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The influence of log soaking temperature, veneer moisture content, hot-pressing temperature, and hot-pressing time on the self-bonding ability of rotary-cut birch veneer

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Abstract

Self-bonding of wood refers to a technique where pieces of wood are bonded by activating wood constituents with heat and pressure without any added adhesive, and it is enabled by chemical and physical changes within wood. The aim of this thesis was to study the self-bonding ability of birch (*Betula pendula*) veneer with Automated Bonding Evaluation System (ABES). The effect of moisture content of veneer and log soaking temperature on tensile shear strength of bond line was examined with different hot-pressing parameters, and also the effect of humidity on bonded veneers was assessed. Furthermore, the results from ABES tests were scaled up to a laboratory level plywood testing.

The results showed that log soaking temperature and moisture content of veneer significantly influenced the self-bonding ability of birch veneer. Especially, log soaking temperature of 20 °C evidently contributed to bond formation compared to soaking at 70 °C. Moreover, joint effect of lower log soaking temperature and approximately 6 % moisture content provided the greatest shear strength values. Self-bonded birch veneer joint also resisted moisture moderately when relative humidity is elevated from 35 % to 65 %, although signs of weakening of the bond was noted.

Based on the results in this thesis the effect of hot-pressing temperature and hot-pressing time on self-bonding was substantial. Formation of a proper bond required at least 220 °C of hot-pressing temperature. Alternatively, higher pressing temperature enabled the use of a shorter hot-pressing time. Although the results gained with ABES considering pressing parameters appears to be unambiguous, the application of the results to plywood manufacture requires further research.

Keywords self-bonding, birch, veneer, moisture content, log soaking temperature, ABES

Tekijä Samuli Kimpimäki

Työn nimi Tukin haudontalämpötilan, viilun kosteuspitoisuuden ja kuumapuristuslämpötilan ja -ajan vaikutus sorvatun koivuviilun itseliimautuvuuteen

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Tiivistelmä

Puun itseliimautumisella tarkoitetaan menetelmää, jossa puukappaleet liitetään toisiinsa aktivoimalla puun ainesosia lämmön ja paineen avulla ilman lisättyä liimaa. Itseliimautumisen mahdollistaa kemialliset ja fysikaaliset muutokset puuaineessa. Tämän diplomityön tarkoitus oli tutkia koivuviilun (*Betula pendula*) itseliimautumisominaisuuksia Automated Bonding Evaluation System (ABES) –laitteen avulla. Viilun kosteuspitoisuuden ja tukin haudontalämpötilan vaikutusta sauman vetoleikkauslujuuteen tutkittiin eri kuumapuristusparametreilla, ja myös myöhemmän tasaannutuskosteuden vaikutus muodostuneen sauman lujuuteen arvioitiin. Lisäksi ABES-koestuksen tulosten perusteella mittakaavaa kasvatettiin laboratoriotason vanerin testaukseen.

Tulokset osoittivat, että tukin haudontalämpötila ja viilun kosteuspitoisuus vaikuttavat tilastollisesti merkitsevästi koivuviilun itseliimautuvuuteen. Erityisesti tukin haudonta 20 °C:ssa edisti sidoksen muodostumista verrattuna haudontaan 70 °C:ssa. Lisäksi alemman haudontalämpötilan ja noin 6 %:n kosteuspitoisuuden yhteisvaikutus tuotti suurimmat vetoleikkauslujuusarvot. Itseliimautuva koivuviilusauma myös säilytti lujuusominaisuutensa kohtuullisesti, kun suhteellinen kosteus nostettiin 35 %:sta 65 %:in, vaikka merkkejä sauman heikkenemisestä havaittiin.

Tämän työn perusteella puristuslämpötilan ja –ajan vaikutus itseliimautuvuuteen osoittautui huomattavan suureksi. Kunnollisen sidoksen muodostumiseen vaadittiin vähintään 220 °C:n lämpötila. Toisaalta korkeampi lämpötila mahdollisti lyhyemmän puristusajan käytön. Vaikka ABES-menetelmällä saadut tulokset koskien puristusparametreja vaikuttavat yksiselitteisiltä, niiden soveltaminen suoraan vanerin valmistukseen vaatii lisätutkimusta.

Avainsanat itseliimautuminen, koivu, viilu, kosteuspitoisuus, tukin haudontalämpötila, ABES

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1 INTRODUCTION

1.1 Background

Humans have joined pieces of wood together since prehistoric times. First wood adhesives were made from birch bark tar and beeswax and tree sap. Other natural binders such as animal glues, egg pastes, and starch from vegetables have also been used throughout the ages (Regert 2004). However the invention of Bakelite and synthetic phenolic based resins in the early 1900s and their further development until the mid-1930s replaced most of the other adhesives and also enabled the process of making durable industrial wood products including plywood (Nicholson et al. 1991, Sellers 1985).

