Lathe check formation in rotary peeled veneer [15]

### *Drying of the veneers*

Usually veneer is dried to 4-7 % moisture content. The moisture content can vary greatly among sheets of veneer and so the moisture content should be monitored to have control of the quality of the veneer. Overdrying of veneer causes surface inactivation and the underdrying veneer decreases adhesive penetration, in both cases leading to poor bonding. Over majority of the energy need of a plywood mill is consumed by the drying process, so it must be economically well managed. The temperature and time of the drying process are dependent on the wood species and thickness of veneer. [16] [17] [18]

***Jointing, composing and patching***

Depending on the drying method used in the mill, the veneer is already cut into a suitable length or it is done after the drying process. Then the quality of the veneer is graded and the sheets are sorted accordingly. This enables to enhance the quality of some of the veneers by patching and gives the opportunity to join smaller pieces together into bigger ones. [14] [9]

***Gluing and pressing***

The most used glue in the plywood industry is phenol formaldehyde adhesive (PF) as it produces panels suitable for exterior use (description of the adhesive in more detail in chapter 1.4 Phenol-formaldehyde resin). Other commonly used adhesives for interior applications are urea formaldehyde (UF) and melamine urea formaldehyde (MUF). The technology for adhesive coating can either be roller coaters, curtain coaters or spray systems. When placing the veneer sheets to be pressed after coating with glue, it is important to make sure that the loose side will be placed against the tight side and both outer veneer plies exhibit the tight side as a visible surface. PF adhesive needs approximately 125-135°C for curing, UF and MUF about 100-120°C. The pressure used is about 1.6-2.0 MPa, depending on the density of the wood species. At the beginning of the pressing cycle the pressure is greatest and then it slowly decreases over time, to maintain the thickness of the panel and to release the water vapor inside the panel. [14] [9]

***Finishing***

The last step in plywood manufacturing can be considered as the finishing, where the surface is sanded, panels are cut into size, checked that the edges are straight and corners are 90°. Then the plywood panels can be laminated with a suitable finishing for different intended applications. [9]

## 1.3 Wood adhesion

The topic of wood adhesion is complex and numerous articles and chapters in books have been published on the subject, delving into many sub-categories. One contributing factor to the complexity of wood adhesion is the complexity of the wood material itself. Beginning with the harvesting time and conditions, different wood species (e.g.

softwoods vs hardwoods), the difference in the microstructure of the different wood species, the effect of different machining procedures on the surface of wood, storage conditions of the wood material, drying conditions of the wood, also the anisotropic and porous quality of wood and many other factors when processing wood material affect the adhesion and adhesive strength of wood joints. In addition, another facet contributing to the complexity of wood adhesion are the possible different adhesives that are available for use and the process of their application to the wood surface.

In order to better understand adhesion theories in regards to wood, it is important to know the structure of wood. In many approaches to wood as a material, it is often described and viewed on different size levels – most commonly on macroscopic and microscopic levels. The macroscopic features can be the tree's trunk, leaves, branches, bark etc. On a microscopic level wood is examined through wood cells (tracheids, vessels, rays, resin canals, fibers etc), the wood cell wall (and its different layes: S1, S2, S3) and further, the basic chemical composition (cellulose, hemicellulose and lignin) is often described in detail. It is understandable as for instance chemical composition plays an important role when delving into the chemical adhesion theory, whereas mechanical interlocking theory draws big attention to the porous nature of wood and to the knowledge of the morphology of different wood cells and the effect of machining procedures.

## 1.3.1 Wettability and surface roughness of wood

### ***Wettability***

Wetteability can be regarded as a measurement of a solid's affinity for a liquid and it is a great predictor of bonding strength [19]. The important parameters to keep in mind are flow, penetration and transfer, as can be seen in Figure 1.5 [9]. Taking into account the aforementioned parameters, for different wood bonding application conditions (e.g. different wood species, type of application), the adhesive qualities are tailored to suit each one [9]. As an example, in the case of plywood, when compared to oriented strandboard (OSB), adhesives have higher viscosities as they are intended to sit more on the surface [9].

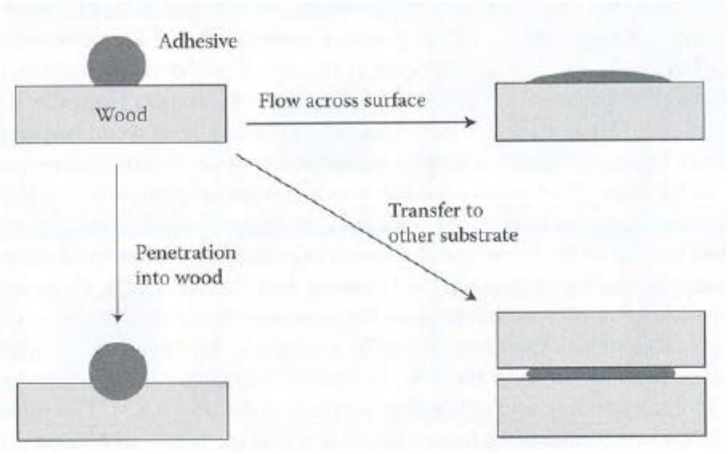

Figure 1.5 Flow, penetration and transfer of adhesive onto wood [9]

Measurement of the contact angle via sessile drop method is a known and simple technique to predict bonding strength and the wettability of a solid [20]. When applied to wood, when a contact angle is very low, it could mean that over-penetration is occurring and thus resulting in a starved joint [9]. However, when a contact angle is very high it might indicate under-penetration that leads to insufficient wood-adhesive interaction [9]. It has also been found that in correlation with surface morphology, the surface roughness will decrease the contact angle for a water droplet on a hydrophilic surface or the contact angle for a water droplet on a hydrophobic surface will increase [21]. A visual representation of a contact angle (θ) is shown in Figure 1.6.

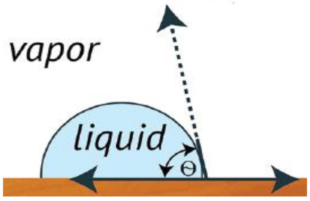

Figure 1.6 Contact angle visualisation [20]

### *Surface roughness*

Surface roughness can also impact the adhesion performance. Often the machining of wood creates a rougher surface, sometimes helping the bonding process but it can also be detrimental. A roughened surface is considered to aid in adhesion when it creates a larger surface area for contact between the adhesive and substrate, aiding in mechanical interlocking. However, in the case of wood, too ’fuzzy’ of a surface where there are too many loose fibers and the surface morphology consists of many peaks and valleys, it might decrease the wetting capabilities (by entrapping air), and lead to under penetration or in case of many valleys, to a starved joint [22].

## 1.3.2 Adhesion theories

Many theories have been proposed to explain the phenomenon of adhesion. The main ones that have been linked to wood have been: mechanical interlocking, weak boundary layer, adsorption, electronic, diffusion, acid-base theory and chemical bonding [21] [23]. In this chapter the mechanical, weak boundary layer and adsorption theories are explained briefly.

***Mechanical interlocking***

Mechanical interlocking theory is based on the notion that an adhesive will fill the irregularities and pores in a substrate (such as wood) and after solidifying, it will create an interlock. Mechanical interlocking is illustrated in Figure 1.7, where dovetailing, friction increase and increase of coating surface area can be observed [23]. In the case of hardwoods, the sufficient penetration and flow of the adhesive into wood cells, vessels and any other voids present (from machining for example) are important to create a stronger mechanical interlock [21]. Penetration of the adhesive of approximately 6–10 cell diameters, whilst displacing trapped air, has been found to be optimal for bonding [21].

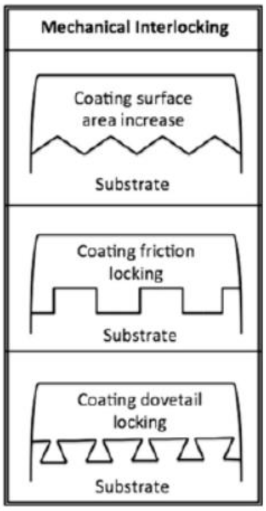

Figure 1.7 Mechanical interlocking [23]

***Weak boundary layer***

This concept deals with adhesion from the standpoint of failure of the glue joint. Weak boundary layer in wood adhesion is usually the layer between the adherend and the substrate – most likely to fail [24]. Often the surface can become contaminated by different particles (dust, loose wood particles) and those can inhibit the replacement of air with adhesive and in general causing poor wetting, leading to a poor adhesive joint [24]. The wood surface can also be covered in crushed cells, that form during machining. The crushed cells can form because (especially earlywood) cells are not strong in tangential and radial direction [9]. Those crushed cells decrease the adhesive's penetration capability into the wood surface [9]. The weak boundary layer can also be caused by the extractives in the wood [23].

***Adsorption***

The explanation for the adsorption theory entails the intermolecular and interatomic forces between a substrate and an adherend [24] [25] [21]. These forces can be any type: London interactions, covalent bonds, van der Waals forces, electrostatic or hydrogen bonds [25] [21]. Important to this theory is the intimate contact between the adherend and substrate, termed wetting [24]. Good wetting can be evaluated by contact angle measurements [24].

## 1.4 Phenol-formaldehyde resin

Phenolic resins are the product of polycondensation of phenols and aldehydes [26].The polycondensation of formaldehyde is the basis for the biggest group of adhesives produced today (over 50% by volume) [27]. These include urea-formaldehyde (UF), phenol-formaldehyde (PF), melamine-formaldehyde (MF), melamine-ureaformaldehyde (MUF), resorcinol-formaldehyde, and others [27].

Phenolic resins are classified in adhesive technology as reactive adhesives which form when prepolymer thermosets are cross-linked [28]. Phenolic resins are widely used in the wood industry and represent one of the largest volumes of any synthetic adhesive [29]. The reason for their widespread use is their dimensional stability, water resistance (important for exterior use), heat resistance and low cost [25].

PF – phenol-formaldehyde resins are divided into two groups: resole resins and novolak resins [30]. The main differences between these PF resins are: resole resins are formed under alkaline conditions and with a molar ratio of phenol to formaldehyde with an excess of formaldehyde (F/P ratio greater than 1); novolak resins are formed under acid conditions with an excess of phenol (F/P ratio less than 1) [31]. Resoles are referred to as single-step resins and novolaks as two-step resins [32]. This is due to the fact that resoles cure by application of heat, novolaks by adding a curing agent [32]. For application in the wood industry, resole resins are used.

### 1.4.1 Resole-type PF resin

Resole-type resins are formed by the polycondensation of phenol ($C_6H_5OH$) and formaldehyde ($CH_2O$) under basic conditions with an excess of formaldehyde and they are thermosetting resins. [28] Polycondensations are stepwise reactions between bifunctional or polyfunctional components with liberation of small molecules, such as water [28]. Polycondensation in PF resins occurs in the aromatic ring with an electrophilic substitution. As a result, there will be hydroxymethyl derivates and an in-between step until the methylene bridges form between the phenolic rings. [28]

Figure 1.8 demonstrates an idealized version of the reaction between phenol and formaldehyde, reactive *ortho* and *para* sites are as dotted lines [33].

