

# Department of Materials and Environmental Technology

# THE EFFECT OF MOISTURE CONTENT ON BONDING STRENGTH OF BIRCH SAPWOOD AND FALSE HEARTWOOD

## NIISKUSSISALDUSE MÕJU KASE MALTSPUIDU JA VÄÄRLÜLIPUIDU LIIMLIITE TUGEVUSELE

# MASTER'S THESIS

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### **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.

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# **Table of contents**

Li	st of s	ymbo	ols and abbreviations	5
Li	st of fi	gure	s	6
Li	st of ta	ables		7
Int	roduc	tion		8
1	Lite	eratu	re review	.10
	1.1	Silv	er birch (Betula pendula) and Downy birch (Betula pubescens)	.10
	1.1	.1	False heartwood in birch	.11
	1.1	.2	Manufacturing process and its effect on veneer properties	.14
	1.1	.3	Birch veneer in plywood production	.17
	1.2	Adh	esion properties of veneer	.19
	1.2	.1	Wettability	.19
	1.2	.2	Surface quality	.20
	1.2	.3	Moisture content	.21
	1.3	Rele	evance of adhesives	.23
	1.3	.1	Bond formation	.24
	1.3	.2	Adhesive bond performance	.25
2	Ma	terial	ls and methods	.27
	2.1	Mat	erials	.27
	2.1	.1	Veneers	.27
	2.1	.2	Adhesives	.28
	2.2	Con	tact angle measurements	.28
	2.2.	.1	Sessile drop method	.29
	2.2.	.2	Time dependence of contact angles	.31
	2.3	Plyv	wood manufacturing and testing	.31
	2.3	.1	Manufacturing at a laboratory scale	.31
	2.3	.2	Shear strength	.33
	2.4	Test	t plan	.36
3	Res	ults	and discussion	.38
	3.1	Con	tact angle measurements	.38
	3.1	.1	Time dependence of contact angles	.40
	3.2	She	ar strength	.41

3.3 Percentage of apparent cohesive wood failure	
Conclusions	
References	
Abstract	
Resümee	53

# List of symbols and abbreviations

- CA Contact angle
- EMC Equilibrium moisture content
- FHW False heartwood
- FSP Fibre saturation point
- HW-Heartwood
- LC Lathe checks
- MC Moisture content
- PWF Percentage of wood failure
- RH Relative humidity
- SW Sapwood
- $UF-Urea\mbox{-} formaldehyde$

# List of figures

Figure 1.1. False heartwood types according to shape. Adapted from [13]	12
Figure 1.2. False heartwood in silver birch (Betula pendula). Adapted from [14]	13
Figure 1.3. Manufacturing process of veneer	15
Figure 1.4. Development of lathe checks during rotary-cutting. Adapted from [22]	16
Figure 1.5. Plywood manufacturing process. Adapted from [27]	18
Figure 1.6. Illustrations of contact angles formed by a sessile drop [30]	20
Figure 1.7. Schematic form of imaginary links of adhesive bond. Adapted from [23]	23
Figure 2.1. Contact angle measuring system	30
Figure 2.2. Hot press	33
Figure 2.3. 3-ply plywood shear strength specimen according to EN 314	34
Figure 2.4. Specimens for shear strength testing	35
Figure 2.5. Instron 5866 for measuring the shear strength	35
Figure 3.1. Contact angle values during the first minute	40
Figure 3.2. The effect of moisture content on shear strength	41
Figure 3.3. Cut-out section of plywood's construction	43
Figure 3.4. Interpretation of the direction of lathe checks: a) checks pulled open, b) check	cks
pulled closed	43
Figure 3.5. The effect of lathe checks on shear strength	44
Figure 3.6. The effect of moisture content on percentage of wood failure	45
Figure 3.7. The effect of lathe checks on percentage of wood failure	46

# List of tables

Table 1.1. Properties of Silver birch and Downy birch [3], [4]	10
Table 2.1. Main properties of adhesives	28
Table 2.2. Test plan	
Table 2.3. Precise test plan for making plywood specimens	37
Table 2.4. Precise test plan for testing shear strength	37
Table 3.1. The effect of moisture content on contact angles	

# Introduction

The most valuable species in Estonian plywood industry are light-coloured Silver birch (*Betula pendula*) and Downy birch (*Betula pubescens*), as they cover more than 30% of the forestland. As the quality of plywood is in a relation with the quality of veneer, it is requisite that veneer properties are superior. Birch species that do not form visible heartwood are suitable for the production of veneer except for the possibility of false heartwood (FHW) formation which has different surface properties for bonding. This biological discolouration decreases the yield and quality of birch veneer.

The State Forest Management Centre of Estonia (RMK) has established a change in the log requirements for veneer. Since 2017 the presence of FHW in grade A logs is allowed in the extent of 1/3 of the log's diameter. So far it has only been allowed in lower grade logs. This states that every log even with higher quality will include FHW. However, the research in the field of birch FHW considering the bonding quality is deficient which refers that there is a need to investigate the bonding properties. One amongst several factors that influence bond formation is the moisture content (MC). As the effect of MC on regular sapwood (SW) has been studied broadly, there is a necessity to find how and if MC affects bonding of FHW veneer.

Bonding quality of veneer sheets is dependent on the adhesion properties, namely, on wettability, surface quality and moisture content. Wettability is used to predict the adhesive bond performance and is characterised by measuring the contact angle formed on the examined surface. In bond formation, wettability is the most important prerequisite; if surface's wettability is low, the necessary amount of adhesive for durable bond to form is not absorbed by the surface resulting in insufficient bond strength. Furthermore, the surface quality of veneer is another determinative factor. Wood has a real surface which contains inevitable irregularity and heterogeneity. Surface roughness causes dry-out, as the adhesive gathers to the valleys leaving peaks dry, and overpenetration as the true surface area is increased. The third adhesion property, the moisture content (MC), similarly to the others may be detrimental to the bond formation if selected incorrectly. Most of the adhesives used are water-based which needs to be taken into consideration as wood absorbs moisture. Too high MC may lead to high adhesive flow and penetration into wood leaving the joint starved and on the other hand, too low MC may cause the fast absorption of water inhibiting the penetration of adhesive.

Considering all mentioned before, the effect of MC on bonding strength is examined in this study. *The aim of this thesis is to evaluate how bonding strength of false heartwood veneer is affected by the moisture content.* Two values of MC were chosen for tests: 4% and 8%. The effect of MC on wettability is examined by contact angle measurements.

Bonding strength is evaluated by shear strength measurements of plywood specimens made with three different industrial urea-formaldehyde adhesives: KF-FE from AB "Achema", UF 234 from Hexion Inc. and Profect® 2702 from PKI Supply A/S. Shear strength results are compared to determine whether adhesive properties have also influence on bonding strength.

# 1 Literature review

Literature review covers the most crucial topics in the field of plywood manufacturing, starting from the raw material, moving through the bond formation and ending with the factors which affect the quality of adhesive-wood bond. The aim of the literature review is to gather as much information about the aspects which influence the bonding quality as possible. The backgrounds of wood bonding are well studied but in the field of birch FHW, the research is somewhat incomplete.

#### **1.1** Silver birch (*Betula pendula*) and Downy birch (*Betula pubescens*)

Forests in Estonia cover nearly half of the mainland, namely 49%. More than 30% of the area of Estonian forests is covered with birch stands, being the second largest wood stands after pine with the percentage of 33%. For a long time, birch was considered to have a low value. At first the value started to grow in the paper industry and soon after also in the plywood industry [1].

In the northern hemisphere, the total amount of different birch species is 35 from which 4 are native to Europe [2]. In Estonia two birch species are used to manufacture veneer: Silver birch (*Betula pendula*) and Downy birch (*Betula pubescens*) which is also known as European White birch. The properties of Silver birch and Downy birch are shown in Table 1.1.

Property	Silver birch (Betula pendula)	Downy birch (Betula pubescens)
Height, m	20-30	10-20
Diameter, m	0,3-0,6	0,3-0,6
Average dried weight, kg/m <sup>3</sup>	640	625
Janka hardness, N	5360	4140
Modulus of rupture, MPa	114,3	122,5
Elastic modulus, GPa	13,96	12,03

Table 1.1. Properties of Silver birch and Downy birch [3], [4]

By appearance Silver birch and Downy birch resemble and are difficult to distinguish based on external morphological traits. With closer examination some changes are visible, e.g. the leaves of Silver birch are triangular as the leaves of Downy birch are relatively round [5].

Birch in general is a broadleaved deciduous tree which does not have major climatic limitations, although Silver birch prefers adequate moisture and air content as it grows vigorously in slightly drier conditions when compared to Downy birch. Downy birch survives even on compacted soils and peatland as Silver birch prefers fertile sandy and silty soils [2], [5]. On growing sites where both are presented, Silver birch is considered to grow faster and has higher yield [2].