Good bonding quality and especially good resistance to water have made formaldehyde based resins an almost irreplaceable option in industrial applications and they have overtaken nearly all previous adhesives. Nevertheless formaldehyde causes environmental problems and health issues in manufacture and in usage (Malaka & Kodama 1990, Salthammer et al. 2010). Because of this and cost issues there has also been minor effort to develop alternative methods to bond wood.

Wood has the ability to produce its own adhesive. Furthermore, it has been discovered that wood has also the ability to self-bond in certain conditions with or without the aid of reagents. There have been several approaches to join wood without any synthetic adhesives. They can be roughly divided into four categories based on the bond formation: hydrolyzing with steam, gas-tight press with cooling boards under pressure, surface activation with oxidants and acids, and friction welding (Cristescu 2008). Although all the processes have slightly different approaches to the self-bonding phenomenon they all share the presence of heat and pressure.

When wood is heated to over 200 °C several chemical changes take place in the wood structure. Additionally, if two pieces of wood are compressed together at the same time with heat for a sufficiently long time, it has been suggested that the pieces make a relatively strong bond. (Cristescu 2006, Ruponen et al. 2014)

1.2 Objectives and limitations

The aim of this thesis was to study how different hot-pressing parameters and veneer characteristics affect the self-bonding ability of birch veneer. The variable hot-pressing parameters that were chosen for this study were pressing time and hot-pressing temperature. To study how the veneer characteristics influence the self-bonding ability it was decided to vary the log soaking temperature prior to peeling as well as the moisture content of the veneer before and after pressing. All of these characteristics were chosen based on work that has been done earlier and on the trial tests in order to get proper results and information concerning the self-bonding mechanisms. The tests were carried out using the Automated Bonding Evaluation System (ABES). ABES was used to study the bonding of two veneers so as to minimize the other effects that may influence the results in the larger scale manufacture of plywood due to irregularity of the wood material and multiple layers of veneer. Bonding quality was evaluated by testing the tensile shear strength. Furthermore, the results that were gained from ABES were applied to laboratory scale plywood manufacture.

Birch (*Betula pendula*) is the only wood species that was used in this thesis. Pressing pressure is held constant at 5 MPa and pressing time and temperature were limited to (60 – 900) s and (180 – 240) °C respectively. Considering veneer properties, 20 °C and 70 °C log soaking temperature and equilibrium moisture content at 35 % RH and 65 % RH conditions were used. Veneers were laminated in a parallel grain direction and the possibilities in cross lamination are discussed though.

2 LITERATURE REVIEW

2.1 Chemical Changes within Wood Components due to Heat

Wood material consists mainly of cellulose, hemicellulose, lignin, and extractives. They all undergo chemical changes and deterioration when they are exposed to heat. Heat changes the chemical composition of the cell wall and the severity of the changes and deterioration is dependent on the treatment time and temperature. (Esteves & Pereira 2008) Figure 1 shows the chemical changes that occur in wood when it is heated.