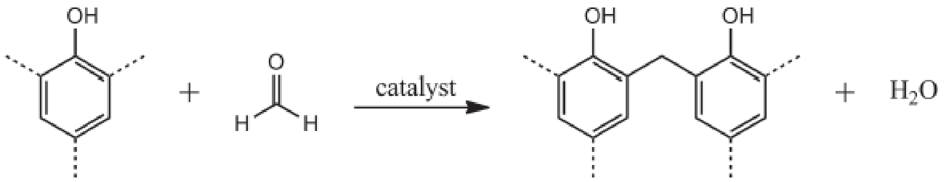

Figure 1.8 Phenol + formaldehyde reaction [33]

The exact reaction mechanism between phenol and formaldehyde is not yet completely known. It is known, however, that the reaction is initiated by the activation of the benzene ring by the hydroxyl group. Also, there are three stages recognized in the phenol- formaldehyde reaction: A-stage, B-stage and C-stage. [27]

A-stage, called resole – it is a soluble phenol-formaldehyde resin, it is a thermoplastic and initial condensation products are mainly alcohols. B-stage, also called resitol – has a higher degree of condensation and some cross-linking, but it is still soluble and not fully cured. C-stage, also called resite – fully cross-linked thermoset resin. [27] The structure of a resol can be seen in Figure 1.9 [32].

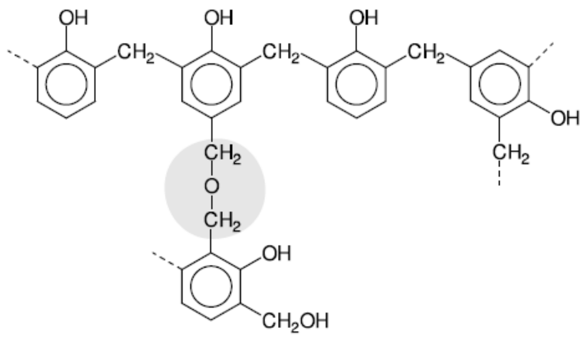

Figure 1.9 Structure of a resole [32]

For adhesive applications, polymerization is stopped short of the gel point by cooling (B-staging) [27]. The product at this point is an intermediate resole phenol-formaldehyde resin [27]. Resins supplied as adhesives are invariably B-stage prepolymers [30].

As resole resins will be adhesives for plywood, they are in principle solutions of low molecular weight condensation products of phenol and formaldehyde in aqueous sodium hydroxide. Low molecular PF resins have been shown to penetrate into the wood cell wall [34]. They provide bond strengths that might generally be greater than that of the wood substrate. Durability is good, and the bond is unaffected by boiling water, mold, or fungus [24]. Resole resins are typically sold in liquid form and contain low molecular weight hydroxymethyl phenol species [33]. Resole phenolic resins have a short shelf life because of their reactivity: less than 1 year (less than 60 days in most cases) [27] [30].

### 1.4.2 Lignin-phenol-formaldehyde resin

As environmental concerns have come into focus more as ever, the attention is turned towards ecological, natural, renewable, environmentally friendly and safe materials in all fields of life. This is also present in the adhesive industry and one example of such trend is the continuing research and development of lignin based adhesives for use in the wood industry.

The appeal of lignin is its low cost (lowering the final cost of product), abundace of the raw material (considering plant material and also waste material from wood pulping) and structural similarity to phenol, also lower formaldehyde emission when in use [35] [36]. However, lignin has significantly lower reactivity towards formaldehyde than phenol, due to having only few phenolic rings and no polyhydroxy phenyl rings, this in turn creating poorer (lower reactivity) systems [24] [36]. In order to successfully use lignin in PF resins for example, many pre-treatments of lignin have been found to increase the reactivity and thus leading to better resin formulations. Some of the pre-treatments are: demethylation, oxidation, methylolation, phenolation, hydrolysis and reduction [37].

Lignin based adhesives have been extensively researched and shown in studies to be successful in creating interior grade plywood without the use of synthetic adhesives as reinforcement, however the industrial application has not been wide at all [21] [38]. One aspect of purely lignin adhesives is that due to the lower reactivity of the raw material, it requires longer pressing times and has been found to be corrosive to equipment, which is a hinderance in the panel industry [21] [35] . Commercial use has been in the field of substitution of some amount (20% to 30%) of pre-treated (methylolated) lignin with phenol in the formulation of phenol-formaldehyde resins (used in some North American plywood mills) [21] [35].

# 2. MATERIALS AND METHODS

## 2.1 Materials

### 2.1.1 Veneer

For this research aspen veneers with varying log soaking temperatures were used and for comparison birch plywood was made in addition to aspen plywood. The chosen veneers were without any knots or visible defects, as 3-ply plywood panels would be made out of them.

The different log soaking temperatures for aspen were: no pre-soaking, 20°C, 40°C and 70°C. The birch veneer had a log soaking temperature of 40°C, which is the most common temperature used in the industry. The log numbers that the veneers were taken from are as follows: no pre-soaking – no marking; , 20°C – log nr 3.2, 40°C - log nr 3.1 and 70°C – log nr 4.1. Birch veneers came from log nr B2. All logs were soaked for approximately 24 h before peeling.

According to the the Laboratory of Wood Technology documentation, the logs were felled in the winter of 2019/2020 from Keila and Piirsalu forest units in Läänemaa county, Estonia. The length of the logs was 2.4 meters and the average age was 60 years. Prior to peeling, the logs were stored in the Laboratory of Wood Technology logyard.

The drying parameters for the aspen veneers were at 170°C and 3 min. The moisture content of the aspen veneers were as follows: no pre-soaking – 4.2%; 20°C – 5.9%; 40°C – 4.2%; 70°C – 4.5%. The drying parameters for birch were 180°C and 2 min; moisture content for the birch veneer was 7.4%.

Both aspen and birch veneers were peeled with a thickness of 1.5 mm. The veneers, that had inital measurements of 990x420 mm were cut into 420x420 mm pieces to be made into plywood panels. Leftover pieces from cutting were used to determine the contact angle, surface roughness and moisture content. The samples cut from the leftover pieces were approximately 120x40 mm.

Following Table 2.1 showcases all the parameters for the veneers that were initially determined, including the moisture content measurement.

Table 2.1 Veneer parameters

| **Species and log soaking temperature** | **Log number** | **Veneer drying conditions** | **Storage conditions** | **Moisture content, %** | **Thickness, mm** |
|---|---|---|---|---|---|
| Aspen, no pre-soaking | *not known* | 170°C ; 3 min | 25°C ; RH 33% | 4.2 | 1.5 |
| Aspen, 20°C | 3.2 | | | 5.9 | |
| Aspen, 40°C | 3.1 | | | 4.2 | |
| Aspen, 70°C | 4.1 | | | 4.5 | |
| Birch, 40°C | B2 | 180°C ; 2 min | 28°C ; RH 25% | 7.4 | |

### 2.1.2 Adhesives

Two different phenolic adhesives were used:

1. Prefere 24J662 resin + Prefere 14J021 hardener

2. Prefere EXPH 051 resin + Prefere EXPH 9500 hardener

The abbreviations for the resins and their respective hardeners and adhesives are marked in this work as follows: LPF as the lignin phenol formaldehyde adhesive and PF as the regular phenol formaldehyde adhesive.

The Prefere hardeners 14J021 and EXPH 9500 are quite similar, just the quantities of the fillers are in a different order. Information from the MSDS (*material safety data sheet)* about the hardeners is compiled into Table 2.2. The information described in the MSDS shows only the substances that have a workplace exposure limit or could be hazardous to health or the environment. Thus, any other ingredients are not required to be listed in the MSDS. [39] [40]

Table 2.2 Hardener comparison between Prefere 14J662 and Prefere EXPH 9500

| **Ingredient name** | **Prefere 24J662 (PF), %** | **Prefere EXPH 9500 (LPF), %** |
|---|---|---|
| Limestone | ≥25 - <50 | ≥25 - <50 |
| Cellulose | ≥10 - <25 | ≥5 - <10 |
| Sodium carbonate | ≥5 - <10 | ≥5 - <10 |
| Starch | ≥5 - <10 | ≥25 - <50 |

The resins, however, are different. Prefere EXPH 051 is a resin with reduced phenol content that is substituted with lignin. Prefere 24J662 is a traditional phenol-formaldehyde resin. Both are formulated to be used in the woodworking industry, mainly as plywood adhesive, meant for exterior use.  The Prefere EXPH 051 is marketed more as an environmentally friendly resin due to it's reduced phenol content. Table 2.3 shows the differences between the resins according to the ingredients and properties listed in their MSDS. As mentioned before, any other ingredients that do not have any workplace exposure limit or are not dangerous to the environment or health, are not required to be listed. [41] [42]

Table 2.3 Resin comparison between Prefere 14J021 and Prefere EPXH 051

| **Ingredient name** | **Prefere 14J021 (PF), %** | **Prefere EXPH 051 (LPF), %** |
|---|---|---|
| Phenol, polymer with formaldehyde | ≥25 - <50 | ≥10 - <25 |
| Sodium hydroxide | ≥5 - <10 | ≥5 - <10 |
| Methanol | ≥0.1 - <0.3 | ≥0.1 - <0.3 |
| **Physical and chemical properties** | **Prefere 14J021 (PF)** | **Prefere EXPH 051 (LPF)** |
| Colour | Brownish-red | Brown (dark) |
| Odour | Phenolic (slight) | Phenolic (slight) |
| pH | 11.6 - 12.2 | 13 |
| Density (at 20°C) | 1.2 - 1.22 g/cm$^3$ | 1.215 g/cm$^3$ |
| Dynamic viscosity (at 20°C) | 250 - 400 mPa·s | 350 mPa·s |

## 2.2 Methods

This section will explain the tests carried out with the veneers and plywood. In Table 2.4 the overall testing plan for veneers is presented, where all the number of test specimens and the number of measurements are shown.

Table 2.4. Testing plan for veneers

| **Species and log soaking temperature** | **Type of test** | | | | | |
|---|---|---|---|---|---|---|
| | **Moisture content** | | **Surface roughness** | | **Contact angle** | |
| | No of test pieces | No of measurements | No of test pieces | No of measurements | No of test pieces | No of measurements |
| Aspen, no pre-soaking | 10 | 10 | 20 | 20x2=40 | 20 | 20x2=40 |
| Aspen, 20°C | 10 | 10 | 20 | 20x2=40 | 20 | 20x2=40 |
| Aspen, 40°C | 10 | 10 | 20 | 20x2=40 | 20 | 20x2=40 |
| Aspen, 70°C | 10 | 10 | 20 | 20x2=40 | 20 | 20x2=40 |
| Birch, 40°C | 10 | 10 | 20 | 20x2=40 | 20 | 20x2=40 |
| **Total:** | 50 | 50 | 100 | 200 | 100 | 200 |

In Table 2.5 the overall testing plan for plywood is presented. It was taken into account that half of the tests would be carried out with the regular PF resin, and half of the tests would be carried out with the LPF resin.

Table 2.5 Plywood testing plan

| **Type of veneer and soaking temperature** | **No of panels** | **Type of test** | | |
|---|---|---|---|---|
| | | **Glue consumption during manufacturing** | **Panel density** | **Bond shear strength** |
| | | No of test pieces and measurements | No of test pieces and measurements | No of test pieces and measurements |
| Aspen, no pre-soaking | 2 PF + 2 LPF = 4 | 1x4=4 | 4x6=24 | 4x12=48 |
| Aspen, 20°C | 2 PF + 2 LPF = 4 | 1x4=4 | 4x6=24 | 4x12=48 |
| Aspen, 40°C | 2 PF + 2 LPF = 4 | 1x4=4 | 4x6=24 | 4x12=48 |
| Aspen, 70°C | 2 PF + 2 LPF = 4 | 1x4=4 | 4x6=24 | 4x12=48 |
| Birch, 40°C | 2 PF + 2 LPF = 4 | 1x4=4 | 4x6=24 | 4x12=48 |
| **Total:** | 20 | 20 | 120 | 240 |

## 2.2.1 Veneer testing

***Moisture content***

Moisture content of the veneers was measured using small veneer pieces (weight up to 1 g) taken from the bigger sheet and weighed, then placed in a ventilated oven at

103°C ± 2°C for about 2 h and then weighed again. The moisture content was calculated with the following formula [7]:

$$Moisture\ \% = \frac{m-m_0}{m_0} \cdot 100 \qquad (2.1)$$

Where m – weight before drying

$m_0$ – weight after drying

From each log soaking temperature group of veneers, 10 specimen were taken for moisture content measurements (altogether 10x5=50 specimen), and then the arithmetic mean was calculated to get the average for each log soaking temperature.