Birch is suitable wood species to be used in the plywood industry. It has a straight to slightly wavy grain with even texture which contributes to the ease of surfacing. Furthermore, the workability of birch is good with machine tools as well as by hands [6]. Birch is suitable for bending making it practical in the production of bendable plywood. Considering drying, the process is fast but the tendency of warping is possible. Birch is mainly used for mass production than high-quality furniture production due to its pale and inexpressive appearance [7]. Moreover, it is soft making it easy to rotary cut and plane. Density is in the range between 630 to 670 kg/m<sup>3</sup> [6]. By its nature, birch is a perishable species as it rots and decays in exposure to microorganisms [3]. This property influences negatively the use of birch as it decreases the yield and quality of end-products.

#### **1.1.1 False heartwood in birch**

As the trees are affected by the inevitable aging process, some changes within wood are carried out. One of the most visible effects of aging is known as heartwood (HW), a darker area in the heart of the trees in contrast to the light-coloured sapwood (SW). In some wood species, i.e. birch, alder and beech, the formation of visibly darker heartwood does not occur. These species may form a discolouration, which is similar to HW, but is known as false heartwood (FHW). This discolouration is also termed as facultative heartwood, red heartwood, stained wood etc., as it does not have an official term.

FHW is a nonhomogeneous discolouration which decreases the degree of utilization of wood and results in an extensive value loss as it limits the applications of logs to rather lower-value end-products [8], [9]. As regular heartwood develops, tissues darken with the death of living cells and depletion of nutrients. FHW is generally related to wood's response to wounding forming the first stage of FHW formation. One assumed factor which causes the severe discolouration within living tree is the activity of microorganisms where discolouration is triggered by the interactions between attacking microorganisms and the living xylem wood cells. Cell wall substance is further on degraded by microorganisms which leads to the death of cells as with regular HW [10]. The other assumed factor is the presence of oxygen within in the trees. It is stated that FHW forms in the axial direction of the tree stem when living parenchyma cells interact with oxygen which enters the stem through injuries or dead branches [11]. As air penetrates into the stem, living cells as a defence begin to change anatomically and chemically by oxidation and development of coloured substances. This provides the prevention of further penetration. Emerged substances deposit on the cells not into the cells as in true HW formation [8]. The amount of penetrating oxygen needs to be high enough not to be consumed by the respiring SW and injury big enough to kill most of SW at the entry point to allow such amount of oxygen into wood. As the development of FHW takes several years and spreading of oxygen only few days, then oxygen cannot be considered as the only factor but oxygen concentrations influence the activity of micro-organisms [11]. This biochemical discolouration of wood is caused by several factors involving biological reactions and presence of oxygen [12].

The contour of FHW does not coincide with the growth rings. In the cross section FHW may be visible as radial, star-like or completely irregular in shape [8]. Figure 1.1 illustrates different shapes of FHW.



Figure 1.1. False heartwood types according to shape. Adapted from [13]

Figure 1.2. shows four cross sectional images of birch disks cut from different lengths of the stem: a) at breast height (1,3 m), b) at  $\frac{1}{4}$  of the stem's height, c) at the half way between of heights b and d, and d) at the diameter of 8 cm. Based on these images, it is evident that the presence of FHW is highest in the lower part of the stem, decreasing with the diameter of the tree [14].



Figure 1.2. False heartwood in silver birch (Betula pendula). Adapted from [14]

As shown in image above, FHW column develops following the longitudinal axis of the stem. The extent of FHW depends on the severity of the wounds and how vigorously tree grows [10]. Based on research results show that FHW initiates from external traits such as dead branches, knots, burls and scars. In a study where the relation between external traits and FHW was investigated it was shown that more than 73% of the external traits were associated with FHW. Despite that burls were not directly connected with FHW, they are good indicators of a dead branch or a branch scar being covered with wood [9].

It is noted that age, average diameter growth rate, height from the ground, and external traits influence the occurrence of FHW [9]. On average the formation of FHW starts in 40-year-old trees [15]. Considerable discolouration is present in trees older than 70-year-old [9]. Studies showed that at an age of 80 years to age of 180 years the proportion of FHW increases from 30% to 95% of the stem's cross section. Moreover, trees with a fork showed a tendency of 50% to form FHW as trees without fork only 30% of the cases. [13]. Term "fork" refers to the presence of two stems with equal diameter growing up from one stem. The effect of FHW on the mechanical properties, such as wood's strength and Janka hardness has not been detected to be remarkable [9]. It is somewhat unclear how and if FHW affects other wood properties as the essence and origin remains unclear.

#### **1.1.2 Manufacturing process and its effect on veneer properties**

Veneer's properties are in a correlation with the manufacturing process. As the quality of processing equipment has been enhanced, it has raised also the quality requirements for veneer sheets. This chapter sets focus on the manufacturing of rotary-cut veneer which is used to make plywood.

Following chart illustrates the general manufacturing process of thin veneer starting from log grading and ending with splicing (see Figure 1.3). Logs in Estonia are graded based on the requirements set by the State Forest Management Centre (RMK). Birch logs are graded as A and BC quality, A marking superior quality and BC inferior, respectively. According to the quality requirements any kind of branches, fissures and soft rot in grade A logs are not allowed; the presence of FHW is allowed only as much as 1/3 of the log's diameter. Grade B logs may contain branches and FHW at any diameter.



Figure 1.3. Manufacturing process of veneer

Heat treatment, termed also as hydrothermal treatment, refers to conditioning the logs under humid conditions, mainly at elevated temperatures [12]. Heat treatment can be done by steaming, soaking or spraying with hot water with the objective to obtain an increased temperature within log as deep as the veneer is peeled [16]. Rotary-cutting implies to producing veneer by revolving a log against a lathe knife, therefore, forming a continuous flat-grained sheet [17]. Peeling occurs subsequent to the heat treatment and debarking which means that green, undried veneer is peeled and afterwards graded according to the visual defects. Substandard parts of the sheet are cut out in the whole width of the sheet and added to waste. The yield from peeled veneer is generally between 60-70% [16]. A narrow "pencil" is the residue of peeling process.

After peeling the wet veneer is dried. In birch plywood industry, the temperatures for veneer drying are about 160-180 °C and drying time 3-4 minutes [18]. Production capacity of veneer production can be achieved by increasing the temperature which reduces the drying time. However, the effect of high drying temperatures on bonding strength is considered to be negative [19]. Drying should occur as fast as possible without causing damage considering species and thickness. Control over the drying process is the key to successful drying. Drying conditions, such as temperature, air circulation, drying time and RH, are critical and may cause severe damage if applied improperly. Improperly controlled drying can lead to drying defects which affect further bonding– surface inactivation, risk of stain, etc. [20]. Moreover, excessive drying can cause following defects: resin exudates, checking, splitting, warping and casehardening etc. [21].

As shown in Figure 1.4, knife moves forward as the log revolves around its axis steadily. Veneer sheet with a constant thickness is forced away from the log at a sharp angle which causes checking on the knife side which are known as lathe checks (LC).



Figure 1.4. Development of lathe checks during rotary-cutting. Adapted from [22]

The knife side is termed as the loose side and the opposite side as tight side. When making plywood, tight side should be finished and loose side bonded. If used oppositely, lathe checks may cause imperfections in the finished surface as open checks absorb more finish. If the adhesive spread rate is set correctly the overpenetration of adhesive into LC is not considered to be problematic [23]. Pressure bar is used on the concave (tight) side to develop pressure high enough to reduce checking and to minimize roughness [16].

The quality of veneer is in correlation with the quality of lumber. The quality of lumber, on the other hand, is related to the size and quantity of defects, such as resin pockets, knots, cracks, fibre misalignment, and other abnormalities, e.g. rot and discolouration. They all contribute to the final value [24]. Furthermore, veneers' properties do not generally differ from properties of lumber; however, the change may occur during manufacturing processes, e.g. cutting and drying [25].

Peeling is one of the manufacturing processes which affects veneer's properties to a higher level of extent. The extent can be adjusted by the preceding heat treatment. The surface of peeled veneer is somewhat rough and irregular, especially the loose side with checking, but heat softens the wood and reduces cutting power consumption, and as wood gets softer it will be easier to cut resulting in comparatively smoother higher-quality veneer [16], [17]. Required heating time depends on density, diameter, temperature of water, initial temperature of wood,

and temperature required for smooth cut [16]. Heat treatment via soaking allows to keep the MC consistently high and provides uniform distribution of temperature within wood [18], [26].

In case of drying, it is important to avoid surface inactivation which can be caused by drying at very high temperatures for prolonged periods to achieve low MC value. Surface inactivation can be due to natural cause as well, as the extractives in wood diffuse to the surface that is freshly cut or planed [23]. Inactivation results in poor wettability which on the other hand results in poor bonding which affects the quality of end-products. Drying also causes discolouration which does not have an impact on wettability but decreases the value considering the appearance [12].