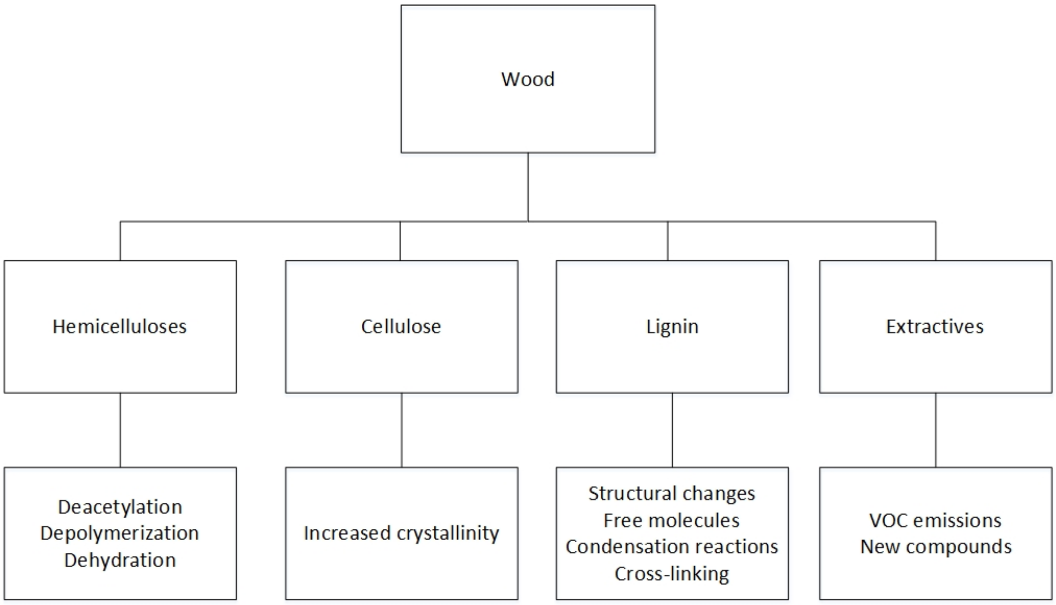


Figure 1. Chemical changes occurring in wood during heat treatment. (Esteves & Pereira 2008)

2.1.1 Hemicelluloses

Hemicelluloses are affected at the lowest temperature of all wood components: minor deterioration of hemicelluloses has been discovered already at 130 °C, yet acceleration of degradation is reported to start below 180 °C (Esteves & Pereira 2008, Tjeerdsma et al. 1998). Tjeerdsma et al. (1998)

suggest that decreasing hemicelluloses catalyzes carbohydrate cleavage, which leads to deacetylation and the formation of acetic acid and eventually the formation of formaldehyde, furfuraldehyde and other aldehydes. This deacetylation reaction also acts as a catalyst to further polysaccharide decomposition (Tjeerdsma et al. 1998, Sivonen et al. 2002). Furthermore, there is a relation between the higher acetic and formic acid content and higher mass loss as well as lower mechanical properties, lower lightness and lower hue of heat treated wood (Sundqvist et al. 2006). Dehydration of hemicelluloses coincides with deacetylation (Weiland & Guyonnet 2003). Also the quantity of hemicelluloses affect the rate of degradation caused by heat. It has been noted that the species that contains more hemicelluloses are more affected by heat treatment (Kocaefe et al. 2008, Weiland & Guyonnet 2003, Zaman et al. 2000). In conclusion, the heating of hemicelluloses acts as a catalyst for self-bonding possibly because of the deacetylation that decomposes the other components of wood. Decomposition of hemicelluloses creates new extractives that may also aid in the self-bonding process.

Figure 2 presents a possible self-bonding mechanism where thermal degradation of hemicelluloses release sugars, which are further hydrolyzed into furfurals. Subsequent to hydrolysis a possible condensation reaction with lignin that forms the adhesive occurs. (Zhang et al. 2015)

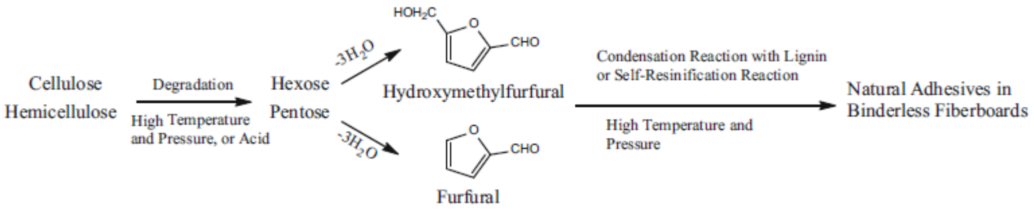


Figure 2. Possible self-bonding mechanism of binderless fiberboard. (Zhang et al. 2015)

2.1.2 Cellulose

Cellulose is the most stable component of wood. Cellulose has the ability to make intra- and intermolecular bonds, which enables the strong partially crystalline structure (Klemm et al. 2005). Furthermore, the structure of cellulose starts to change at a temperature near to 230 °C when a decrease in amorphous cellulose and an increase in crystallinity has been discovered. The decrease of amorphous cellulose is mainly caused by liberation of acetic acid from hemicelluloses. (Sivonen et al. 2002)

Higher moisture content increases crystallinity in cellulose compared to oven dry wood. Highly moist wood is reported to produce nearly twice as much crystallization in cellulose than in dry wood. The reason for this may be the lower stresses in wood components because of the moist conditions, which allows the molecules in cellulose to rearrange freely. Additionally, cellulose in wood crystallizes more than pure cellulose, which likely refers to the fact that the degradation of other components in wood accelerates the crystallization in cellulose. (Bhuyian et al. 2000)

2.1.3 Lignin

The proportion of lignin rises at the same rate as the proportion of hemicelluloses decreases when the wood is heated (Zaman et al. 2000). However, certain parts of lignin start also degrade changing the ratio of different constituents in lignin. (Windeisen 2007).