***Surface roughness***

Equipment used for the surface roughness measurement was Mitutoyo Surftest SJ-210 (see Figure 2.1), which performed roughness measurements via stylus method conforming to ISO 4288:1997 standard (profile:R, parameter:3, filter: Gauss, λc: 2.5, λs: 8). The samples were placed onto a veneer panel that had 1.5 mm cutout space (equal to the thickness of the veneer) and fixed from the sides with clamps to provide as level of a surface as possible in order to be able to carry out the surface roughness measurements. Distance of one measurement was 12.5 mm.

Surface roughness measurements were taken from smaller veneer specimen of approximate sizes 120x40 mm. The measurements were taken across the sample (across the grain) on the tight side of the veneer (the side without lathe checks).

For each log soaking temperature, 20 specimen were picked and from each specimen 2 measurements of surface roughness were intended to be taken, so total 20x5x2 = 200 measurements.

Figure 2.1 Mitutoyo Surftest SJ-210 surface roughness measuring device [43]

Each surface roughness measurement gave an output of three parameters: Ra, Rq and Rz. Rz indicates the average maximum height of profile (amplitude parameter), Rq is the root mean square value of the ordinate values (*ordinate value – the height of the assessed profile at any position*) and Ra is the arithmetic mean of absolute ordinate values within a sampling length [44]. In this research the main focus was on describing the parameters Rz and Ra.

### ***Contact angle measurement***

The contact angle measurement was done with DataPhysics OCA 15 EC Contact Angle measuring device, using computer software SCA20. The measuring technique is shown in Figure 2.2.

For each specimen 2 contact angle measurements were taken by dropping distilled water onto the wood surface (altogether 200 measurements). The contact angle was observed on the tight side of the veneer (without lathe checks) noting the decrease of the contact angle in the direction of the grain. The veneer was clamped down at the sides to provide as smooth as a surface as possible.

The recording of the contact angle was started on the software as soon as the water drop came into contact with the wood surface and the observation time was 40 s. Of the values obtained, the change of the contact angle for later analysis was noted starting from 1 s and then at 5 s, 10 s, 15 s, 20 s, 25 s, 30 s, 35 s and 40 s.

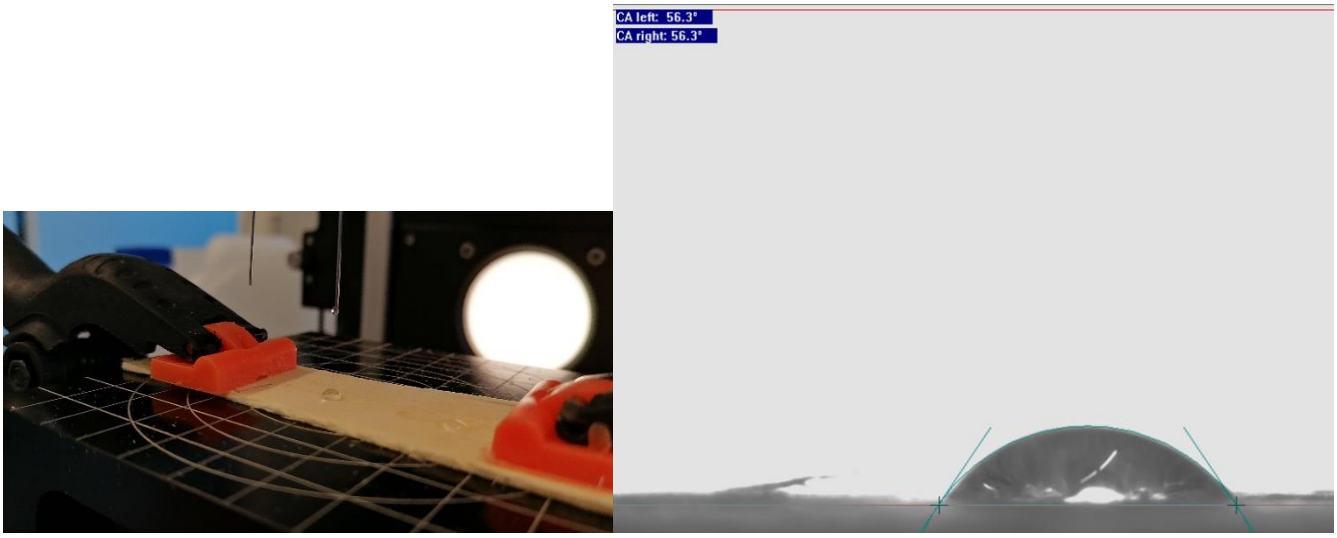

Figure 2.2 Measuring the contact angle. Left: specimen fixed with clamps. Right: software measuring the contact angle.

### 2.2.2 Making of plywood

For this research 3-layer plywood panels were prepared. The dimensions of the plywood panel were approximately 420x420 mm. It was set that for each log soaking temperature two panels would be made. In total there were 5(log soaking temperatures) x 2(adhesives) x 2(panels per log soaking temperature) = 20 panels.

Plywood panels were made at the Taltech Laboratory of Wood Technology using roller coater technology for adhesive application (the adhesive was mixed approximately for 1 hour before application) and INFOR hotpress for hot pressing and cold pressing. Plywood was cold-pressed for 10 min at 1.2 MPa and then hot pressed at 130°C for 5 min at 1.6 MPa.

#### *Cutting plan*

The cutting plan for the specimens (bond shear strength and thickness) was mapped out according to the relevant standard EVS-EN 326-1:2002 by modifying it a little, and the cutting plan can be seen in Figure 2.3 [45]. The bond shear strength specimens were cut as 6 specimens from the edge and 6 specimens from the ’center’, however due to the size of the panel the ’center’ was regarded as just not from the edge. For each group, 3 specimens had the neccessary grooves cut from the bottom and for 3 from the top of the panel. The bond shear strength specimen dimensions were 120x25 mm. The number of thickness specimen for each panel was 6 and their dimensions were 50x50 mm and at least one specimen was cut from the edge.

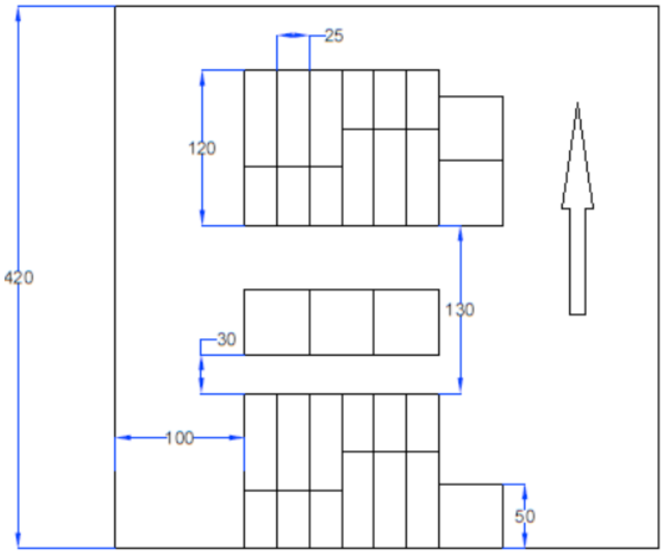

Figure 2.3 Plywood specimen cutting plan, grain directon showed on face veneer with an arrow

### 2.2.3 Plywood testing

***Glue consumption***

Glue consumption was calculated for each plywood panel. Every panel had only one sheet that was coated by adhesive (due to the roller coater technology and the fact that it was 3-ply plywood) so 20 measurements were taken. The veneer sheet was weighed before and after coating with adhesive and so knowing the surface area of the veneer sheet, the glue consumption was calculated to g/m$^2$.

***Panel thickness***

Panel thickness was measured along 8 points that were positioned equally 50 mm from the edge according to EVS-EN 324-1:2002. The average was taken for each panel out of the 8 measurements. [46]

***Panel density***

Panel density determination was done according to EVS-EN 323:2002 [47]. 50x50 mm specimens were cut out, from each panel 6 specimens, so altogether 120 specimens.

The specimens were conditioned in a climate chamber at 65% relative humidity and at 20°C. When the specimens were ready according to EVS-EN 323:2002, they were weighed and measured (side length and thickness, see Figure 2.4) and the density was calculated. [47]

Figure 2.4 Plywood thickness measuring on density specimen

***Bond shear strength***

Bond shear strength was tested according to EVS-EN 314-1 and EVS-EN 314-2. The pre-treatment was chosen as class 3, which is meant for plywood that is used in exterior conditions (typical for phenolic adhesive use). The pre-treatment sequence was as follows [48] [49]:

1. soaking 24 h at (20 ± 3) °C ;
2. soaking (72 ± 1) h in boiling water;
3. cooling in (20 ± 3) °C for at least 1 h.

After the pre-treatment the specimens were tested with the Instron tensile testing machine, obtaining the maximum force to pull apart the specimen, and the cohesive wood failure percentage was noted down. Shear strength was calculated as the maximum force recorded during testing divided by shear area (dimensions taken before pre-treatment) in N/mm$^2$.

# 3. RESULTS AND DISCUSSION

## 3.1 Surface roughness

Average values for surface roughness are shown in Table 3.1 and in Figure 3.1, lowest values for aspen are highlighted in light blue, highest values in yellow, standard deviation is shown in brackets.

It was very difficult to obtain the measurements from aspen veneers, as often measurement points were over the range for the Mitutoyo Surftest SJ-210 device. This could have been caused by the loose fibers on the surface of aspen that disrupted the measurement process. In some aspen groups the taken measurements were 1 or 2 less than intended. This also means that the results for aspen are the lowest roughness readings and possibly the actual surface roughness is a little bit higher.

Table 3.1 Average Ra and Rz values

| **Parameter** | **Log soaking temperature** | | | | |
|---|---|---|---|---|---|
| | **Aspen no pre-soaking** | **Aspen 20°C** | **Aspen 40°C** | **Aspen 70°C** | **Birch 40°C** |
| Ra, µmm | 24.77<br>(5.07) | 24.12<br>(5.71) | 22.91<br>(6.48) | 29.51<br>(6.40) | 13.38<br>(3.00) |
| Rz, µmm | 152.68<br>(24.18) | 147.99<br>(35.74) | 146.84<br>(31.31) | 179.24<br>(31.73) | 98.12<br>(12.71) |

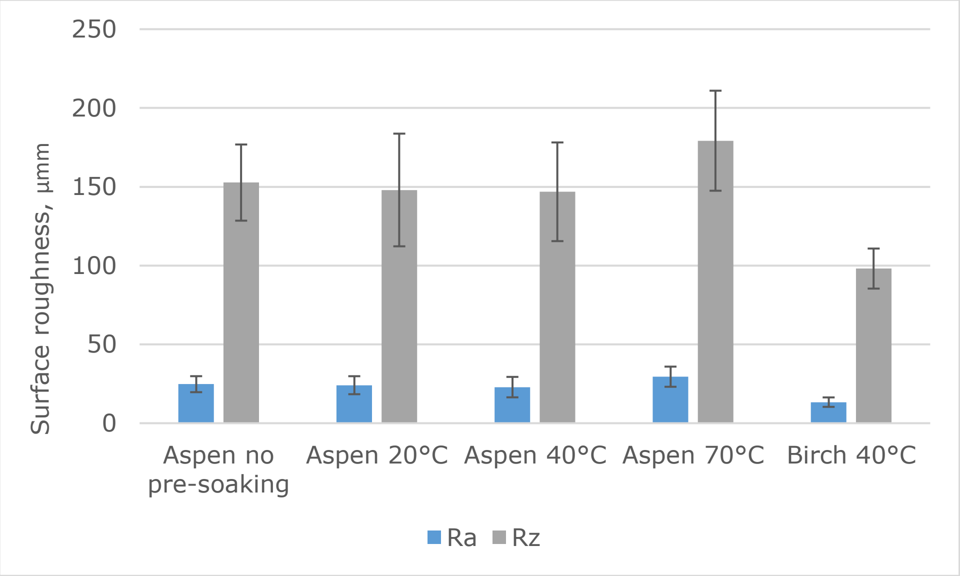

Figure 3.1 Averages of surface roughness parameters

According to single factor ANOVA analysis performed in MS Excel (α=0.05), there is a significant statistical difference between some of the averages of groups (i.e. between the Ra means) of the different log soaking temperature groups.