#### 1.1.3 Birch veneer in plywood production

Silver birch is the most widely used wood species in Estonia in plywood production. Plywood used in furniture industry needs to be superior by its quality and meet the standards which FHW veneer cannot reach. FHW is considered not to have the required level of quality and for that reason it is being treated as waste to burn for energy.

Plywood is a cross-laminated wood-based panel in which neighbouring layers have different grain direction. By its composition, it has a multi-layered structure with even or odd number of veneer sheets [24]. The number of plies is always an odd number but the number of veneer sheets does not have to be certainly odd. For example, it is possible to produce plywood with 3 plies and 4 sheets of veneer. The grain direction of 2 inner sheets needs to be perpendicular to the grain direction of face veneers.

The perpendicular grain direction of adjacent layers results in a panel with the best ratio between strength and weight [24]. Comparing with solid wood the main advantages of plywood are greater resistance to splitting and checking, equal strength properties along the length and width throughout the panel, dimensional changes nearly equal in length and width as MC value changes, and the efficient utilization of wood raw material. [17]. As wood is characterized by anisotropy, the properties in plywood panels are equalized in both directions, in length and in width.



Following chart illustrates the manufacturing process of plywood (see Figure 1.5).

Figure 1.5. Plywood manufacturing process. Adapted from [27]

The preparation of veneer sheets was visualised in Figure 1.3 in previous sub-chapter 1.1.1. The manufacturing of plywood begins by sorting out veneer sheets followed by preparation of veneer sheets and adhesive mixture. In plywood industry, the glue spreading is regulated to ensure that right amount of adhesive is applied considering the surface roughness. Pre-pressing of laid-up panels in a cold press before hot-pressing is used to reduce misplacement of veneer sheets. Hot-pressing is done in hydraulically powered presses to cure the adhesive by resin polymerization and make the contact between sheets as tight as possible. Pressures used in hot-pressing vary from 0,75 MPa to 1,5 MPa, depending on the density [16].

The MC of veneer sheets before pressing into plywood is important factor influencing the bonding quality. The MC of opposing plies needs to be as similar as possible when glued. If the plies have varying MC values, the shrinkage/swelling properties of different areas vary and may result in warping of the panel [17]. A more common issue with plywood, besides warping, is panel delamination, which occurs when the adhesive bond does not show strength high enough in many cases due to surface roughness of veneer sheets [28]. Likewise, delamination is dependent on the MC. MC and its effect is described more specifically in chapter 1.2.3.

#### **1.2** Adhesion properties of veneer

This chapter points out three main characteristics of veneer surfaces which define the extent of adhesion: wettability, surface quality, and moisture content.

Main requirement for satisfactory bond formation is the preparation of the surface by making it clean, smooth and chemically receptive considering the adhesive used [29]. This preparation determines the bond strength and its resistance to aging and promotes the adhesion which has the utmost importance in bonding. Surface properties affect wettability, flow and penetration that might disturb bond formation and further on the quality of end-product [25]. The apparent surface of wood is a layer of extractives, dirt, oil and other contaminants which affect adhesion [29]. Such contaminants are hydrophobicc by their nature which interferes with adhesive bonding with wood [23]. Preferably, adhesive should be spread on a surface that has been recreated by sanding or planing within 24 hours [25]. Once the surface is re-created and cleaned from debris, the substrate needs to be handled as little as possible before bonding [29].

Considering the MC, it is preferable if the wood is dried to the level of MC compliance with its end-use condition [29].

#### 1.2.1 Wettability

From different surface properties, wettability is the one which helps to describe how the surface acts in gluing processes and predict the performance of adhesive-wood bond. Wettability is often characterized by measuring the contact angle (CA) which is formed when liquid is placed on the surface.

The foremost prerequisite for bond formation is the wetting of the wood surface by the liquid adhesive. Three main parameters are bound with the wettability of a surface – CA formed on the wood surface, the spreading of the liquid, and the penetration of the liquid into the porous substrate [24]. Based on the value of CA formed on the surface, the wetting behaviour can be determined which indicates also the behaviour in bond formation.

If CA is less than 90° the wetting of the surface is favourable. When CA approaches to  $0^{\circ}$ , surface has high wettability and liquid spreads across the surface. Liquid forms beads on the surface when CA is more than 90° and these kinds of surfaces are described as hydrophobic (see Figure 1.6). Hydrophobicity of surfaces disturbs adhesion and bond formation [30].



Figure 1.6. Illustrations of contact angles formed by a sessile drop [30]

As the surface of wood is heterogeneous, hygroscopic, porous, and rough, it is challenging to measure the wettability through CA measurements. The intrinsic cell structure pronounces surface roughness specific to wood and with different machining operations, the surface structure will be even more damaged [31].

Hydrophobic extractives within wood influence bonding both physically and chemically [24]. As they are stored in resin channels, then after machining operations as a fresh surface is revealed some of them migrate to the exterior layer. This results also in making the surface hydrophobic [31]. While measuring the wettability, also the influence of seasonal variations needs to be considered. This aspect is particularly essential when using wood harvested in winter. In winter, the freshly harvested wood has a higher content of extractives. The CA measured on a freshly harvested winter wood are higher than the ones of stored wood as wood components oxidize during storage and this reduces the diffusion of extractives to exterior layers [24].

#### **1.2.2 Surface quality**

In case of veneer, the examined surface is defined as a surface containing natural roughness and heterogeneity.

The surface qualities of substrates are extremely important to satisfactory bond performance. The wood surface should be smooth and free of contaminants and surface irregularities. Otherwise, surface interferes with wetting, adhesion and as well as with development of bond strength [25]. Veneer roughness is affected by mechanical processing variables, e.g. knife cutting angle, sharpness, peeling rate and feed rate of the veneer [32]. Surface roughness can be minimized by sanding or planing. Before the adhesive is spread on the surface, surface needs to be renewed and made chemically receptive by removing contaminants. After 24 hours, the surface inactivation takes place and the ability to bond is interfered [25], [33].

Wood surfaces may appear smooth by visual examination, but microscopic examination reveals peaks and valleys which are the causes of blockages and air pockets preventing the complete wetting of a surface. After the adhesive has cured these conditions lead to stress concentrations [23]. Furthermore, the presence of peaks and valleys results in adhesive bonds with inferior quality as the contact between two substrates is not as intimate as could be with smooth veneer when veneer sheets are rotated by 90° [32]. Roughness promotes conditions of dry-out and overpenetration of adhesive. Considering the conditions of dry-out, adhesive flows into the valleys and but the peaks as the primary surfaces for bonding are left without necessary amount of adhesive. Result is a starved joint. In case of overpenetration, as the surface area increases the proportion of glue decreases [28].

The adhesive bond strength is higher with smooth surfaces. Smoothing rough surface enhances bonding during the hot-pressing cycle. As roughness increases, the glue consumption also increases leading to higher production costs. In order to decrease production costs and increase the quality, veneer surface properties need to be considered [34].

#### **1.2.3 Moisture content**

Moisture content has impact on wettability, penetration, flow and cure of aqueous adhesives. Generally, optimal adhesion properties are achieved between 6% and 14% MC [23].

Water in the cell cavities is in the form of free water, and water in the cell walls as bound water. When wood dries the free water starts to evaporate. When all of the free water has evaporated from the cavities, yet cell walls are fully saturated, the fibre saturation point (FSP) has been reached. If drying continues, cell walls start to lose water until the amount of bound water has reached equilibrium with the surrounding moisture. This MC is known as the equilibrium moisture content (EMC). EMC can be expressed as a ratio of RH and temperature [35]. As wood strives towards the balance with surrounding environment, it shrinks and swells depending on the MC of surroundings. Swelling occurs when the FSP has been achieved and the MC of environment is higher. On contrary, shrinkage takes place below FSP [36].

The cell walls of wood are hygroscopic by their nature as the structure is made of cellulose and hemicellulose which have several hydroxyl (OH) groups. Lignin as binder of cellulose and hemicellulose is relatively hydrophobic which means that the hydrophobicity of cell walls is limited by lignin [37]. Polar liquids like water have the ability of forming hydrogen bonds with OH groups present in cell walls [38].

During gluing the MC of wood plays important role as it affects the bond quality and the performance of the panel in service [17]. High temperatures used during hot-pressing cause the panel to adopt a lower MC under certain RH. It is recommended that veneer sheets should be conditioned at 30-40% RH for interior use and 65% RH for exterior use [20]. Before gluing, the MC of veneers should be below 7%. A MC of 3–5% at the time of hot-pressing is labelled as satisfactory for hardwood plywood for furniture production [19]. Variations in MC release internal tensions and in many cases, such variations are the causes of distortions [36]. Moreover, it is crucial to take under consideration the moisture that is added by the adhesive which usually contain water as a carrier. The MC of wood united with the water from glue needs to be as near as possible to the MC attained in service [17]. The amount of adhesive is regulated by distribution. If the adhesive is distributed unevenly on the surface, the moisture will likewise be distributed unevenly which on the other hand causes problems with the panel's stability in the form of warping [36].