The occurrence of cleavage in lignin leads to autocondensation, and the cleaved lignin starts to form methylene bridges. This consequently leads to greater hygroscopicity and dimensional stability because of the increased cross-linking between lignin and carbohydrates. Increased cross-linking reduces the possibility for cellulose microfibrils to expand; hence the capacity of cellulose chains to adsorb water reduces. The impact of heat on lignin also increases the amount of natural phenolic resin in wood, which increases the water repellency of wood. (Tjeerdsma et al. 1998)

During the hot pressing of veneers lignin is reported to move closer to bond lines. Veneers pressed at 225 °C have been noted to have more occurrences on surface of lignin containing units than veneers that are not hot pressed. Furthermore, there are signs of similar migration of monosaccharides generated near surfaces. (Cristescu & Karlsson 2013)

2.1.4 Extractives

Two types of extractives occur during the heating of wood: native extractives and generated extractives. Native extractives denote the extractives that are naturally discovered in wood prior to thermal modification. Whereas, thermal modification generates new degradation products to replace native extractives. (Cristescu 2015) According to Esteves & Pereira (2008) most of the native extractives vaporize relatively quickly during heating, leaving the degradation products of the wood cell wall components to operating as new extractives.

Wood material includes a large variety of extractives. Extractives can be divided into two groups based on their solubility: hydrophilic and oleophilic components. These two types of extractives also have a different effect on the self-bonding ability of wood. Hydrophilic extractives increase the reactivity on the wood surface and improve the mechanical properties of self-bonded wood, whereas oleophilic compounds have a contrary effect on the bonding properties by decreasing the reactivity. (Alvarez et al. 2015)

As previously mentioned, the degradation of hemicelluloses and lignin generates new extractives during heating. Hakkou et al. (2006) heat treated beech at different temperatures and reported that the extractive content starts to increase after reaching 160 °C, which is also at same range where the degradation of hemicelluloses begins. The maximum extractive content is reached at 240 °C (Hakkou et al. 2006).

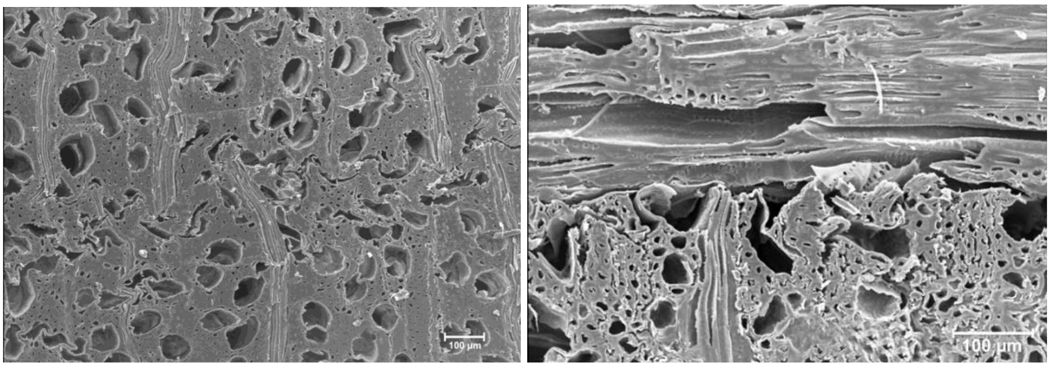
Cristescu & Karlsson (2013) noted that monosaccharides such as glucose and fructose start to migrate near the surface of the veneer during hot pressing. This also results in increased water-soluble phenols, furfural, and 5-(hydroxymethyl)furfural (HMF) content especially near bond lines. However the HMF content is highest at 200 °C and starts to decrease at higher temperatures. This may due to the formation of new reaction products or evaporation of HMF. Nevertheless, there are also signs of water repellent phenolic structures, although the occurrence of such structures is not confirmed.

2.2 Microstructure of Self-bonded Wood

Wood is a porous material. Especially hardwoods contain vessels to carry water and nutrients inside wood material. Vessels of birch are diffused over the cross-section evenly. (Harada & Côté 1985) The walls of vessels are relatively thin and they are not strongly affected by heat at least at temperatures near 180 °C (Biziks et al. 2013). Navi & Girarded (2000) have compared the effects of thermo-mechanical compression and thermo-hydro-mechanical (THM) compression of beech, which has microstructure very similar to birch. The self-bonding process also involves mechanical compression combined with high pressing temperature. During thermal compression the walls of vessels collapse but the lumens stay open. This enables water to fill the cells easily. THM compression where the wood is saturated with hot steam while it is compressed closes the lumens and makes wood more hygroscopic. Wood swelling when the compressed sample is soaked in water has been reduced to near 10 % with this treatment (Navi & Girarded 2000).