After applying the Tukey-Kramer test as post hoc, it was noted that the significant statistical differences were found between the comparison of aspen 70°C group (Ra=29.511μmm, Rz=146.840 μmm) with all other groups, and quite understandably with birch 40°C group (Ra= 13.380 μmm, Rz =98.116 μmm) with all other groups. That means for aspen veneers: no pre-soaking, soaking at 20°C and soaking at 40°C gave statistically equal averages, meaning no real effect of log seaking temperature on surface roughness was observed.

However, when looking at the data purely mathematically, it must be noted that lowest average surface roughness parameters by log soaking temperature were found on birch 40°C followed by aspen 40°C (best from aspen groups), aspen 20°C, aspen no pre-soaking and the roughest parameters were found on aspen 70°C. The following Table 3.2 and Table 3.3 show the most important statistical parameters for the data collected.

Table 3.2 Ra values

| **Statistic** | **Aspen no pre-soaking** | **Aspen 20°C** | **Aspen 40°C** | **Aspen 70°C** | **Birch 40°C** |
|---|---|---|---|---|---|
| No of data points | 40 | 39 | 39 | 38 | 40 |
| Mean | 24.767 | 24.123 | 22.908 | 29.511 | 13.380 |
| Standard deviation | 5.072 | 5.710 | 6.482 | 6.396 | 3.001 |
| Minimum value | 15.969 | 14.097 | 12.880 | 16.404 | 9.198 |
| 1st quartile | 21.682 | 20.285 | 18.973 | 25.822 | 11.294 |
| Median | 23.807 | 24.927 | 21.289 | 29.344 | 12.700 |
| 3rd quartile | 27.821 | 27.805 | 27.530 | 33.218 | 15.012 |
| Maximum value | 34.211 | 36.573 | 37.296 | 41.808 | 21.907 |

Table 3.3 Rz values

| **Statistic** | **Aspen no pre-soaking** | **Aspen 20°C** | **Aspen 40°C** | **Aspen 70°C** | **Birch 40°C** |
|---|---|---|---|---|---|
| No of data points | 40 | 39 | 39 | 38 | 40 |
| Mean | 152.677 | 147.994 | 146.840 | 179.240 | 98.116 |
| Standard deviation | 24.177 | 35.741 | 31.308 | 31.733 | 12.706 |
| Minimum value | 103.840 | 89.355 | 99.609 | 110.200 | 78.569 |
| 1st quartile | 133.585 | 125.620 | 124.125 | 154.443 | 88.980 |
| Median | 151.205 | 154.310 | 141.230 | 183.290 | 95.759 |
| 3rd quartile | 171.238 | 170.700 | 168.520 | 201.643 | 106.040 |
| Maximum value | 199.180 | 213.360 | 219.460 | 234.600 | 129.560 |

Figure 3.2 and Figure 3.3 show box and whisker chart for Ra and Rz parameter to illustrate the distribution of data and the most important markings for statistical analysis of data (all of the data points are presented in Table 3.2 and Table 3.3). As can be seen from the charts, birch 40°C shows the lowest values, whereas aspen 70°C shows the highest values for surface roughness. It can be seen from the chart that there is a big difference between the birch and aspen veneer surface roughness and that they are not close in value. Also, birch shows far less variance in the data than all of the aspen veneers. For all the groups of data there is a slight skewness.

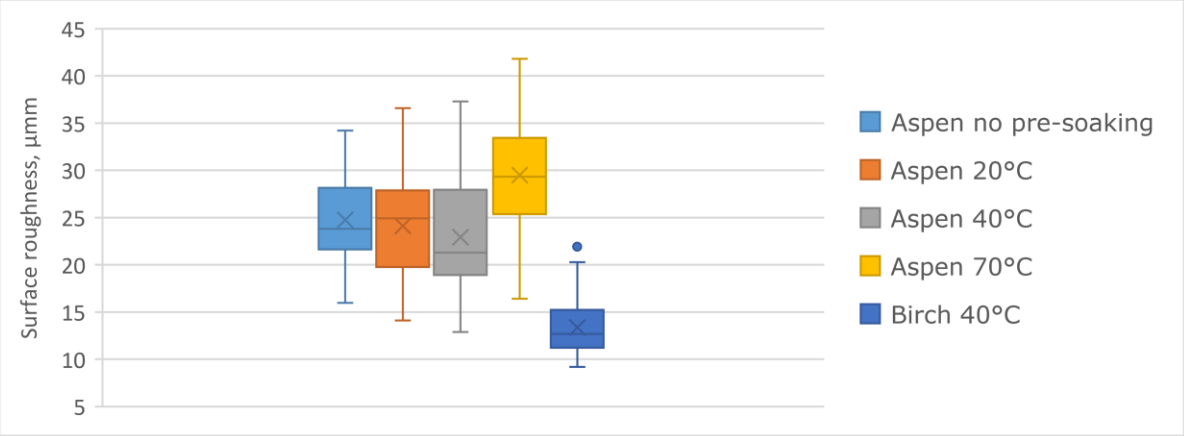

Figure 3.2 Box and whisker chart for Ra parameter

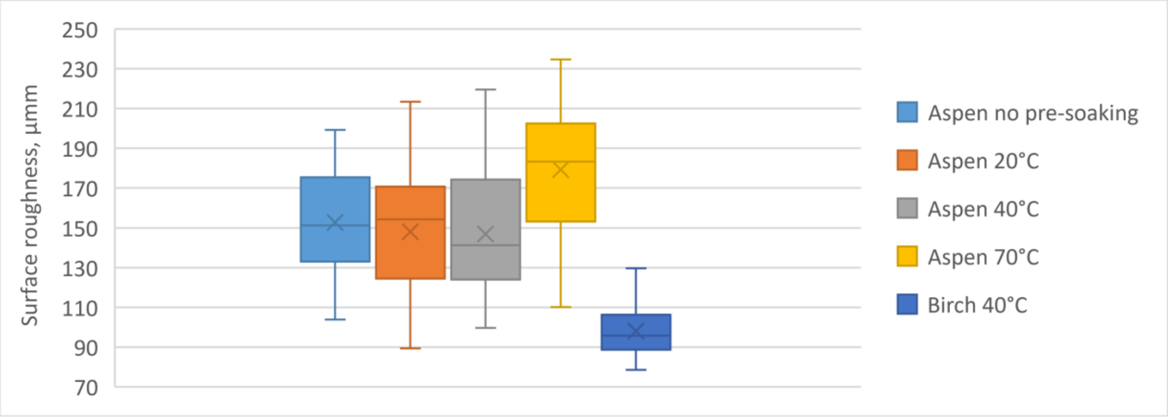

Figure 3.3 Box and whisker chart for Rz parameter

Figure 3.4, Figure 3.5 and Figure 3.6 demonstrate the typical surface roughness profile obtained with Mitutoyo Surftest SJ-210 to compare aspen 40°C (best result from aspen),

aspen 70°C (worst result from aspen) veneer surface profile with birch 40°C (control group) veneer surface profile.

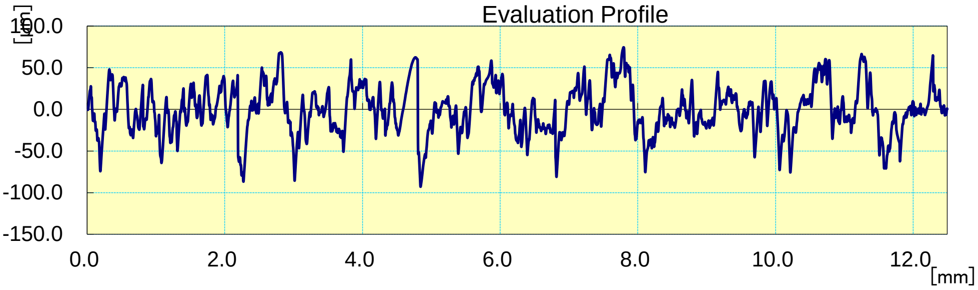

Figure 3.4 Average 40°C aspen veneer surface profile (sample's Ra=22.89 μmm, Rz=145.55 μmm)

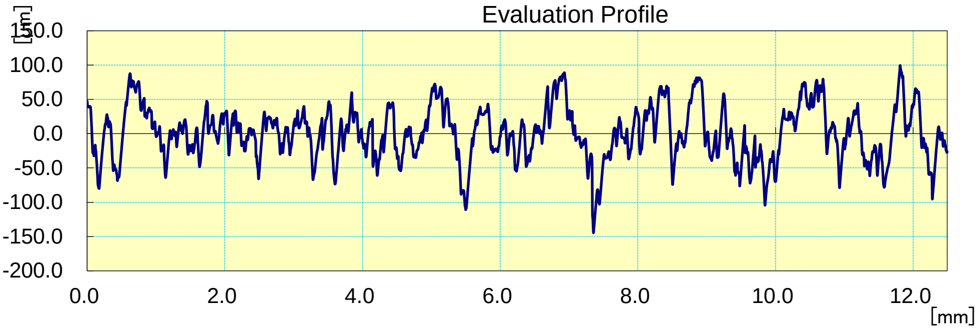

Figure 3.5 Average 70°C aspen veneer surface profile (sample's Ra=30.72 μmm, Rz=183.09 μmm)

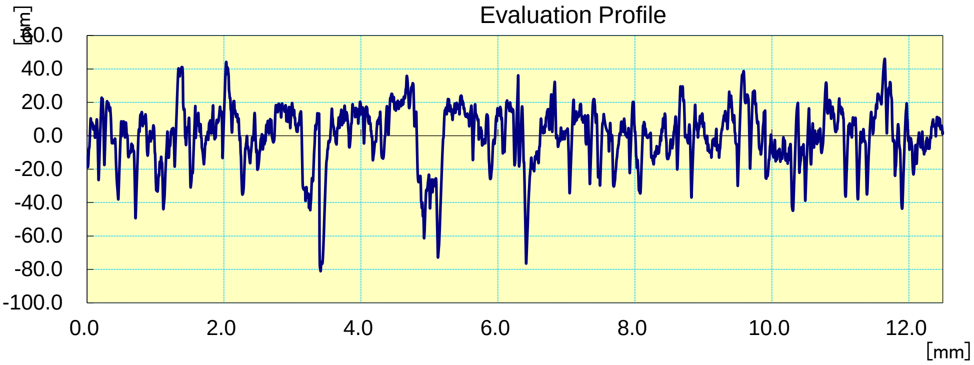

Figure 3.6 Average 40°C birch veneer surface profile (sample's Ra=13.61 μmm, Rz=97.96 μmm)

So in this study log soaking temperature affects the surface roughness of aspen when it is at an elevated temperature - 70°C – causing an increase in roughness. Studies have shown contradictory results on how higher log soaking temperature affects the surface roughness – some have found it to increase the surface roughness and some to decrease, however this could be due to the different wood species observed - birch (surface roughness increased) and spruce (surface roughness decreased) [50] [51].