When waterborne adhesives are applied to wood with low MC value, a condition termed as dryout can occur. Wood with low MC absorbs water from the adhesive mixture fast and the adhesive flow and penetration into wood is inhibited. It is found that below 3% MC wood may resist wetting as the water within wood is not sufficient to establish intermolecular attraction forces with the water in adhesive [23]. Furthermore, very dry veneer is difficult to handle as it cracks easily and reabsorbs moisture [17]. On the other hand, too high MC values can lead to higher flow and enhanced penetration of adhesive into the wood resulting in starved adhesive bond lines. In addition, high amount of water within wood results in high steam pressure generated during hot-pressing which expresses as blistering [24]. The superior bonding results considering bonding strength are named to be obtained with veneer sheets having 4–6% MC [39].

#### **1.3 Relevance of adhesives**

Adhesives are used in various wood products to increase the strength and stiffness in compression and shear by means of transferring and distributing loads between component [23]. The adhesive bond between two substrates is imagined as a chain where the stresses and loads are transferred from one link to another (see Figure 1.7). In the end, the collaboration of the individual links in the chain determines bond performance [23].



Figure 1.7. Schematic form of imaginary links of adhesive bond. Adapted from [23]

Four main requirements exist for a material to perform as an adhesive. First of them, the wetting of the surface; adhesive must wet the surface by flowing over the surface and displacing all air and other contaminates present. Secondly, after flowing over the surface it must adhere to the surfaces by becoming tacky. Third requirement is the development of strength which takes place after resin has polymerized or dried. And the fourth requirement is to endure its stability by remaining unaffected by age, environmental conditions and other factors which may influence the bond [29]. Besides these four requirements, the choice of the adhesive depends on the properties required for wood-based panels, on the production conditions, and often on the expenses for the adhesive system, including glue spread rate, shelf life, production capacity of the panels and etc. [24].

Wood adhesives contain water as a carrier as wood absorbs water and it is an inexpensive and environmentally friendly solution [27]. The whole gluing process is dependent on the wood's ability to absorb moisture from the adhesive [36]. The role of water in the bond formation is crucial but the amount needs to be set correctly. As it was stated in the previous paragraph, adhesive must wet the surface, but too high proportion of water results in extensive flow which on the other hand can alter bonding quality [24].

#### **1.3.1 Bond formation**

Wood-adhesive bond is more than a visible surface bond line due to adhesives' ability to penetrate into wood [40]. The bond formation phenomenon is affected by numerous factors in the production process, e.g. pressure, temperature and assembly time, as well as by substrate's surface quality and adhesive's properties. If these factors are controlled within a reasonable range about the optimum for each, high-quality adhesive bonds are received [17].

During bond formation, the adhesive goes through three stages. In the first stage, the adhesive flows over the surface and wets the substrate. This presumes that the surface energy of the substrate is higher than the one of adhesive. The difference in surface energies between the substrate and the adhesive is determined by the CA [41]. As the liquid adhesive flows over the surface the molecules of adhesive must come into direct contact with molecules of wood. In this way, intermolecular attraction between adhesive and wood is provided and mechanical interlocking ensured [23].

In the second stage liquid adhesive turns into a solid via polymerization into cross-linked structures, loss of solvent through evaporation and diffusion into wood or solidification from melt [41]. As water is used as a carrier for most wood adhesives, loss of water and chemical polymerization often takes place concurrently [23]. Rheological properties are also important considering the viscosity of the adhesive; highly viscous adhesive may not wet the surface as much as necessary. During polymerization, the viscosity increases which means that adhesive loses gradually its ability to wet the surface by the time that the surface needs to be covered by adhesive. In the last stage, the bonded assembly needs to retain its ability to stay intact under end-use conditions [41]. The bond is formed once the adhesive turns into a solid, but the strength of the bond may develop for days until it has reached its full strength [23].

The end-product depends on bond formation which makes it important to understand the interaction of the adhesive with the wood. One accepted method to determine the performance of adhesive is to produce a product and apply tests based on standards. By understanding the interactions, the possibility of achieving better results can be achieved through product development [41]. There can be problems with the adhesive, substrate or their combination. Weak bond line can be caused if the adhesive has either dried or cured too much before hot-pressing and as pressed together with adjacent sheet it does not wet the surface [36].

In wood panel industry, the relative importance of the technology of the adhesive is equal with the importance of the technology of manufacturing the panels. It is said that "One can make very poor panels using an excellent adhesive, and still make good panels when using a poorer adhesive" [24].

#### **1.3.2** Adhesive bond performance

Bond performance is a very important issue in the production of wood-based panels. Bond performance can be characterized with bond strength or with the resistance to aging and weathering.

It was outlined in the beginning of chapter 1.3 that wood-adhesive bond visualized as a chain of interrelated links: wood layer, wood interphase, the interface of wood-adhesive, adhesive interphase and adhesive layer. Bond performance is determined by the weakest link [24].

The bond performance depends on the properties of the adhesive and the substrate. Wood surface plays a decisive role on bonding and on the quality of wood-based panels. Low or even no bond strength can be caused by unfavourable properties of the wood surface, e.g. due to low wettability [24]. Penetration is important factor considering the bond performance since a strong adhesive bond requires that the surface is covered with the adhesive despite the roughness and cavities of the wood surface [42]. During hot-pressing adhesive partly penetrates into the wood surface. Whether the extent of penetration is too high or low, bond is negatively affected. Adhesive overpenetration results in starved adhesive bond lines, on the other hand, low penetration inhibits the contact surface between substrate and adhesive [24].

Primary mechanism when considering wood bonding is mechanical interlocking. If the adhesive is able to penetrate two to six cells in depth into sound wood, the interlocking between substrates is effective. Moreover, deeper penetration increases the surface area of contact between wood and adhesive, resulting in even stronger bond which by strength can even exceed the strength of wood [23]. Penetration also promoters a greater distribution of stresses during hot-pressing [42]. It is believed that when adhesive penetrates deeply into the cell cavities and diffuses into cell walls, the most durable bond can be achieved. In this way, the adhesive makes a contact with cellulose and hemicelluloses in molecular level [23].

Due to the long lifespan of bonded products, accelerated tests are usually used to determine strength [41]. Accelerated tests are used to determine different mechanisms of bonding which are helpful when reaching a conclusion if the same phenomena exist in service. In most of the cases, the behaviour of wood-based panels with conventional materials is known and need for further testing is only necessary when experimenting with new materials [40].

## 2 Materials and methods

Based on the material gathered in the literature review, an experimental part has been assembled. It is divided into two main divisions: CA measurements and the testing of manufactured plywood specimens.

#### 2.1 Materials

This research is focused on characterizing two different veneer surfaces: regular birch SW and birch FHW. Plywood panels are made by using three different adhesives to find the most appropriate which provides better results considering the shear strength and make conclusions about the properties of adhesives that could influence bonding strength. Moreover, the effect of MC on the bond strength is likewise examined. Considering literature and previous research, veneer sheets were conditioned to 4% and 8% MC.

#### 2.1.1 Veneers

Veneer was delivered from Tarmeko Spoon AS. There the logs are soaked at the temperature of  $35-40^{\circ}$ C for 24 h. After peeling veneer is dried at 140-160°C, drying rate is 4-5 m/min. The MC of veneers when leaving Tarmeko is approx.  $6\pm 2\%$ . Veneer thickness is 1,5 mm.

In order to receive reliable results and to ensure that the comparison between regular SW and FHW is accurate, the samples need to be taken from the same batch and preferably from the same sheets. This fact increases the amount of labour as the size of samples is limited. The smaller the sheets are, the smaller are plywood panels produced and the less samples can be cut from the panel. For example, when the panel size is 260x260 mm, approximately 18 pcs of 120x25 mm can be cut from it. Furthermore, it the panel size is 200x200 mm, only 6-7 pcs can be received.

Furthermore, the surface quality of every piece needs to be as similar as possible which also limits the number of samples.

## 2.1.2 Adhesives

Three different UF adhesives are being tested: KF-FE from AB "Achema", UF 234 from Hexion Inc. and Profect® 2702 from PKI Supply A/S. Their properties are shown in Table 2.1.

Properties	Achema KF-FE, Casco 2545 hardener	PKI Profect® 2702, Profect 9708 hardener	Hexion UF 234, MUFH 6028 hardener		
Dry matter, %	67±1	68	59-61		
Relative viscosity, mPa*s	75-140	2250	300-600		
рН	7,5-8,7	8-9	8,5-10		
Applications	Plywood production, mould-pressing	Bended plywood production	Plywood production		

Table 2.1. Main properties of adhesives

#### 2.2 Contact angle measurements

CA is defined as the angle between a solid surface and the surface of a liquid in contact with it. It is used to predict adhesion properties, determine the surface tension of a liquid and calculate surface free energy of a solid [43].