The tensile strength of wood rays (radial parenchyma cells) can be three times higher than the tensile strength of wood itself (Burgert & Eckstein 2001). Rays are aligned perpendicularly to other wood fibers and their function in the living tree is to supply nutrients between pith and bark (Harada & Côté 1985). It has been suggested that rays, because of their direction and high strength, could be able to transport vapors and gases that are formed during hot pressing away from the surface (Cristescu 2015).

Figure 3 shows scanning electron microscopy (SEM) images of self-bonded beech veneers with parallel and perpendicular lamination. In parallel orientation, the bond line of the veneers is very irregular and there is lots of entanglement and surface where the veneers are in connection. Moreover, especially near bond line the vessels are almost completely collapsed, and rays are strongly penetrated into weaker wood material. Perpendicularly oriented veneers show no entanglement while the stronger rays block the possibility to penetrate deeper into wood. Also the collapse of vessels near bond line seems to be at a lower level in the case of perpendicular fiber orientation.



a) b)

Figure 3. Cross-sectional scanning electron microscopy view of boards with a) parallel and b) perpendicular fiber orientation of bonded veneers. 200x magnification. (Cristescu 2015)

2.3 Wood Softening

Thermal softening of amorphous polymers begins when the temperature of the material reaches its glass-transition temperature. Above the glass-transition temperature normally stiff and glassy polymers start to lose their stiffness and become more rubbery and elastic. It has been suggested that thermal expansion increases the amount of free volume, which leads to easier movement of molecules and chains eventually causing the stiffness to drop significantly. (Morsing 2000)

The crystallinity of cellulose and strong secondary forces between the molecules restricts the softening of cellulose (Morsing 2000). Furthermore, Morsing (2000) states that cellulose begins to degrade prior to the glass-transition temperature of crystalline cellulose is reached.

Lignin has a complex structure and also lots of cross-linking occurs, which are both preventive factors in terms of softening. However, lignin is considered to be the influential component considering softening of wood because of its cementing function in the cell wall. The highly amorphous structure with a high concentration of hydroxyl groups and little possibility for cross-linking makes the hemicellulose a relatively easily softened component in wood. (Morsing 2000) Figure 4 illustrates the softening temperature of cellulose, hemicelluloses, and lignin as a function of moisture content.

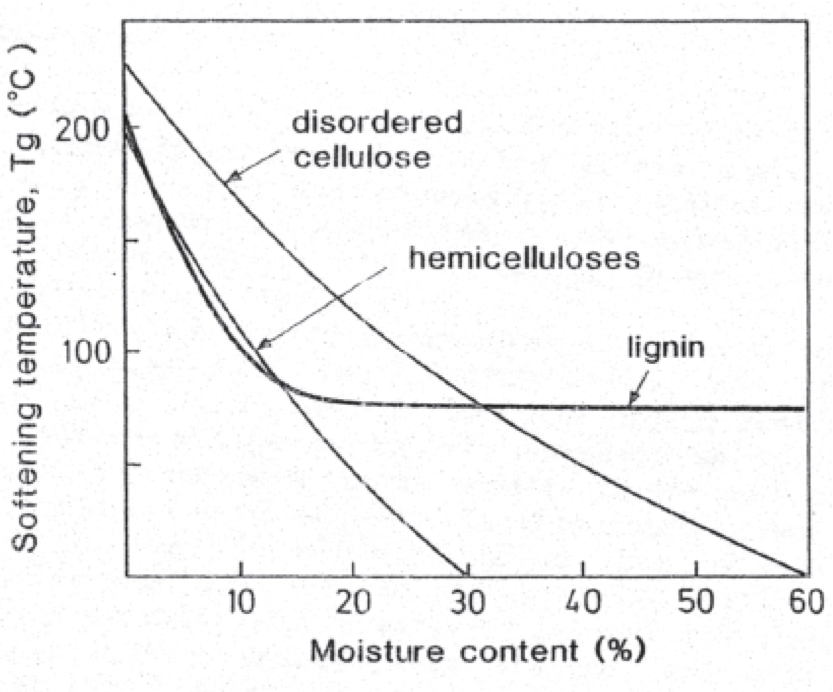


Figure 4. Softening temperature of amorphous cellulose, hemicellulose, and lignin as a function of moisture content (Salmén 1990).