The aim of soaking logs at higher temperatures prior to peeling is to plastify the wood and to decrease the severity of lathe checks, which in turn leads to better bonding qualities [14]. Perhaps when soaking aspen at a higher temperature, as it is already quite a soft wood, it causes the wood surface to have a higher roughness when peeling as the cutting knife is not sharp enough to deal with such a soft material. Another explanation might be that the higher temperature pre-treatment causes chemical changes in the wood, degrading the cell structure, leading to a roughened surface [51].

Research on aspen plywood has shown that the agglomeration of loose fibers on the surface of aspen veneer could be minimised by peeling just above the freezing point [52]. However this in turn causes deeper lathe checks which are detrimental to bonding strength, so a compromise is made – aspen can be peeled at ambient temperatures [52]. In this study, peeling of aspen with no pre-soaking yielded in a satisfying result on surface roughness – statistically it didn't matter if the aspen was peeled with no log pre-soaking, soaking at 20°C or soaking at 40°C.

In principle, often the rougher the surface the better the adhesion, as surface roughness creates more opportunities for mechanical interlocking [29]. However, there might be an optimum surface roughness for plywood manufacturing. When the surface roughness of the veneer is too high, it might hinder forming a good adhesive joint as it prevents good wetting, essentially causing a weak boundary layer. This is due to the extra fibers present on the surface restricting the replacement of air with the adhesive. Also, for example, when the lathe checks are very deep it causes over penetration of the adhesive, leading to a starved joint. In addition, when the surface is rougher, the glue consumption is higher as well because the surface area that needs to be covered with the adhesive is bigger – meaning it has an economical effect on the production of plywood as well.

## 3.2 Contact angle

Averages of contact angles by time, highest values for aspen highlighted in light blue, lowest values in light yellow, are shown in Table 3.4 and graphically in Figure 3.8.

Table 3.4 Averages of contact angle by time

| **Time, s** | **Log soaking temperature** | | | | |
|---|---|---|---|---|---|
| | **Aspen no pre-soaking** | **Aspen 20°C** | **Aspen 40°C** | **Aspen 70°C** | **Birch 40°C** |
| 1 | 79.77 | 76.19 | 82.71 | 84.38 | 81.99 |
| 5 | 68.24 | 53.09 | 69.23 | 67.62 | 68.65 |
| 10 | 57.42 | 38.94 | 60.93 | 55.70 | 61.50 |
| 15 | 48.14 | 31.92 | 54.09 | 47.17 | 55.23 |
| 20 | 42.72 | 27.43 | 49.38 | 40.36 | 51.04 |
| 25 | 37.80 | 24.43 | 46.13 | 35.14 | 47.52 |
| 30 | 33.99 | 22.52 | 43.48 | 31.46 | 44.67 |
| 35 | 30.98 | 21.31 | 40.80 | 27.85 | 42.39 |
| 40 | 28.96 | 20.32 | 38.00 | 25.35 | 40.79 |

It can be seen that by average contact angles, the highest results for aspen were obtained from the log soaking temperature of 40°C (CA=38.00), which is quite comparable to birch veneer (CA=40.79). In Figure 3.8 it can be seen that for birch 40°C and aspen 40°C the contact angle averages nearly overlap.

Using the one factor ANOVA analysis ($\alpha=0.05$) and Tukey-Kramer as post hoc, it was concluded that between birch 40°C and aspen 40°C group averages there is no significant statistical difference. The only significant statistical differences were found between birch 40°C and every other aspen group (except for aspen 40°C) and between aspen 20°C and 40°C pairing. All other pairing options resulted in the decision of no significant statistical difference between the results of the aspen veneers.

Box and whisker chart is shown in Figure 3.7 to demostrate the collection of data and Table 3.5 shows all the values necessary for the chart. Data is shown for contact angle measurement at 40 s. It can be seen that aspen 20°C has got quite a few outliers and except for birch 40°C, the data is skewed. Whereas the ANOVA analysis showed that aspen 40°C and birch 40°C groups there is no statistical significance between the means, when looking at the chart it can be seen that aspen 40°C has got a greater spread of data, birch 40°C having lower variation and the minimum contact angles found for birch are of higher value than the minimum contact angles fund for aspen 40°C.

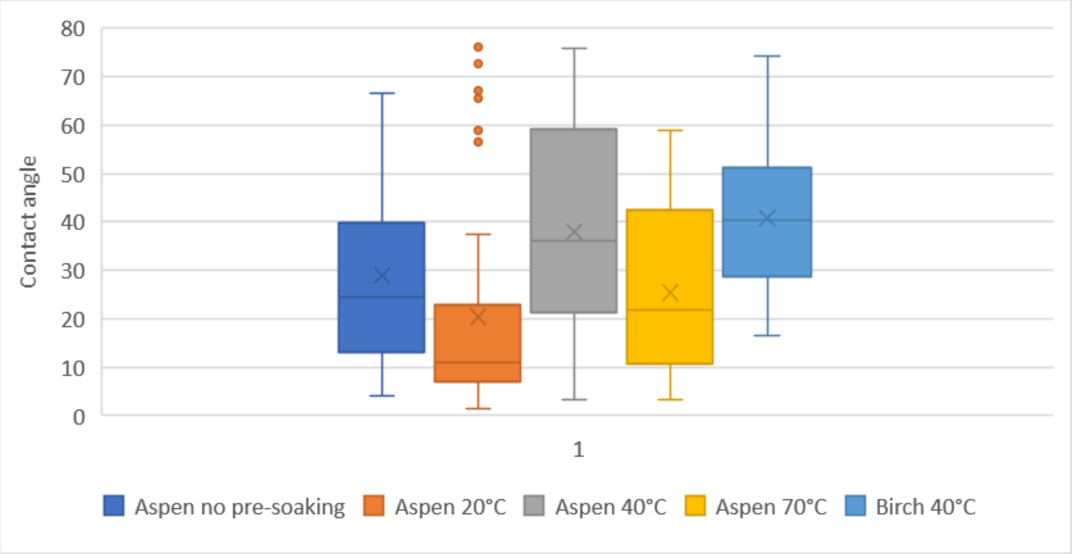

Figure 3.7 Box and whisker chart for data collected of contact angles at 40 s

Table 3.5 Data values for contact angle at 40 s

| Statistic | Aspen no pre-soaking | Aspen 20°C | Aspen 40°C | Aspen 70°C | Birch 40°C |
|---|---|---|---|---|---|
| No of data points | 40 | 38 | 40 | 39 | 40 |
| Mean | 28.96 | 20.32 | 38.00 | 25.35 | 40.79 |
| Standard deviation | 19.23 | 21.89 | 21.02 | 16.53 | 13.32 |
| Minimum value | 4.10 | 1.33 | 3.26 | 3.20 | 16.59 |
| 1st quartile | 13.71 | 7.15 | 21.77 | 10.88 | 30.40 |
| Median | 24.59 | 10.93 | 36.14 | 21.81 | 40.32 |
| 3rd quartile | 34.99 | 18.16 | 58.80 | 38.64 | 51.07 |
| Maximum value | 66.53 | 76.07 | 75.92 | 58.84 | 74.22 |

When looking at the contact angle averages over time in Figure 3.8 it can be seen that aspen 40°C and birch 40°C almost overlap, whilst aspen 20°C is a clear outlier, showing lowest average contact angles.

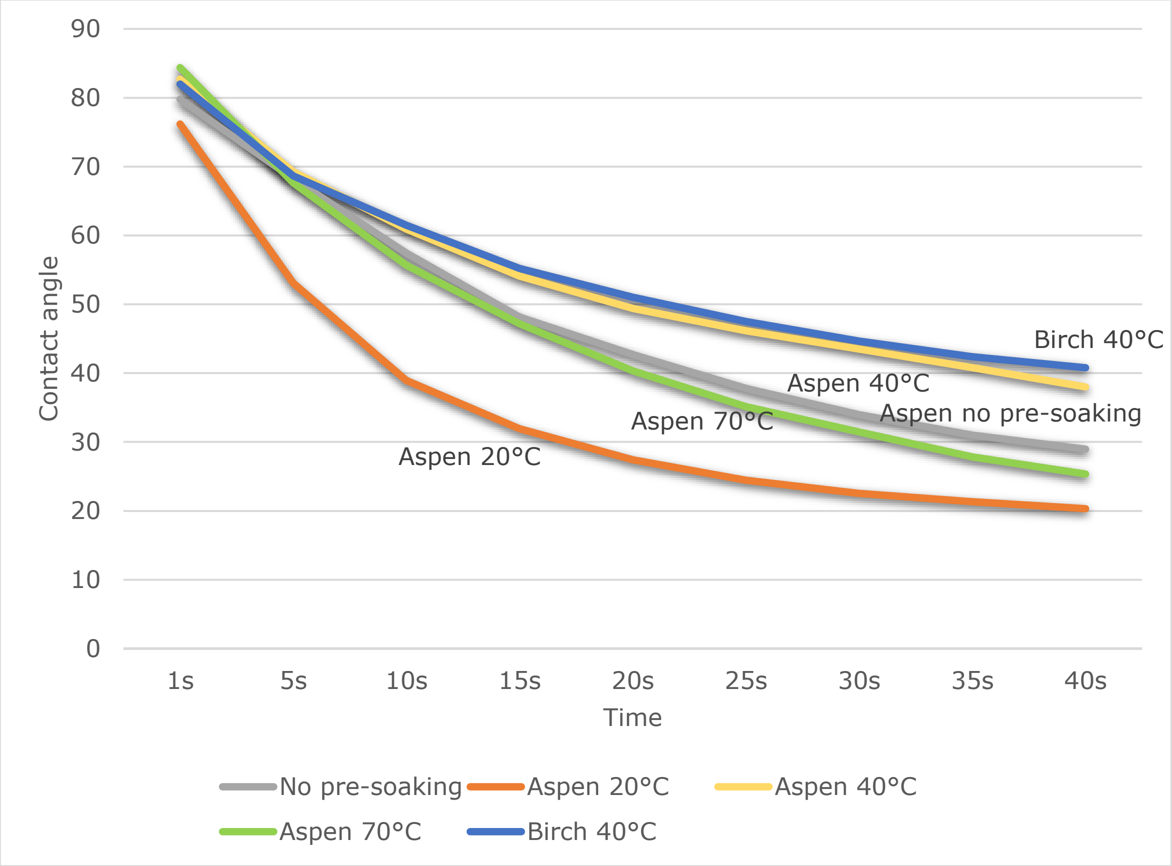

Figure 3.8 Comparison of averages of contact angles

As previously established, aspen 20°C has surface roughness comparable to aspen 40°C, but the moisture content of the veneers differ slightly, having moisture contents of 5.9% (highest for aspen) and 4.2% (lowest for aspen), respectively, which could explain the differences between the contact angles when the surface roughness is similar. Actually, each of the aspen group's moisture content corresponds to it's contact angle – the higher the moisture content, the lower the contact angle.

Again out of all the groups, aspen 40°C has got the highest contact angles and closest to the birch results. In this research, as birch veneer is the standard reference, it is assumed the closer the contact angle is to the birch, the 'better'. Surface roughness of aspen is quite similar among the different groups and it doesn't really affect the wetteability, however the birch veneer is significantly smoother from aspen's and the contact angle is higher and also the data collected for birch has less variation.

The contact angle is found to predict bond strength, however the values of the contact angle that determine the bond strength vary greatly – depending on the wood species and liquid used. For example, a study found that for birch veneer with an average moisture content of approximately 6%, a lower contact angle (CA=31.19) measured with de-ionized water indicated better bond strength than higher contact angles (CA=54.92 and CA=101.31) when testing with ABES method [51].