CA value can be measured by using a telescope with a goniometer eyepiece or taking a photograph of a liquid drop on a substrate. The most widely used technique for measuring the CA is a direct measurement of the tangent angle at the three-phase contact point on a sessile drop profile [30].

Due to the intricate nature of wood, CA measurements are fairly troublesome. The porosity, hygroscopic nature, anatomic complexity, heterogeneity and extractive content are some characteristics of wood which affect the measurements. Moreover, measurements are dependent on time of measuring the angle as a drop of water is absorbed into the surface and the size is in constant change. It is a quite unsatisfactory approach from both an experimental and a statistical point of view as it is highly dependent on many variables [31].

#### 2.2.1 Sessile drop method

This study focuses on finding an apparent CA with drop shape method using a sessile drop on the surface of birch veneer.

Sessile drop is the standard arrangement for measuring CA. The CA is measured using the image of a sessile drop at three-phase contact line between the drop contour and the projection of the surface. A drop acquires a spherical shape in order to minimize surface area [44]. The camera in the measuring system may be placed to visualize the drop horizontally or looking downwards at a small degree, for example 3°. A small down-looking angle affects finding the baseline which in turn affects the reading. Baseline inaccuracy is the primary contributor to inaccuracy of CA values [45].

Instrument used to measure CAs was video-supported instrument DataPhysics OCA 20 (shown in Figure 2.1) and for analysis of the drop contour and determining the value SCA 20 software was used. Object of research is a static sessile drop. Testing system consisted of 1) lighting, 2) sample carrier, 3) dosing needle with solution, 4) optical system catching the image of a drop, and 5) screen presenting the image [46].



Figure 2.1. Contact angle measuring system

Firstly, the measuring device and analysis software needs to be switched on. Sample is placed onto the carrier and fixed with clamps or tape to ensure that the sheet is fixed tightly and straight. Surface needs to be as even as possible in order to achieve accurate results. The sharpness of the image displayed in the screen can be adjusted with zooming. Secondly, when the image has been adjusted, few drops are dripped onto the surface for testing if the software detects the baseline of the drop correctly. If so, then testing can continue. One drop is dripped onto the specimen and measurement shall be started immediately after the completion of dosing. The dosing in this research was done by hand and image taken as soon as possible. Image needs to be taken during 15 seconds according to the standard EVS-EN 828:2013. CA values were measured on the tight side of the veneer.

#### 2.2.2 Time dependence of contact angles

As the values received from CA measurements can be associated with wettability, the immediate CA value does not always reflect the accurate condition of the substrate considering its wetting properties. CA values of a certain liquid change in time, making wettability also time-dependent. Surfaces with different properties absorb liquids variously; time dependence of wettability is the rate of absorption.

In order to compare regular SW and FHW, it is necessary to compare the results after a certain period of time. In this research, the period of time was set to be 60 seconds. Liquid which was chosen to form the CA was distilled water. First value was measured right after dosing the drop; next values were measured as follows:

- 0 s 1 s at interval of 125 ms;
- 1 s 30 s at interval of 5 s;
- 30 s 60 s at interval of 10 s.

In order to determine the time dependence of wettability every drop is recorded, and afterwards the CA values can be measured from the recording.

#### 2.3 Plywood manufacturing and testing

The second division of the experimental part – testing of plywood bond quality – includes conditioning veneer sheets to a certain MC, laying up the panel, hot-pressing, cutting specimens and testing shear strength.

#### 2.3.1 Manufacturing at a laboratory scale

Prior hot-pressing veneer sheets are conditioned in a climate chamber under following conditions:

- 1) 20 °C and 40% RH 8% MC;
- 2) 20 °C and 15% RH 4% MC.

Specimens were held in a climate chamber for at least 24 hours to achieve required MC.

As wood absorbs moisture from the surrounding environment and the conditions in laboratory were not controllable, veneer sheets were wrapped into a cling film as tightly as possible to prevent the change in MC. At once, three veneer sheets of the same size were removed from the climate chamber and transported to the laboratory. One sheet at a time was removed from the film for weighing and applying adhesive. The glue consumption was 156 g/cm<sup>2</sup>.

Before applying adhesive, veneer sheet needs to be weighed to be able to determine the mass of adhesive applied. As the sheet is completely covered with adhesive, it is re-weighed. If the mass is smaller than necessary, more glue is applied. Otherwise, adhesive needs to be removed to achieve the required mass.

The end-product is a 3-ply plywood panel. In plywood industry, the middle sheet moves through rollers which apply adhesive to both of the surfaces. In this study, adhesive was applied to two sheets to be able to weigh the sheets. Panel size varied from 140x140 mm to 260x260 mm.

Panel laying-up consists of placing one sheet with the loose side glued onto a metal plate covered with foil, then placing the second sheet with one side glued, and lastly, adding the third unglued sheet and a second metal plate on top. A slight pressure was carefully applied by hands to fix the sheets closer together to prevent them moving when inserting the panel between the heated platens of hot press shown in Figure 2.2. Hot press was heated to  $110 \pm 5$  °C as the temperature could not be held under certain value due to the usage of a separate heating element. Pressing time was 3 minutes and pressure load was 1,27 MPa.



Figure 2.2. Hot press

After hot-pressing the panels were cooled down at room temperature and afterwards cut into 120x25 mm specimens for shear strength testing. Every panel was marked with a number to later on know from which panel specimen is cut out. In total 36 panels were made and the panel size varied from 140x140 mm to 260x260 mm.

# 2.3.2 Shear strength

Shear strength of the plywood specimens is measured to determine the bonding quality. Preparation of the specimens (Figure 2.3) was done in accordance with the European Standard EVS-EN 314-1:2005 and testing the shear strength was conducted with Instron 5866.



Figure 2.3. 3-ply plywood shear strength specimen according to EN 314

Before testing, specimens were kept at room temperature for at least 7 days to assure that the highest possible bond strength is achieved. Shear strength specimens are shown in Figure 2.4. As it is stated in the standard, the specimens should be subjected to pre-treatment in the form of immersion for 24 hours in water at the temperature of  $20\pm3$  °C [48]. In this study, the specimens were not pre-treated. Testing samples without soaking them in water can provide information about bonding quality faster and it does not diminish veneer properties, as pre-treatment in the form of soaking causes swelling and softening of the wood [12], [18].



Figure 2.4. Specimens for shear strength testing

Figure 2.5 illustrates Instron 5866 during measuring the shear strength. Specimen is placed between the clamps, fixed firmly and measurement can begin. Pulling speed was set 10 mm/min. Maximum load is given and shear strength can be calculated from it.



Figure 2.5. Instron 5866 for measuring the shear strength

Shear strength of some specimens was measured with Electric Test Stand provided by Sauter. It works by the same principle as Instron and shows also the maximum load applied to the bond before it breaks.

According to the standard, the percentage of wood failure (PWF) is determined. Failure should normally occur in wood or in the glue lines between the saw cuts [48].

The evaluation of PWF is generally subjective as it is done by visual observation and is dependent upon the evaluator. It is difficult to visually measure where the failure has occurred, whether in the adhesive or adhesive-wood interface, or under the wood surface [18].

## 2.4 Test plan

Following table illustrates test plan of this study. It is divided between four activities, pointing out the time consumed and specimens made.

Activity	Date	Duration, h		Specimen size, mm	Number of specimens	Unit
Time dependence of	14.03	3	6	100-100	60	recordings
contact angles	05.04	3	0	100x100	00	
Measuring contact	18.05	1	2	100v100	100	drops
angles	26.05	1	2	1002100	100	
	19.04	2				panels
	21.04	3	- 13		36	
Making alaunaad	02.05	1,5				
	04.05	1,5		various		
	17.05	3				
	18.05	2				
Testing shear strength	26.04	2,5				
	18.05	1,5	7	120x25x4	301	specimens
	26.05	3				

Table 2.2. Test plan

In total, it took an average of 28 hours with the activities to fulfil the aims. The most timeconsuming activity was making plywood as the veneer sheets were conditioned and composed into plywood in different rooms. After applying glue to the sheets and placing them between platens of hot press, next three sheets could be taken out of the climate chamber and transported into the laboratory. This test plan does not include time spent on analysing the results as calculating the time spent is subjective and cannot be measured. Table 2.3 and Table 2.4 show precise test plans for making plywood and measuring the shear strength.