The softening properties of dry wood and wet wood differ. Softening of wood depends on temperature and moisture content and moisture acts as a plasticizer in wood (Morsing 2000, Salmén 1990). With dry wood the glass transition temperature of lignin is approximately 200 °C. Therefore, the pressing temperature should be at least 200 °C in order to gain at least minor changes in terms of the cell wall plasticization of dry wood. However, proper softening of the cell wall requires a temperature of approximately 380 °C, which is the point where heat starts to affect crystalline cellulose. (Shiraishi 1991)

If wood material is wet there is more space for polymers to move. This enables reduced interaction between cellulose and lignin. The glass-transition temperature of lignin has been noted to govern the softening temperature of the wood material. (Morsing 2000) A relation with degradation of β-O-4 bonds in lignin and the loss of rigidity of wood is significant, also the proportion of β-O-4 bonds is higher in hardwood lignin than in the lignin of softwood (Assor et al. 2009). Therefore softening of lignin is stated to be the most important factor in terms of softening of whole wood. In order to obtain good quality veneer by peeling, the wood material of logs needs to be softened. Usually the softening is performed by soaking the logs in ambient temperature or in heated water (Dupleix et al. 2013).

2.4 Physical Properties of Self-bonded Wood

2.4.1 Density

Density has the greatest effect on the strength properties of wood. Generally higher density induces higher strength properties. (Kärkkäinen 2003) During compression the density of wood increases. Compression causes bending of the cell walls and the deformation is dependent on the softening properties of the cell wall components. Especially the condition of lignin is stated to be a critical factor considering the elastic properties of wood. (Morsing 2000) However, the spring back phenomenon of densified wood is a major issue especially if wood is exposed to moisture or water. The spring back reaction of mechanically densified wood caused by moisture can be reduced for example with heat treatment or steam (Inoue et al. 1993, Navi & Girardet 2000).

The self-bonding process of veneers that includes relatively high pressure and temperature induces densification in wood. A self-bonded board that is pressed with the most severe conditions in terms of temperature and pressure has reported to be 67 % denser than the veneers of the board prior to hot-pressing. There is also a relation between high density and low water absorption, low thickness swelling, high shear strength, and high bending strength. (Cristescu et al. 2015b) Considering the process parameters, temperature affects density more than pressing pressure. As the pressing time increases with higher pressure and temperature there are signs of a decrease in density (Cristescu 2015). This loss in density is presumably caused by thermal degradation.

The distribution of density is not equal. The highest density after self-bonding of veneers is close to bond lines and the surface of the board (Cristescu 2015). Moreover, densification of veneer is reported to influence the properties of the surface, in particular, it decreases surface roughness (Bekhta et al. 2014, Fang et al. 2012). However, if the board is manufactured by pressing the veneers for 60 minutes with high temperature the density of the board becomes so high that the density peaks in the bond-lines merge with the rest of the veneer (Mansouri et al. 2010).

2.4.2 Effect of Moisture on Bond Formation and on Bond Integrity

The water resistance of self-bonded plywood increases as temperature, pressing pressure, and treatment time increase. Also boards that are manufactured under the most severe conditions do not delaminate when soaked in water. (Ando & Sato 2009, Cristescu et al. 2015b) Furthermore, the bonds that do not delaminate during water soaking are also the strongest ones under mechanical stress. Thus these results support the hypothesis that there is a relation between the strength of self-bonded plywood and chemical bonds in the bond line. (Cristescu et al. 2015b)

Swelling caused by the spring back phenomenon in the thickness direction inflicted by water is still relatively high because of the mechanical compression during hot pressing (Cristescu 2015). However the chemical changes that affect the nearest bond lines also give more protection against water, despite the fact that bond lines are also the most densified parts of the board. Presumably the joint effect of thermally modified, cross-linked lignin and the degradation products of the hemicelluloses are the main reason for the increased water repellency, although the mechanism is not fully known (Cristescu 2015, Karlsson et al. 2012, Repellin & Guyonnet 2005). Ruponen et al. (2015a) studied the effect of thermal treatment on the internal bond strength of welded wood. Thermal modification reduces the water sensitivity in the bond line but improvements in internal bond strength were only achieved with small samples, with larger samples the deformation by thermal treatment is too large. Additionally, the water repellency of self-bonded plywood has been reported to increase due to thermal treatment (Ruponen et al. 2014).

Soaking in water for a long period of time is a rather extreme test for self-bonded plywood. Also ambient humidity affects the bond strength between veneers. When the self-bonded boards are transferred to moister conditions swelling occurs and the weakest bonds break (Cristescu 2015).