In general, lower contact angle indicates better wetteability of the material, leading to stronger adhesive bond. However, too low of a contact angle might be a sign of over penetration, leading to a starved joint and causing a weak boundary layer determined to fail more easily.

## 3.3 Thickness, density and glue consumption

Adhesive markings in Table 3.6 and Table 3.7 are: regular phenol-formaldehyde adhesive: PF (Prefere 24J662 resin + Prefere 14J021 hardener); reduced phenol content and added lignin content adhesive: LP (Prefere EXPH 051 resin + Prefere EXPH 9500 hardener). Table 3.6 and Table 3.7 are arranged by the panels made (20 panels, 2 panels per each log soaking temperature). Highest values for aspen are highlighted in light blue, lowest values in yellow.

Table 3.6 Plywood panels density and thickness results

| **Wood species and log soaking temperature** | **Density, kg/m³** | | **Thickness, mm** | |
|---|---|---|---|---|
| | **PF** | **LPF** | **PF** | **LPF** |
| Aspen no pre-soaking | 545.6 | 627.8 | 3.50 | 2.95 |
| Aspen no pre-soaking | 571.2 | 562.5 | 3.17 | 3.17 |
| Aspen 20°C | 570.9 | 594.4 | 3.75 | 3.44 |
| Aspen 20°C | 622.7 | 640.3 | 3.24 | 3.23 |
| Aspen 40°C | 592.8 | 581.7 | 3.54 | 3.65 |
| Aspen 40°C | 566.0 | 599.2 | 3.76 | 3.72 |
| Aspen 70°C | 600.5 | 591.6 | 3.40 | 3.56 |
| Aspen 70°C | 587.6 | 575.8 | 3.46 | 3.56 |
| Birch 40°C | 676.1 | 675.5 | 4.10 | 4.08 |
| Birch 40°C | 667.6 | 689.3 | 4.15 | 3.95 |

It can be seen that birch has got higher density and thickness values for the plywood panels. The tolerance condition of 1 mm was fulfilled among all the panels according to EVS-EN 324-1:2002. The tolerance for nominal thickness was 0.92 mm for both the aspen and birch plywood (nominal thickness = 4 mm) [53].

By checking with a paired t-test with 2-tailed distribution (α=0.05), no significant statistical difference was found between either thickness or density between panels made with either PF or LPF adhesive. By log soaking temperature, no significant statistical difference was found between the aspen plywood panels when carrying out

ANOVA analysis. The only difference was found between birch and every other aspen category. This is explained by the higher density of birch wood, causing the panels to be denser and thicker.

Table 3.7 shows glue consumption by log-soaking temperature and adhesive. Again, best (lowest) values are highlighted in blue, worst (highest) values in yellow. The highest values are considered the worst as they indicate economical loss due to high volume of adhesive required to bond the veneer layers.

Table 3.7 Glue consumption results

| **Wood species and log soaking temperature** | **Glue consumption g/m$^2$** | |
|---|---|---|
| | **PF** | **LPF** |
| Aspen no pre-soaking | 183 | 178 |
| Aspen 20°C | 188 | 155 |
| Aspen 40°C | 191 | 152 |
| Aspen 70°C | 193 | 203 |
| Birch 40°C | 123 | 119 |

It can be seen that glue consumption values for birch are significantly lower for both PF and LPF. When manufacturing the plywood, the aspen veneer sheets had to be put through the roller coater twice, the birch veneer sheets only once. This was because after one pass through the roller coater, every aspen veneer sheet had insufficient glue coverage showcasing dry spots with no adhesive. After a second pass through, the aspen veneer sheets were properly coated with the adhesive. This problem did not occur with birch veneer.

The PF adhesive consumption values overall are higher in almost every case. However, when applying the paired t-test with 2-tailed distribution ($\alpha$=0.05), no significant statistical difference was found between PF and LPF. It must be noted that the sample size is perhaps too small to make any reliable conclusions about PF and LPF adhesive consumption or about the aspen log soaking temperature effect on glue consumption. Figure 3.9 shows the comparison between PF and LPF glue consumption among the different log soaking categories.

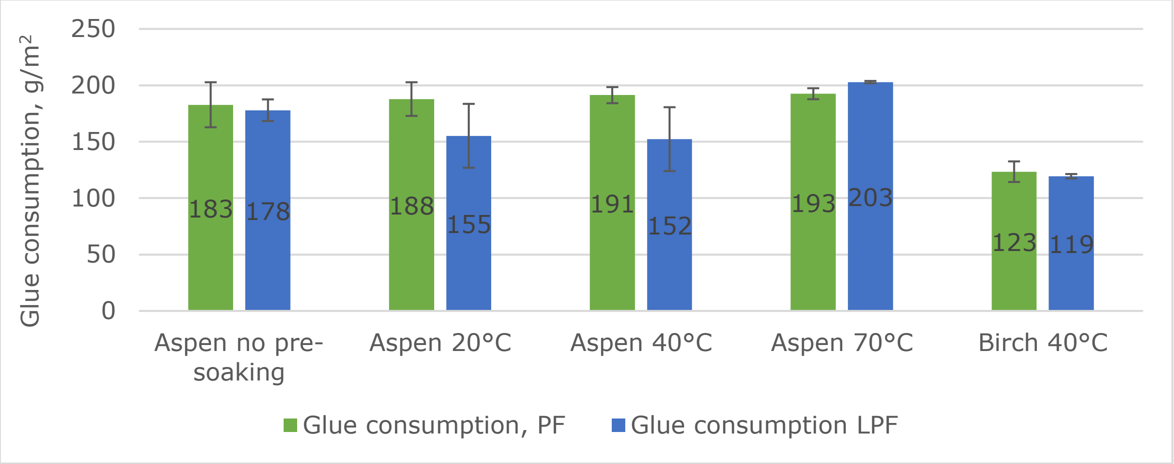

Figure 3.9 Glue consumption comparison between PF and LPF

It can be said that in density, thickness and glue consumption cases, birch plywood consistently outperforms all of the aspen plywood panels. In glue consumption the signficantly higher rates for aspen can be tied to the surface roughness, which also caused the neccessity for a double pass through the roller coaters. Aspen 70°C veneers had the highest surface roughness and it was a statistically different group from others, and the glue consumption was also the highest. Birch had the lowest surface roughness and lowest glue consumption. The contact angle measurements do not predict the glue consumption rates, possibly because as the liquids used were different (distilled water versus PF, LPF adhesives).

## 3.4 Bond shear strength

Firstly, the bond shear strength results are examined in Table 3.8 and in Table 3.9 by the panels made and by the adhesive used (the standard deviation is in brackets). Adhesive markings in the tables are: regular phenol-formaldehyde adhesive: PF; reduced phenol content and added lignin content adhesive: LPF. Highest values for aspen are higlighted in light blue, lowest values in light yellow.

Table 3.8 Bond shear strength results for PF adhesive

| Panel nr | Log-soaking temperature | Wood species | Bond shear strength, MPa | Grand mean, MPa | Adhesive |
|---|---|---|---|---|---|
| 1 | no pre-soaking | aspen | 1.63 (0.21) | 1.59 (0.03) | PF |
| 2 | no pre-soaking | aspen | 1.56 (0.37) | | |
| 3 | 20°C | aspen | 1.83 (0.21) | 1.71 (0.12) | |
| 4 | 20°C | aspen | 1.59 (0.17) | | |
| 5 | 40°C | aspen | 2.01 (0.15) | 1.91 (0.10) | |
| 6 | 40°C | aspen | 1.80 (0.17) | | |
| 7 | 70°C | aspen | 1.92 (0.15) | 1.85 (0.07) | |
| 8 | 70°C | aspen | 1.78 (0.09) | | |
| 9 | 40°C | birch | 2.19 (0.33) | 2.25 (0.06) | |
| 10 | 40°C | birch | 2.31 (0.06) | | |

Table 3.9  Bond shear strength results for LPF adhesive

| Panel nr | Log-soaking temperature | Wood Species | Bond shear strength, MPa | Grand mean, MPa | Adhesive |
|---|---|---|---|---|---|
| 11 | no pre-soaking | aspen | 0.90 (0.07) | 0.96 (0.06) | LPF |
| 12 | no pre-soaking | aspen | 1.02 (0.11) | | |
| 13 | 20°C | aspen | 1.54 (0.15) | 1.44 (0.11) | |
| 14 | 20°C | aspen | 1.35 (0.22) | | |
| 15 | 40°C | aspen | 1.80 (0.25) | 1.80 (0.01) | |
| 16 | 40°C | aspen | 1.81 (0.17) | | |
| 17 | 70°C | aspen | 1.65 (0.12) | 1.61 (0.05) | |
| 18 | 70°C | aspen | 1.56 (0.12) | | |
| 19 | 40°C | birch | 1.79 (0.25) | 1.78 (0.01) | |
| 20 | 40°C | birch | 1.78 (0.33) | | |

After carrying out ANOVA analysis and Tukey-Kramer test (α=0.05), it was noted that among PF results there was no statistical difference between the following groups: aspen 20°C and no-pre soaking; aspen 20°C and aspen 70°C, aspen 70°C and aspen 40°C. All other group pairings were statistically significant from each other. The best result was obtained from birch 40°C (2.25 MPa) and the worst result from aspen with no pre-soaking (1.59 MPa).

Among the LPF groups there was only one pairing that showed no significant statistical difference with the bond shear strength results: aspen 40°C and birch 40°C. It can be seen from Table 3.9 that actually the aspen showed a higher strength result (1.80 MPa) than birch (1.78 MPa). All other pairings of groups showed significant statistical difference. The worst result was obtained again from aspen with no pre-soaking (0.96 MPa).

It would seem that neither the contact angle nor surface rougness measurements would not have predicted the worst result. However, it would have predicted the best result. Aspen 40°C had the lowest surface roughness (closest to birch 40°C) and the highest contact angle (also closest to birch 40°C) yielding the best bond shear strength results form aspen.

Table 3.10 and Figure 3.10 both show that in all cases PF showed higher results than LPF adhesive for bond shear strength. The lowest difference between the results of these adhesives was 5.8% (aspen 40°C) and the biggest difference was 39.6% (aspen no pre-soaking). On average LPF adhesive bond shear strength was 19% lower than PF adhesive. As mentioned before, for both PF and LPF adhesive, the highest shear strength was obtained from aspen 40°C and lowest from aspen with no pre-soaking.