	Making plywood												
Data	Sapwood						False heartwood						Total
Date	4% MC			8% MC		4% MC		8% MC			amount		
	Ach	PKI	Hex	Ach	PKI	Hex	Ach	PKI	Hex	Ach	PKI	Hex	1
19.04				2						4			6
21.04					5						4		9
02.05	2						2						4
04.05		2						2					4
17.05						1						6	7
18.05	1		2				1		2				6
Total	3	2	2	2	5	1	3	2	2	4	4	6	36

Table 2.3. Precise test plan for making plywood specimens

	Testing shear strength												
Data	Sapwood						False heartwood						Total
Date	4% MC			8% MC		4% MC		8% MC			amount		
	Ach	PKI	Hex	Ach	PKI	Hex	Ach	PKI	Hex	Ach	PKI	Hex	
26.04				33	22					31	21		107
18.05		32						32					64
26.05	14		33			18	14		20			31	130
Total	14	32	33	33	22	18	14	32	20	31	21	31	301

Table 2.4. Precise test plan for testing shear strength

## **3** Results and discussion

This chapter provides an overview of the results received from the experimental part. This research was conducted with the aim of apprehending the effect of MC on bonding quality of plywood specimens, especially those made of FHW veneer. Shear strength specimens were examined to evaluate the percentage of apparent cohesive wood failure and the effect of lathe checks direction. In addition, it was expected to discover connections between the effect of MC on CA values and on the bonding strength of plywood specimens. As three different adhesives were used to make plywood, a relation between adhesive properties and bonding strength was hoped to find.

#### **3.1** Contact angle measurements

The results from CA measurements can reveal essential characteristics about wetting properties of the surface.

CAs were measured on SW and FHW. Sheets were conditioned to 4% and 8% MC to examine, how CA values are affected by changes in moisture within the wood. When the MC within wood is below FSP, wood absorbs moisture from the surrounding environment to achieve EMC. Veneer samples with the size of 100x100 mm were cut out from the same sheet and adjacent to each other to ensure that the surface properties are as similar as possible by visual observation.

Table 3.1 shows the average value of 50 drops per sheet received from CA measurements with standard deviation in parenthesis. Ratios marked with yellow show the proportion of 4% samples to 8% within one veneer type and blue ratios show the proportion of SW samples to FHW within certain MC.

Moisture content	Sapwood	False heartwood	Ratio
4%	73,82 (5,02)	65,04 (5,99)	1,13
8%	77,12 (5,64)	77,83 (4,58)	0,99
Ratio	0,96	0,84	

Table 3.1. The effect of moisture content on contact angles

When considering SW, results do not show remarkable difference between 4% and 8% MC. The wettability of both, 4% and 8% MC samples is below  $80^{\circ}$ , i.e. wetting of the surface by the liquid is favourable. The decrease in MC resulted in a slight decrease in CA value, from  $77,12^{\circ}$  to  $73,82^{\circ}$ . Based on these results, the effect of MC on SW veneer is not apparent.

On the other hand, the difference between 4% and 8% MC is evident when concerning FHW veneer. On this basis, the wettability of FHW veneer conditioned to 8% MC is lower than of veneer conditioned to 4% MC. The CA value decreased from 77,83° to 65,04°.

CA results are highly dependent on the surface properties of the substrates. The amount and depth of lathe checks, surface roughness, and surface inactivation by extractives or over-drying are the factors which influence measurements. The first two can be easily detected and samples can be chosen in a way that these factors are minimal. Surface inactivation is a substrate's condition which cannot be detected before testing and analysing the results. One method to detect surface inactivation is by sanding the surfaces.

In both cases the wettability is high and CA values do not show that bonding FHW veneer should be problematic concerning the wettability.

#### **3.1.1** Time dependence of contact angles

In order to analyse the time dependence, a drop needs to be recorded for a certain period of time. In total 60 recordings were made, 30 with SW and 30 with FHW. 100x100 mm veneer sheets were unconditioned, kept at room temperature under same conditions to compare the results of SW and FHW veneer. The MC was determined by oven-dry method. The MC of SW veneer was 6,15% and 6,60% MC with FHW.



Figure 3.1. Contact angle values during the first minute

As the initial CA value on SW differs from the final CA value as much as 28,42°, the difference with FHW is 46,62°. The noticeable difference between the initial CAs can be caused by the higher MC of FHW veneer. Moreover, the surface properties cannot be identical for every piece, leading to altered wettability. Figure 3.1 shows that even if the initial CA of FHW veneer is higher than of SW veneer, it does not have inferior wettability compared to SW. Within the first 30 s water absorbed faster into FHW, after that the absorption rate with FHW and SW was relatively comparable. In conclusion, it can be stated that there does not seem an evident problem with wettability of FHW.

#### 3.2 Shear strength

Shear strength was measured to describe the bonding quality of plywood specimens. Specimens were made out of plywood panels which, in turn, were made from veneer sheets conditioned to 4% and 8% MC to evaluate the effect of MC on bond strength. Furthermore, three different UF adhesives were used to make the panels in order to compare the results and find out which one provides superior results. This study used shear strength specimens without being pre-treated via soaking.



Results are shown in Figure 3.2.

Figure 3.2. The effect of moisture content on shear strength

The increase in MC increases the shear strength, excluding with Achema FHW specimens. With Achema 8% MC FHW specimens the change from 4% to 8% MC resulted in strength loss by 21,38%, but showed slightly increased strength with SW specimens by 4%. The results with Hexion were expected to be higher. The reason for such low results can be due to faulty mixing of the adhesive as the mixture was not stirred sufficiently. But the effect of MC can be considered as the ratio between MC values does not depend on the strength values. With SW specimens, the strength increased by 11,21% and with FHW 21,01%. The change from 4% to 8% MC was the greatest with PKI adhesive, 21,50% with SW and 37,13% with FHW.

From CA values the surface energy of a solid surface can be measured. If liquid forms low CA when in contact with solid surface, the surface energy is high as the work per area for creating a new surface between solid and water is greater. Otherwise, with high CA values the surface energy of a solid is low. Based on previous studies and current results it can be concluded that the surface energy of SW is higher.

Based on studies it is stated that water may affect penetration in two ways. Firstly, wood with low MC will absorb water and resin. The other manner suggests that as wood has low MC, water is absorbed by cell walls, the effective dry matter of adhesive is increased leading to accelerated coalescence and higher viscosity. It has been noted that with increasing the MC, the penetration of adhesive is greater [42]. At 4% MC wood absorbs water within but adhesive with higher viscosity does not penetrate into wood as much as needed. The viscosity of Achema is the lowest compared to Hexion and PKI. CA measurements showed that the CA value decreased with the increase of MC with Achema 8% MC FHW which refers that the wettability at 4% MC is higher. This shows that the different result with Achema FHW specimens can be related with the viscosity of the adhesive. When bonding with PKI and Hexion adhesives, the results are higher at 8% MC, and when using Achema adhesive, greater shear strength occurs with specimens bonded at 4% MC.

As the final shear strength measurements were done with Electric Test Stand by Sauter which works by the same principle as Instron 5866, the change of machinery was not believed to disturb the results. The pulling speed was in both cases 10 mm/min. Following samples were tested with Sauter: Hexion 4% and 8% MC SW and FHW, and Achema 4% MC SW and FHW samples.

Specimens were investigated if the bond failed from wood or from adhesive in order to clarify the results. In a 3-ply plywood, two joints are formed, one between the loose sides and the other between a loose and a tight side of the veneers (see Figure 3.3). Whether the loose-loose (L-L) bond or loose-tight (L-T) bond was tested, it could be discoverable only if the depth of lathe checks (LC) was deep enough as shown in Figure 3.3. Specimens where the LC were shallow, the bond construction (L-L or L-T) could not be detected.



Figure 3.3. Cut-out section of plywood's construction

Moreover, the direction of LC when pulled has been found to be another factor which influences the strength results. If the direction of LC in the surface where shear occurs coincides with the direction of force (F) applied, LC are pulled open (see Figure 3.4). Otherwise, LC are pulled closed, and which is more preferable as it increases the strength [18].



Figure 3.4. Interpretation of the direction of lathe checks: a) checks pulled open, b) checks pulled closed

To clarify the results, results of five panels where the presence and direction of LC could be detected are shown below in Figure 3.5.



Figure 3.5. The effect of lathe checks on shear strength

It is noticeable that the shear strength is remarkably higher when the lathe checks are pulled closed and lower as the checks are pulled open. Values of shallow LC/smooth demonstrate the results of specimens where LC depth was minimal or shear occurred on the smooth side of the veneer.

#### 3.3 Percentage of apparent cohesive wood failure

Percentage of wood failure (PWF) is determined by visual observation of shear strength specimens. In the European standard EVS-EN 314-1:2005 the pre-treatment of specimens is required and failure is expected to occur within wood. When the failure occurs from the wood, the adhesive-wood bond strength exceeds the strength of wood. On this basis, the highest shear strength results should be received from those specimens showing the highest PWF.

PWF of Achema and PKI specimens is examined, as the failure with Hexion specimens occurred mainly from the adhesive.

Figure 3.6 shows the average PWF of Achema and PKI specimens considering the MC. Results show that PWF of FHW veneer was the highest with PKI 8% MC specimens reaching up to 94%, and with SW veneer highest PWF was 51% with Achema 8% MC specimens.



Figure 3.6. The effect of moisture content on percentage of wood failure

Based on these results it can be stated that the highest shear strength values of SW should have been received from Achema 8% specimens and considering FHW veneer from PKI 8% specimens. In case of Achema, the amount of failure from adhesive was higher, and adhesive failure was not taken into account when calculating PWF. The effect of MC is the same as it was on shear strength.