The equilibrium moisture content (EMC) of wood is dependent on relative humidity and temperature: higher temperature causes lower EMC at the same relative humidity. Furthermore the higher pressing temperature of the self-bonded board decreases EMC. This reduction is probably caused by dehydration of carbohydrates due to the thermal degradation of wood, and a reduction of hydroxyl group and other groups that contribute to the hydrophilic character of wood. (Cristescu 2015)

As discussed in Section 2.3, moisture content and temperature affect the softening of wood. There is also a connection with moisture content and self-bonding ability of veneer. Results by Ruponen et al. (2014) suggest that wet veneer forms stronger bonds than drier veneer (conditioned in 65 % RH). Also the moisture is stated to be essential in bond formation due to the increased cross-linking of lignin and increased space for the lignin and hemicellulose matrix (Ruponen et al. 2014, Ruponen et al. 2015b). However, higher moisture content also increases internal vapor pressure during hot-pressing. Because of the high vapor pressure delamination of the bonding and even explosion has been reported when the pressure is released (Ruponen et al. 2015b, Cristescu et al. 2015b). Additionally, the mechanical properties of wood with higher moisture content are reported to reduce more when temperature rises than the mechanical properties of wood with lower moisture content (Gerhards 1982). Nonetheless, Pinitiaux et al. (2015) studied the binderless bonding properties of various lignocellulosic fiber materials and concluded that the optimal moisture content of the raw material is approximately 5 – 7 %, although the optimal moisture content is dependent on pressing conditions. For example 0 % moisture content significantly reduces the molding properties of the raw material and requires more heat to soften. (Pinitiaux et al. 2015). Alternatively, high temperature (at least 180 °C) is enough to soften lignin sufficiently to make a bond (Okuda et al. 2006).

2.4.3 Color Changes due to Heat

During the self-bonding of veneers the temperature of the hot-press plates is over 200 °C. A temperature this high causes darkening in wood. Color change is a complex phenomenon that can be explained by the degradation of hemicelluloses, lignin, and extractives (Sundqvist 2004). Bekhta & Niemz (2003) studied the effect of heat treatment time and temperature on the color changes of spruce. They found that the color starts to drastically change in the region of 200 °C; also a relationship with strength properties was noted. Also other properties such as mass loss and density loss correlate with an increase in darkness after heat treatment (Todorovic et al. 2012).

Considering the self-bonding of beech veneer, Cristescu et al. (2015c) discovered that the hardness of a board increases as the brightness decreases. Color changes may also be linked to the formation of new extractives and the rate of hemicellulose degradation (Kocaefe et al. 2008, Sundqvist 2004). Additionally, it is stated that as hemicelluloses and cellulose do not contribute to the change of color, the lignin content with hemicellulose-derived extractives could be the decisive factor in color change (Kocaefe et al. 2008). Therefore, the more drastic color change of birch compared to aspen is explained by the higher density and thus the higher actual amount of lignin in birch (Kocaefe et al. 2008).

2.5 Mechanical Properties of Self-bonded Wood

2.5.1 Bending and Shear Strength

The strength properties of self-bonded boards are dependent on the severity of treatment (Cristescu et al. 2015b). The high temperature of the press degrades the wood material and lowers the strength, but in conjunction with compression the strength properties improve (Blomberg et al. 2005, Fang et al. 2012). However, the main improvement in strength properties of self-bonded board is caused by better bonding performance. In shear tests performed on beech plywood, the weakest section in the board is in the bond line. In bending tests two kinds of failure types are noted. The boards pressed under milder conditions broke due to interlaminar shear in the bond line. The boards pressed in more severe conditions broke due to tensile forces in the bottom layer. (Cristescu et al. 2015b) In conclusion the strength properties of self-bonded plywood increase simultaneously with the occurrence of chemical changes in the bond line, even though thermal degradation weakens the wood material. Therefore, water absorption of the board is at its lowest point when shear strength reaches its maximum as seen in Figure 5 (Cristescu 2015).

Compression of veneer induces a smoothening of surfaces and conglutination of lathe checks (Fang et al. 2012), which are reported to have an effect on the shear strength properties of plywood (Rohumaa et al. 2013). In traditional plywood manufacture the lathe checks of surface veneers are normally faced inside and are not visible in the plywood surface. However, facing lathe checks on each other may cause delamination in self-bonded plywood. (Ruponen et al. 2014)