Table 3.10 PF and LPF adhesive shear strength results comparison

| **Log-soaking temperature, °C** | **Wood species** | **Bond shear strength, MPa** | | **Difference, %** |
|---|---|---|---|---|
| | | **PF** | **LPF** | |
| no pre-soaking | aspen | 1.59 | 0.96 | 39.6% |
| 20 | aspen | 1.71 | 1.44 | 15.8% |
| 40 | aspen | 1.91 | 1.80 | 5.8% |
| 70 | aspen | 1.85 | 1.61 | 13.0% |
| 40 | birch | 2.25 | 1.78 | 20.9% |
| | | | **Average:** | 19.0% |

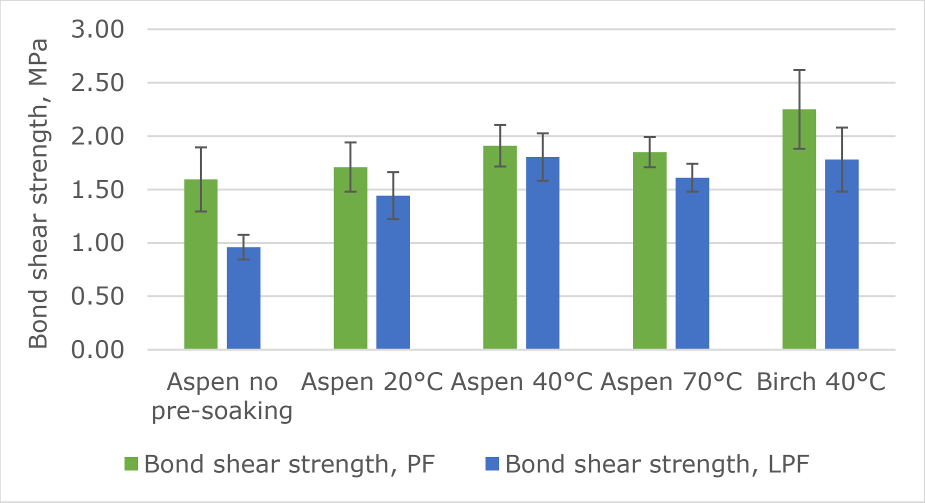

Figure 3.10 Bond shear strength results

The lower bond shear strength of the LPF adhesive could be explained by the effect of the pre-treatment used which inclused soaking and boiling in water. Pizzi and Younesi-Kordkheili state that „Decreasing wet strength of the panels by addition of lignin can be related to the amphiphilic structure of lignin. Lignin has both aromatic (polar) and aliphatic parts (nonpolar) in its structure; some free parts of lignin which do not contribute to cross-linking with phenol and formaldehyde can be degraded by water absorption." [36]. And in addition when discussing a specific type of lignin: "… soda bagasse lignin always contains sulfonic acid groups in lower or higher proportions, and these attract and bind water to such an extent that the water resistance of LPF resins decreases.". [36] Other studies have shown that lignin phenol formaldehyde adhesives provide sufficient dry shear strength (in some case even better than PF) but when it comes to wet shear strength it is always inferior to PF adhesive [36] [54] [55].

According to EVS-EN 314-2 there is no requirement for the determination of visual wood failure if the shear strength is greater than 1 MPa [49]. So only for panel nr 11 (0.9 MPa, aspen no pre-soaking, LPF) it was determined that the visual wood failure was not up to standard as the required minimum mean apparent cohesive wood failure for 0.9 MPa is 40% [49]. The specimens are shown in Figure 3.11. The other parameters that can be tied to this low result are that this panel had the lowest density and lowest thickness. Contact angle and surface roughness measurements however do not indicate a poor bonding strength result.

Figure 3.11 Shear stength specimens after testing, panel nr 11 (bond shear strength: 0.9 MPa; adhesive: LPF; wood species: aspen; log soaking temperature: no pre-soaking)

According to EVS-EN 12369-2:2011, which describes the characteristic values for structural plywood, the shear strength values achieved by laboratory made plywood fulfill the necessary requirement for both PF and LPF adhesives and for both wood species (required minimum planar shear strength 0.9 MPa) [56]. However, it would not be up to par with the requirements in the Handbook of Finnish Plywood, as birch plywood with nominal thickness of 4 mm should have a shear strength of 2.77 MPa [57]. Birch plywood produced in this study has shear strength of 2.25 MPa for PF adhesive and 1.78 MPa for LPF adhesive, falling below the requirement.

## 3.5 Conclusions

Table 3.11 showcases all of the main results obtained during this research for both the veneers and plywood.

Table 3.11 Overall table with the testing results

| | **Quality / Characteristic** | **Aspen no pre-soaking** | | **Aspen 20°C** | | **Aspen 40°C** | | **Aspen 70°C** | | **Birch 40°C** | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Veneers** | Thickness, mm | 1.5 | | | | | | | | | |
| | Moisture, % | 4.15 | | 5.88 | | 5.88 | | 4.54 | | 7.38 | |
| | Ra, μmm | 24.77 | | 24.12 | | 22.91 | | 29.51 | | 13.38 | |
| | Rz, μmm | 152.68 | | 147.99 | | 146.84 | | 179.24 | | 98.12 | |
| | Contact angle (40s) | 28.96 | | 20.32 | | 38 | | 25.35 | | 40.79 | |
| **Plywood** | **Quality / Characteristic** | **Aspen no pre-soaking** | | **Aspen 20°C** | | **Aspen 40°C** | | **Aspen 70°C** | | **Birch 40°C** | |
| | | **PF** | **LPF** | **PF** | **LPF** | **PF** | **LPF** | **PF** | **LPF** | **PF** | **LPF** |
| | Thickness, mm | 3.34 | 3.06 | 3.49 | 3.33 | 3.65 | 3.69 | 3.43 | 3.56 | 4.13 | 4.01 |
| | Density, kg/m$^3$ | 558 | 595 | 595 | 617 | 579 | 590 | 594 | 584 | 672 | 682 |
| | Glue consumption, g/m$^2$ | 183 | 178 | 188 | 155 | 191 | 152 | 193 | 203 | 123 | 119 |
| | Bond shear strength, MPa | 1.59 | 0.96 | 1.71 | 1.44 | 1.91 | 1.80 | 1.85 | 1.61 | 2.25 | 1.78 |

1. It can be said that the log soaking temperature affects the aspen surface roughness only at a higher temperature (70°C), making the surface roughness slightly increase. The increased surface roughness also increased the glue consumption. There seemed to be no correlation between the surface roughness and contact angle nor between surface roughness and bond shear strength in this study when examining the aspen 70°C group.

2. The best values were consistently achieved by aspen 40°C, however according to ANOVA analysis, it was not always statistically significantly different from all other groups. Aspen 40°C had values closest to birch out of all the aspen groups, when observing veneer contact angle, veneer surface roughness and thickness of the plywood panel.
3. The worst bond shear strength values were achieved by aspen no pre-soaking group, however the contact angle nor surface roughness measurements would have predicted it.
4. The phenol formaldehyde adhesive gave a stronger adhesive bond shear strength than lignin phenol formaldehyde adhesive, on average by 19%. This could be explained by water degrading the lignin in the adhesive system during the pre-treatment of the specimens.
5. Aspen’s wood is in general softer, less dense and has got a significantly rougher surface than birch. This causes the plywood panel density, thickness and bond shear strength to be inferior to that of birch’s. However, according to the relevant EVS-EN 12369-2:2011 standard, which describes the characteristic values for structural plywood, the bond shear strength requirements are fulfilled by all the panels manufactured.

# SUMMARY

Aspen plywood has not been researched widely and especially it’s bonding characteristics when veneer is peeled from logs soaked at various temperatures. It is worth researching if it would be suitable for plywood manufacturing according to relevant standards and if it is comparable to birch plywood. Aspen has got a sufficient growing stock in Estonia and could be used to diversify the plywood product line.

In this thesis the aspen plywood bonding characteristics were investigated by veneer contact angle, veneer surface roughness, glue consumption and plywood thickness, density and bond shear strength. The different log soaking temperatures for aspen prior to peeling were no pre-soaking, soaking at 20°C, soaking at 40°C and soaking at 70°C. As a control group, birch veneers were selected for testing and for making plywood with a log soaking temperature of 40°C prior to peeling.

The results showed that a higher log soaking temperature of 70°C increased the surface roughness of aspen, which in turn resulted in a higher glue consumption rate. The higher surface roughness obtained from all the aspen veneer groups, when compared to the lower surface roughness of birch veneer, yielded in a reduced bond shear strength.

It would have seemed that the surface roughness measurements nor the contact angle measurements could have predicted the worst bond strength result (aspen no pre-soaking), however they did indicate what might be the best bond strength result (aspen 40°C). There is no clarity in this matter as ANOVA analysis indicated that aspen 40°C was not always statistically significantly different from the other groups.

Also two different adhesives were used: phenol formaldehyde (PF) and lignin phenol formaldehyde (LPF). When comparing the phenol formaldehyde adhesive and lignin phenol formaldehyde adhesive then phenol formaldehyde adhesive was superior in bond strength. This could be explained by the detrimental effect of water pre-treatment on the specimens of LPF bonded panels.

Birch plywood outperformed aspen in almost every aspect with only one exception in the case of LPF adhesive bond strength where it was inferior to aspen 40°C. Birch plywood had higher density, thickness, bond shear strength, contact angle and lower surface rougness. However almost all of aspen and birch plywood panels fulfilled the relevant EN standard requirements, the only exception being the bond shear strength for aspen with no pre-soaking bonded with LPF.

This thesis found that aspen’s bonding qualities for plywood suffice the relevant EN standards when bonded with PF adhesive. This in turn means that further research could be carried out to find out of how good quality aspen plywood could be manufactured. In the future the bending strength, modulus of elasticity, swelling and shrinking and other parameters could be investigated in the case of aspen plywood to see if it can hold up to appropriate standards.

# KOKKUVÕTE

Haavavineeri ei ole eriti laialdaselt uuritud, eriti tema liimühenduse omadusi, kui spooniks treitavad palgid on leotatud erinevatel temperatuuridel. Tasub uurimist, kuidas haavapuit sobib vineeri valmistamiseks ning kas vineer täidab määratud kvaliteedinõudeid. Haaval on Eestis piisav metsatagavara, et väärtustada seda puiduliiki ka vineeritööstuses.

Antud magistritöös uuriti haavavineeri liimühendust, määrates spooni kontaktnurka, spooni pinnakaredust, mõõtes vineeri valmistamisel liimikulu ja määrates vineeri paksust, tihedust ja liimühenduse kvaliteeti. Erinevatel haavaspoonidel olid palkide leotustemperatuurid järgmised: ilma leotamata palk, 20°C, 40°C ja 70°C. Etalonrühmaks võeti kasespoon, mile palgi leotustemperatuur oli 40°C.

Tulemused näitasid, et kõrgema palgi leotustemperatuuri, 70°C, juures, suurenes haavavineeri pinnakaredus, mis omakorda suurendas liimikulu vineeri valmistamisel. Kõikidel haavaspoonidel oli kõrgem pinnakaredus võrreldes kasespooniga ning neist valmistatud vineeril oli madalam liimühenduse kvaliteet kui kasevineeril.

Ilmnes nagu pinnakareduse ja kontaktnurga mõõtmised spoonidel ei suuda ennustada halvimat liimühenduse kvaliteeti (milleks oli antud uurimuses vineer valmistatud haavaspoonist, mille palki ei olnud eelnevalt leotatud). Kuid siiski nende mõõtmiste parimate tulemuste järgi sai ennustada parimat liimühenduse kvaliteeti (milleks oli vineer valmistud haavaspoonist, mille palgi leotustemperatuur oli 40°C). Siiski, dispersioonanalüüs ANOVA ei määratlenud kõikidel juhtudel haavaspooni ja – vineeri katsetuste tulemusi kui statistiliselt oluliselt erinevaks teistest gruppidest.

Vinneri valmistamisel kasutati ka kahte erinevat liimi: fenool formaldehüüd (PF) ja ligniin fenool formaldehüüd liim (LPF). Kui võrrelda kahte liimi, siis PF andis pea alati paremaid liimühenduse kvaliteedi tulemusi. Seda võib seletada asjaoluga, et katsekehade eeltöötlus seoses vees loetamisega enne liimühenduse kvaliteedi testimist oli kahjustava mõjuga LPF-ga liimitud vineeriplaatidel.

Kask andis pea alati paremaid tulemusi kui haab, välja arvatud LPF-ga liimitud haavavineeri puhul, kui leotustemperatuur haavapalgil oli 40°C. Kasevineeril oli kõrgem tihedus, paksus, liimühenduse kvaliteet ja kasespoonil oli kõrgem kontaktnurk ning madalam pinnakaredus võrreldes haavaga. Siiski, kõik vineeriplaadid peale ühe (LPF, haab ilma palgi eelneva leotuseta) täitsid vastavate standardite nõudeid.

Antud magistritöös leiti, et haava liimühenduse kvaliteet on piisav vineeri valmistamiseks tööstuses ning haavavineer täidab vastavaid EN standardi nõudeid. Edaspidistes uurimustes võiks tähelepanu pöörata haavavineeri paindeomadustele, pundumisele, kahanemisele jt olulistele parameetritele, mille kaudu määrata lõplikult haavavineeri sobivust valmistamiseks ja kasutamiseks tööstuslikul tasemel.