The influence of LC direction on PWF was also investigated. This analysis is based on PKI specimens where the direction was detectable. Results are illustrated in Figure 3.7.

Figure 3.7. The effect of lathe checks on percentage of wood failure

Similar results were illustrated in Figure 3.5 where the effect of LC direction on shear strength was examined. The highest PWF occurs when LC are pulled closed and the lowest when pulled open.

The standard deviation is high in both Figure 3.6 and Figure 3.7. The highest PWF values did not always occurred with specimens with LC pulled closed.

# Conclusions

Plywood industry is facing many determinative factors within the production of birch plywood, namely the presence of false heartwood (FHW). The backgrounds of FHW formation and its effect on veneer properties are relatively unclear.

Understanding the behaviour of wood under certain conditions is the foremost requirement when manufacturing high-quality wood-based products. The properties of FHW veneer are insufficiently studied, especially those related to bonding. Bond formation and its performance in service are the most relevant indicators in plywood production and are dependent on following properties of veneer: wettability, surface quality and moisture content (MC). The effect of MC on wettability was examined by contact angle (CA) measurements.

The effect of MC on bonding strength of birch sapwood (SW) and false heartwood was examined by measuring the shear strength of plywood specimens made with three different industrial urea-formaldehyde adhesives: KF-FE from AB "Achema", UF 234 from Hexion Inc. and Profect® 2702 from PKI Supply A/S. Specimens were conditioned to 4% and 8% MC.

Conclusions based on the results are following:

- CA measurements showed that the wettability of FHW veneer increases remarkably as SW veneer showed only a slight increase when decreasing the MC from 8% to 4%. Based on the results, the wetting of FHW and SW veneer is favourable at both MC values.
- The effect of MC on shear strength was evident with all of the specimens tested. In most of the cases as the MC increased the shear strength also indicated higher results, excluding with Achema FHW specimens.
- Based on shear strength measurements it can be concluded that *the effect of MC on* bonding strength of FHW plywood bonded with adhesive with lower viscosity shows higher results with 4% MC. Furthermore, adhesives with higher viscosity form stronger bonds with FHW at 8% MC.

• In addition, the effect of LC was examined. As the LC are pulled closed, the shear strength value is higher than when pulled open. The direction of LC affects the results only when the depth is greater. The depth of LC was evaluated visually. With specimens with smooth surface or shallow checking the effect of LC is not considered.

The aim of this thesis was fulfilled. The conclusions received from this study can be used in plywood manufacturing. Depending on the target MC value of veneer sheet either low or high viscosity adhesives should be used when bonding FHW veneer.

For further studies, it is proposed to examine the effect of 12% MC on bonding strength. Shear strength measurements with Hexion UF 234 adhesive should be repeated. When testing shear strength, the direction of LC should be considered before making the panels to prevent excessive work.

# References

- [1] "Eesti statistika aastaraamat," 2015. [Online]. Available: http://www.stat.ee/valjaanne-2016\_eesti-statistika-aastaraamat-2016.
- [2] P. Savill, *The silviculture of trees used in British forestry*, no. Ed.2. 2013.
- [3] "Silver Birch | The Wood Database Lumber Identification (Hardwood)." [Online]. Available: http://www.wood-database.com/silver-birch/. [Accessed: 03-Jun-2017].
- [4] "Downy Birch | The Wood Database Lumber Identification (Hardwood)." [Online]. Available: http://www.wood-database.com/downy-birch/. [Accessed: 04-Jun-2017].
- [5] J. Hynynen, P. Niemisto, A. Vihera-Aarnio, A. Brunner, S. Hein, and P. Velling, "Silviculture of birch (Betula pendula Roth and Betula pubescens Ehrh.) in northern Europe," *Forestry*, vol. 83, no. 1, pp. 103–119, Jan. 2010.
- [6] E. Saarman, *Puiduteadus*, 2nd ed. Tartu: Vali Press, 2006.
- [7] N. Gibbs, *Puiduliikide piibel*. Tallinn: Sinisukk, 2008.
- [8] M. Prka, A. P. B. Krpan, D. Vusić, and Ž. Zečić, "Characteristics and Share of European Beech False Heartwood in Felling Sites of Central Croatia," *Izvorni Znan. rad*, vol. 30, no. 1, pp. 37– 49, 2009.
- [9] B. Belleville, A. Cloutier, and A. Achim, "Detection of red heartwood in paper birch (*Betula papyrifera*) using external stem characteristics," *Can. J. For. Res.*, vol. 41, no. 7, pp. 1491–1499, 2011.
- [10] A. L. Shigo and W. E. Hillis, "Heartwood, Discolored Wood, and Microorganisms in Living Trees," 1973.
- [11] J. Sorz and P. Hietz, "Is oxygen involved in beech (Fagus sylvatica) red heartwood formation?," *Trees Struct. Funct.*, vol. 22, no. 2, pp. 175–185, 2008.
- [12] A. Yamamoto, "Chemical characteristics of surface colour of birch veneer (Betula pendula)," Aalto University, 2015.
- [13] T. Knoke, "Predicting red heartwood formation in beech trees (Fagus sylvatica L.)," Ecol. Modell., vol. 169, no. 2–3, pp. 295–312, 2003.
- [14] S.-O. Lundqvist, T. Grahn, and L. Olsson, "Properties of materials from Birch Variations and relationships Part 1: Growth, wood density and biomass," 2013.
- [15] G. Giroud, A. Cloutier, and J. Alteyrac, "Occurrence, proportion, and vertical distribution of red heartwood in paper birch," *Can. J. For. Res.*, vol. 38, no. 7, pp. 1996–2002, 2008.
- [16] R. Shmulsky and P. D. Jones, *Forest Products and Wood Science An Introduction*. Oxford, UK: Wiley-Blackwell, 2011.
- [17] M. L. Selbo, "ADHESIVE BONDING OF WOOD," 1975. [Online]. Available: http://www.woodcenter.org/docs/tb1512.pdf. [Accessed: 03-Jun-2017].
- [18] A. Rohumaa, "The impact of log pre- heating on birch veneer surface quality, bond formation

and plywood performance," Aalto University, 2016.

- [19] C. Demirkir, Ş. Özsahin, I. Aydin, and G. Colakoglu, "Optimization of some panel manufacturing parameters for the best bonding strength of plywood," *Int. J. Adhes. Adhes.*, vol. 46, pp. 14–20, 2013.
- [20] W. T. Simpson, "Drying and control of moisture content and dimensional changes," in *Wood Handbook: Wood as an Engineering material*, Madison: Forest Products Laboratory, 2010, p. 13.1-13.19.
- [21] I. Aydin, G. Colakoglu, S. Colak, and C. Demirkir, "Effects of moisture content on formaldehyde emission and mechanical properties of plywood," *Build. Environ.*, vol. 41, no. 10, pp. 1311–1316, 2006.
- [22] A. Schramm, "Lathe Checks in Rotary Peeled Veneer," *Columbia Forest Products*, 2009.
  [Online]. Available: http://archive.constantcontact.com/fs058/1102117931485/archive/1102890340140.html.
   [Accessed: 03-Jun-2017].
- [23] C. R. Frihart and C. G. Hunt, "Chapter 10 Adhesives with Wood Materials Bond Formation and Performance," *Wood Handb. Wood as an Eng. Mater.*, pp. 1–24, 2010.
- [24] A. Aguilera and J. P. Davim, *Research developments in wood engineering and technology*. IGI Global, 2013.
- [25] I. Aydin, "Activation of wood surfaces for glue bonds by mechanical pre-treatment and its effects on some properties of veneer surfaces and plywood panels," *Appl. Surf. Sci.*, vol. 233, no. 1–4, pp. 268–274, 2004.
- [26] D. Devallance and J. Funck, "Influence of several preparation conditions on Douglas-fir plywood gluebond quality test results," *J E For. Prod. J. Feb*, vol. 56, no. 2, 2006.
- [27] A. K. Kulshreshtha and C. Vasile, *Handbook of polymer blends and composites*. Rapra Technology, 2002.
- [28] I. Aydin and G. Colakoglu, "Effects of surface inactivation, high temperature drying and preservative treatment on surface roughness and colour of alder and beech wood," *Appl. Surf. Sci.*, vol. 252, no. 2, pp. 430–440, 2005.
- [29] "Polymers and Surface Coating: Adhesives," *Chemical Processes in New Zealand*, 1998.
  [Online]. Available: https://nzic.org.nz/ChemProcesses/chem\_processes.html. [Accessed: 03-Jun-2017].
- [30] Y. Yuan and T. R. Lee, "Contact Angle and Wetting Properties," *Surf. Sci. Tech.*, vol. 51, pp. 3–34, 2013.
- [31] M. Walinder, "Wettability Phenomena on Wood," Dr. Thesis, Dep. Manuf. Syst. Wood Technol. Process. KTH-Royal Inst. Technol. Sweden., no. February, p. 61, 2000.
- [32] D. B. DeVallance, J. W. Funck, and J. E. Reeb, "Douglas-fir plywood gluebond quality as influenced by veneer roughness, lathe checks, and annual ring characteristics.," J. For. Prod., vol. 57, no. 1/2, p. 21, 2007.
- [33] S. Ebnesajjad, Handbook of adhesives and surface preparation : technology, applications and manufacturing, 1st ed. William Andrew/Elsevier, 2011.