# LIST OF REFERENCES

[1] S. Treial, *SPOONI JA VINEERI TOOTMINE EESTIS,* Tallinn: TTÜ, 2018.

[2] Eesti Metsa- ja Puidutööstuse Liit, „Toodangustatistika - EMPL,‟ [WWW]. Available: http://empl.ee/statistika/toodangustatistika/. [Accessed 07.08.2020].

[3] Erametsakeskus, „Puidu hinnainfo - Erametsakeskus,‟ [WWW]. Available: https://www.eramets.ee/uuringud-ja-statistika/hinnainfo/. [Accessed 08.08.2020].

[4] Ü. Tamm, Haab Eestis, Tartu: Eesti Loodusfoto, 2000.

[5] P. C. Rogers, B. D. J. Pinno, B. R. Albrectsen, G. Li, N. Ivanova, A. Kusbach, T. Kuuluvainen, S. M. Landh€ausser, H. Liu, T. Myking, P. Pulkkinen, Z. Wen ja D. Dominik Kulakowski, „A global view of aspen: Conservation science for widespread keystone systems,‟ *Global Ecology and Conservation,* Vol 21 (March), 2020.

[6] Keskonnaagentuur, „Aastaraamat Mets 2018,‟ Keskkonnaagentuur, 2020.

[7] E. Saarman ja U. Veibri, Puiduteadus, Tartu: Vali Press, 2006.

[8] K. Elias, „Osoon uuris, kuidas Eesti haavapuust valge kuld saab,‟ ERR, 04 08 2017. [WWW]. Available: https://novaator.err.ee/588745/osoon-uuris-kuidas-eesti-haavapuust-valge-kuld-saab. [Accessed 31.07.2020].

[9] R. M. Rowell, %1 *Handbook of Wood Chemistry and Wood Composites*, Taylor & Francis Group, LLC, 2013.

[10] Latvias Finieris, „All Plywood,‟ [WWW]. Available: https://www.finieris.com/en/products/plywood/all-plywood. [Accessed 06.07.2020].

[11] Metsä Wood, „Engineered Wood Products,‟ [WWW]. Available: https://www.metsawood.com/global/Products/Pages/Products.aspx. [Accessed 03.06.2020].

[12] UPM, „Product catalogue,‟ [WWW]. Available: http://www.wisaplywood.com/Products/product-catalogue/Pages/default.aspx. [Accessed 06.03.2020].

[13] „Wisa Plywood,‟ [WWW]. Available: http://www.wisaplywood.com/Products/about-plywood/Pages/default.aspx. [Accessed 19.05.2020].

[14] M. P. Ansell, Wood Composites, Cambridge: Elsevier, 2015.

[15] Columbia Forest Products, [WWW]. Available: http://archive.constantcontact.com/fs058/1102117931485/archive/1102890340140.html. [Accessed 01.08.2020].

[16] A. W. Christiansen, „HOW OVERDRYING WOOD REDUCES ITS BONDING TO," *Wood and Fiber Science,* Vol 22 (4), pp. 441-459, 1990.

[17] R. Shmulsky, Forest Products and Wood Science: an Introduction, Chichester: Wiley-Blackwell, 2011.

[18] R. Varis, Puulevyteollisuus, Porvoo: Bookwell Oy, 2017.

[19] Z. Quin, Q. Zhang, Q. Gao, S. Zhang ja J. Li, „wetteability of Sanded and Aged fast-growing Poplar Wood Surfaces: II. Dynamic Wetting Models," *BioResources,* Vol 9 (4), pp. 7176-7188, 2014.

[20] A. Rudawska, Adhesives - Applications and Properties, InTechOpen, 2016.

[21] R. N. Kumar ja A. Pizzi, Adhesives for Wood and Lignocellulosic Materials, John Wiley & Sons, 2019.

[22] M. Stehr ja I. Johansson, „Weak boundary layers on wood surfaces," *Journal of Adhesion Science and Technology,* Vol 14 (10), p. 1211–1224, 2000.

[23] K. L. Mittal, Progress in Adhesion and Adhesives, John Wiley & Sons, Incorporated, 2015.

[24] E. M. Petrie, Handbook of Adhesives and Sealants, McGraw-Hill, 2000.

[25] A. Pizzi ja K. L. Mittal, Handbook of Adhesive Technology, Marcel Dekker, Inc, 2003.

[26] P. Christjanson, Adhesioon ja adhesiivid, Tallinn: TTÜ, 2004.

[27] A. Pizzi ja C. C. Ibeh, Handbook of Thermoset Plastics / Chapter 2, Elsevier, 2014.

[28] H. Lippmaa, Polümeerisõnastik, Tallinn: Euroülikool, 2001.

[29] S. Ebnesajjad ja A. H. Landrock, Adhesives Technology Handbook (3rd Edition), Elsevier, 2015.

[30] D. D. Stokke, Q. Wu ja G. Han, Introduction to Wood and Natural Fiber Composites, John Wiley & Sons, 2013.

[31] C. R. Frihart, Handbook of Wood Chemistry and Wood Composites / Chapter 9, CRC Press, 2013.

[32] J. K. Fink, Reactive Polymers Fundamentals and Application, Elsevier, 2013.

[33] R. L. Cox, Engineered Tribological Composites - The Art of Friction Material Developement, SAE International, 2012.

[34] M.-P. G. Laborie, L. Salmén ja C. E. Frazier, „A morphological study of the wood/phenolformaldehyde," *Journal of Adhesion Science and Technology,* Vol 20 (8), pp. 729-741, 2006.

[35] A. Pizzi, „Recent developments in eco-efficient bio-based adhesives for wood bonding: opportunities and issues," *Journal of Adhesion Science and Technology,* Vol 20 (8) pp. 829-846, 2006.

[36] A. Pizzi ja H. Younesi-Kordkheili, „Properties of plywood panels bonded with ionic liquid-modified lignin–phenol–formaldehyde resin,“ *The Journal of Adhesion,* Vol 94 (2), pp. 143-154, 2018.

[37] A. F. Anga, Z. Ashaaria, S. Leeb, P. Md Tahirb ja R. Halisa, „Lignin-based copolymer adhesives for composite wood panels – A review,“ *Journal of Adhesion Science and Technology.*

[38] P. Navarrete, H. Mansouri, A. Pizzi, S. Tapin-Lingua, B. Benjelloun-Mlayah, H. Pasch ja S. Rigolet, „Wood Panel Adhesives from Low Molecular Mass Lignin and Tannin without Synthetic Resins,“ *Journal of Adhesion Science and Technology,* Vol 24, pp. 1597-1610, 2010.

[39] Prefere Resins Finland Oy, *SAFETY DATA SHEET. EXPH 9500,* 2015.

[40] Prefere Resins Finland OY, *SAFET DATA SHEET. Prefere 24J662,* 2015.

[41] Prefere Resins Finland Oy, *SAFETY DATA SHEET. Prefere 14J021,* 2015.

[42] Prefere Resins Finland Oy, *SAFETY DATA SHEET. EXPH 051,* 2017.

[43] Mitutoyo Corporation, „Mitutoyo, Product: Surftest SJ-210 [mm],“ [WWW]. Available: https://shop.mitutoyo.eu/web/mitutoyo/en/mitutoyo/1292249246959/Surftest%20SJ-210%20[mm]/$catalogue/mitutoyoData/PR/178-560-01D/index.xhtml. [Accessed 09.31.2020].

[44] ISO, *EVS-EN ISO 4287:1999 Toote geomeetriline kirjeldus ja tehnilised andmed (GPS). Pinnatekstuur: profiilimeetod. Terminid, määratlused ja pinnatekstuuri parameetrid,* Eesti Standardikeskus, 1999.

[45] CEN, *EVS-EN 326-1:2002. Wood-based panels - Sampling, cutting and inspection - Part 1: Sampling and cutting of test pieces and expression of test results,* Eesti Standardikeskus, 2002.

[46] CEN, *EVS-EN 324-1:2002 Wood-based panels - Determination of dimensions of boards - Part 1: Determination of thicness, width and legth,* Eesti Standardikeskus, 2002.

[47] CEN, *EVS-EN 323:2002 Wood-based panels - Determination of density,* Eesti Standardikeskus, 2002.

[48] CEN, *EVS-EN 314-1:2005 Plywood - Bonding quality - Part 1: Test methods,* Eesti Standardikeskus, 2005.

[49] CEN, *EVS-EN 314-2:1999 Plywood - Bonding quality - Part 2: Requirements,* Eesti Standardikeskus, 1999.

[50] I. Aydin, G. Gursel Colakoglu ja S. Hiziroglu, „Surface characteristics of spruce veneers and shear strength of plywood as a function of log temperature in peeling process,“ *International Journal of Solids and Structures,* Vol 43, p. 6140–6147, 2006.

[51] A. Rohumaa, A. Yamamoto, C. G. Hunt, C. R. Frihart, M. Hughes ja J. Kers, „Effect of Log Soaking and the Temperature of Peeling on the Properties of Rotary-Cut Birch (Betula pendula Roth) Veneer Bonded with Phenol-Formaldehyde Adhesive,“ *BioResources,* Vol 11 (3), pp. 5829-5838, 2016.

[52] S. W. Good, *An Evaluation of Aspen Plywood and It's Market Potential in the Cabinet Indusrty,* Michigan: Michigan State University, 1967.

[53] CEN, *EVS-EN 315:2002 Plywood. Tolerances for dimensions,* Eesti Standardikeskus, 2002.

[54] X. Chen, X. Xi, A. Pizzi, E. Fredon, G. Du, C. Gerardin, C. ja S. Amirou, „Oxidized demethylated lignin as a bio-based adhesive for wood bonding,“ *The Journal of Adhesion,* 2020.

[55] „Phenolated Lignins as Reactive Precursors in Wood Veneer and Particleboard Adhesion,“ *Industrial Engineering & Chemical Research,* Vol 55, p. 5231−5237, 2016.

[56] CEN, *EVS-EN 12369-2:2011 Wood-based panels - Characteristic values for structural design - Part 2: Plywood,* Eesti Standardikeskus, 2011.

[57] Finnish Forest Industries federation, „Handbook of Finnish Plywood,“ 2002. [WWW]. Available: https://www.metsateollisuus.fi/uploads/2017/03/30041750/887.pdf. [Accessed 07.05.2018].

[58] CEN, *EVS-EN 828:2013 Adhesives - Wettability - Determination by measurement of contact angle and surface free energy of solid surface,* Eesti Standardikeskus, 2013.

[59] W. D. Detlefsen, Adhesion Science and Engineering / Chapter 20, Elsevier, 2002.

[60] L. Pauling, General Chemistry (3rd Edition), New York: Dover Publications, 1988.

[61] A. Gardziella, L. Pilato ja A. Knop, Phenolic Resins. Chemistry Applications, Standardization, Safety and Ecology, Springer-Verlang Berlin Heidelberg, 2000.

[62] A. Rohumaa, „The impact of log pre-heating on birch veneer surface quality, bond formation and plywood performance,“ Aalto University, Helsingi, 2016.

[63] J. C. F. Walker, *Primary wood processing principles and practice*, Dordrecht, Springer, 2006.

[64] „Wood Handbook - Wood as an Engineering Material.,“ U.S. Department of Agriculture., U.S. Forest Service; Forest Products Laboratory, 1999.

[65] H. P. A. Younesi-Kordkheili, „Properties of plywood panels bonded with ionic liquid-modified lignin–phenol–formaldehyde resin,“ *The Journal of Adhesion,* Vol 94 (2) pp. 143-154, 2018.