- [34] C. Demirkir, I. Aydin, S. Çolak, and G. Çolakoğlu, "Effects of plasma treatment and sanding process on surface roughness of wood veneers," *Turkish J. Agric. For.*, vol. 38, no. 5, pp. 663– 667, 2014.
- [35] J. Hartley and J. Marchant, "Methods of determining the moisture content of wood," *Res. Div.*, pp. 1–61, 1995.
- [36] L. Blomqvist, D. Sandberg, and J. Johansson, "Influence of veneer orientation on shape stability of plane laminated veneer products," *Wood Mater. Sci. Eng.*, vol. 9, no. 4, pp. 224–232, 2014.
- [37] S. V. Glass and S. L. Zelinka, "Moisture Relations and Physical Properties of Wood," in *Wood Handbook-Wood as an Engineering Material*, Wisconsin: Forest Products Laboratory, 2010, p. 4.1-4.19.
- [38] M. S. Moghaddam, "Wettability of modified wood," KTH Royal Institute of Technology, 2015.
- [39] M. Sernek, J. Resnik, and F. A. Kamke, "Penetration of liquid urea-formaldehyde adhesive into beech wood," *Wood Fiber Sci.*, vol. 31, no. 1, pp. 41–48, 1999.
- [40] C. R. Frihart, "Adhesive Groups and How They Relate to the Durability of Bonded Wood," *J. Adhes. Sci. Technol.*, vol. 23, no. 4, pp. 601–617, 2009.
- [41] C. R. Frihart, "Adhesive interaction with wood," *Fundam. Compos. Process. Proc. a Work.*, pp. 29–54, 2004.
- [42] F. A. Kamke and J. N. Lee, "ADHESIVE PENETRATION IN WOOD—A REVIEW," Oregon State University, 2005.
- [43] C. Schaschke, A dictionary of chemical engineering. Oxford University Press, 2014.
- [44] "Contact angle measurement in practice (1)," *Krüss GmbH*, 2007. [Online]. Available: https://cmi.epfl.ch/metrology/files/KrussDSA30/kruss-tn311-315-en.pdf. [Accessed: 03-Jun-2017].
- [45] R. P. Woodward, "Contact Angle Measurements Using the Drop Shape Method," *First Ten Angstroms*. [Online]. Available: http://www.firsttenangstroms.com/pdfdocs/CAPaper.pdf. [Accessed: 03-Jun-2017].
- [46] "EVS-EN 828:2013. Liimid. Märgavus. Tahke aluspinna kontaktnurga mõõtmine ja kriitilise pindpinevuse määramine." Eesti Standardikeskus.
- [47] T. Antikainen, A. Rohumaa, C. G. Hunt, M. Levirinne, and M. Hughes, "Estimating the spread rate of urea formaldehyde adhesive on birch (Betula pendula Roth) veneer using fluorescence," *Eur. J. Wood Wood Prod.*, vol. 73, no. 1, pp. 69–75, 2014.
- [48] "EVS-EN 314-1:2005. Vineer. Liimühenduse kvaliteet. Osa 1: Katsemeetodid." Eesti Standardikeskus.

## Abstract

As the quality of plywood is in a relation with the quality of veneer, it is requisite that veneer properties are superior. Birch species do not form a visibly darker heartwood making it suitable for the production of veneer except for the possibility of false heartwood (FHW) formation which decreases the yield and quality of birch veneer.

The aim of this study was to evaluate how bonding strength of FHW veneer is affected by the moisture content (MC). Based on literature, two MC values were chosen: 4% and 8%. Moreover, three industrial urea-formaldehyde adhesives were used to make plywood to compare the results and determine whether adhesive properties have also influence on bonding strength. Following adhesives were used: KF-FE from AB "Achema", UF 234 from Hexion Inc. and Profect® 2702 from PKI Supply A/S.

Wettability of sapwood (SW) and FHW was examined with contact angle (CA) measurements. Results showed that with 8% MC specimens the CA values with both veneers were similar and below 80° meaning that wetting is favourable. With 4% MC, the difference between SW and FHW veneer was 12,8°. The effect of MC with SW was slight, as the average CA values increased only by 4%, as the increase with FHW veneer was 16%. Time-dependence of CA values of unconditioned veneers were also examined. Results of FHW show that the difference between initial and final CA is greater than with SW veneer during the first minute.

Shear strength measurements were done in order to determine the effect of MC on bonding strength. Based on the results it can be concluded that the effect of MC on bonding strength of FHW plywood bonded with adhesive with lower viscosity shows higher results with 4% MC. Furthermore, adhesives with higher viscosity form stronger bonds with FHW at 8% MC. As adhesive includes water as a carrier and has low viscosity, at 4% MC the penetration of adhesive is high. At 8% MC, high flow and overpenetration of resin into wood can be the cause of lower strength results. Adhesives with higher viscosity show greater penetration of adhesive with higher MC values. Highest percentage of apparent cohesive wood failure (PWF) was measured from PKI 8% MC FHW specimens which also showed the highest shear strength. Likewise, the LC direction showed that when LC pulled open the PWF was lower than when pulled closed.

Keywords: False heartwood, birch, veneer, plywood, lathe checks, shear strength, adhesion, contact angle measurements

## Resümee

Vineeri kvaliteet on suures ulatuses seotud spooni kvaliteediga, mistõttu on spoonile esitatavad kvaliteedinõuded väga piiritletud. Eestis vineeritööstuses laialdaselt kasutatud arukask (*Betula pendula*) ja sookask (*Betula pubescens*) on heledavärvilised maltspuidulised liigid ehk nähtavalt tumedamat lülipuitu kasel ei kujune. Kuid kase puidule iseloomulikult võib tekkida lülipuidule sarnane värvuse muutus, mida tuntakse väärlülipuidu (VLP) nime all ja mis langetab kasespooni saagikust ja kvaliteeti.

Antud töö eesmärgiks oli hinnata, kuidas mõjutab niiskussisaldus kase VLP spooni liimliite tugevust. Uuritavateks niiskussisaldusteks valiti 4% ja 8%. Valiti välja kolm erinevat tööstuslikult toodetavat karbamiidliimi firmadelt AB "Achema", PKI Supply A/S ja Hexion Inc., mida kasutati kase maltspuidust (MP) ja VLP katsekehade valmistamisel. Nihkekatsete tulemuste põhjal selgitatakse välja, millised liimiomadused võivad mõjutada kase VLP spooni omadusi liimliite tugevuse kujunemisel.

Kase MP ja VLP märgumisomadusi mõlematel niiskussisaldustel hinnati kontaktnurga (KN) mõõtmisega. Tulemused näitasid, et VLP spooni korral niiskussisalduse vähenedes vähenes KN ligikaudu 13%, MP spooni korral ainult 4%. Mõlemal juhul näitas KN väärtus, et pinna märgumine vedeliku poolt on soodustatud. KN sõltuvust ajast hinnati konditsioneerimata MP and VLP spooni peal, et võrrelda, kuidas erinevad MP ja VLP omadused olles hoiustatud samadel tingimustel. Spoonide niiskussisalduse mõõtmisel selgus, et VLP niiskussisaldus oli suurem kui MP spoonil, mis põhjustas ka kõrgemat esialgset KN. Tulemused näitasid VLP spooni korral kiiremat vedeliku sisse imbumist.

Niiskussisalduse mõju hindamiseks teostati nihketugevuse katsed. Tulemuste põhjal võib järeldada, et spooni niiskusel ja liimi viskoossusel on oluline mõju kase VLP liimliite tugevusele. Madala viskoossusega liimide puhul saavutati suuremad nihketugevused katsekehadega, mille niiskussisaldus oli 4%. Kõrgema viskoossusega liimid omakorda näitasid paremaid tulemusi niiskussisaldusel 8%. Nihketugevuse katsekehadel hinnati puidust purunemise protsentuaalset väärtust. Kõrgeima puidust purunemise väärtuse andsid VLP katsekehad, mis valmistati PKI Profect® 2702 liimiga ning millega saavutati ka suurimad nihketugevused. Katsekehadel hinnati ka treimisel tekkivaid lõhesid, mille suund mõjutab märgatavalt nihketugevuse suurust. Kui nihketugevuse mõõtmisel tõmmati lõhed lahti, siis tugevuse väärtus oli madalam, kui tõmmates lõhesid kinni.