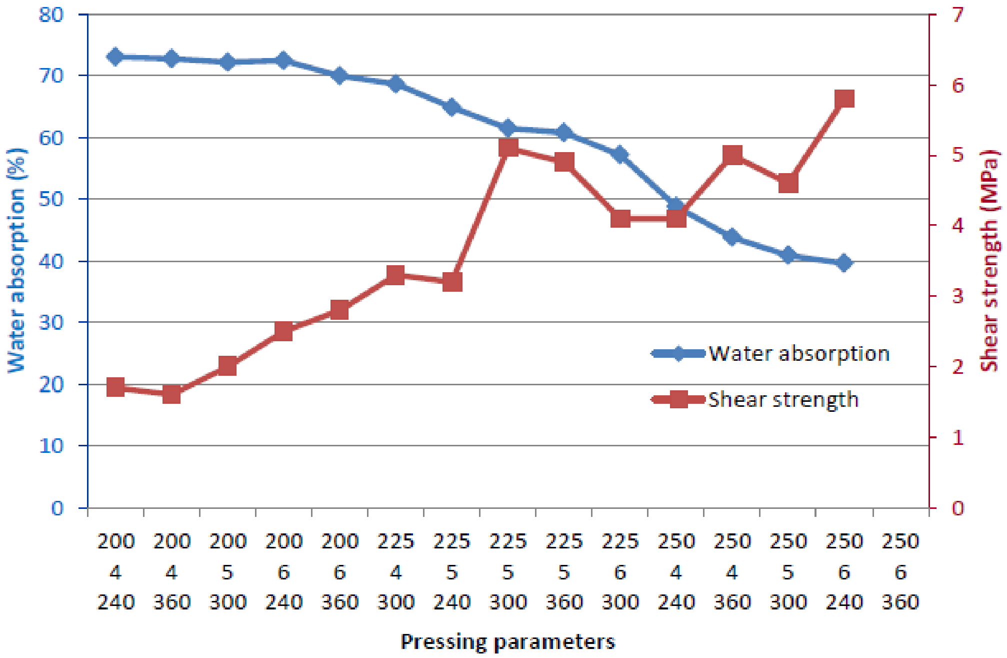


Figure 5. Effect of pressing parameters on water absorption and shear strength of self-bonded beech plywood. (Cristescu 2015) Pressing parameters: 200 °C, 225 °C, 250 °C; 4 MPa, 5 MPa, 6 MPa; 240 s, 300 s, 360 s.

2.5.2 Hardness

Hardness is a mechanical property that is most affected by a combination of heat and pressure and densification is reported to increase the hardness of veneer by over two times compared to a reference sample (Fang et al. 2012). Hardness is additionally highly dependent on the increase in density while strength properties are mostly improved along with the degree of bonding (Cristescu 2015c). Although heating during densification positively affects hardness, heat treatment for over five hours after densification is reported to reduce hardness (Morsing 2000). This is suggested to be caused by drying and degradation of wood material due to long exposure to heat (Laine et al. 2013). Additionally, loss of elasticity is noted to decrease the hardness of densified wood, which suggests that the softening and deformation capability especially near surface are important factors in terms of hardening (Laine et al. 2013, Rautkari, et al. 2011).

2.6 Effect of Log Soaking Temperature on Veneer Properties

Prior to peeling the log is normally soaked in hot water heated in order to soften the wood material and to produce a better quality veneer by reducing cutting forces (Marchal et al. 2009, Dupleix et al. 2013). However, log soaking temperature has also been noted to influence the chemical and physical properties of birch veneer (Rohumaa et al. 2016). The various effects of log soaking temperature on veneer properties are discussed in this section.

Significant changes in wood properties have been noted when the log soaking temperature is elevated from 50 °C to 70 °C. At temperatures below 50 °C the changes are minimal (Yamamoto et al. 2015b). Soaking the log at 70 °C is reported to decrease the proanthocyanidin content of birch veneer and the pH of wet veneer (Yamamoto et al. 2015b). Proanthocyanidins are unstable phenolic molecules that may affect polymerization in acidic conditions and they also oxidize easily (Yamamoto et al 2015b). Proanthocyanidins have been suggested to work as cross-linking agents in dental and wood adhesives (Bedran-Russo et al. 2007, Ping et al. 2011). Alternatively, log soaking at high temperature has been noted to increase the share of extractable monosaccharides, especially glucose and fructose, and also extractable lipophilic matter possibly due to the degradation of hemicelluloses (Yamamoto et al. 2015a, 2015b). However, the slight degradation of wood material and decrease in proanthocyanidin content may reduce the veneer's protection against biological attack (Yamamoto et al. 2015b).

Regarding physical properties, a log soaking temperature of 70 °C substantially darkens the color of veneer especially in long term storage, which is probably caused by a decrease in the proanthocyanidin content (Yamamoto et al. 2015b). Furthermore, differences in veneer surface roughness, wettability and bonding ability have been noted with veneers that are soaked at different temperatures. Soaking at higher temperature for at least 48 h has reported to have an effect on surface roughness, but the results are somewhat controversial and differ in heartwood and sapwood and on different sides of veneer (Aydin et al. 2006, Dundar et al. 2006, Rohumaa et al. 2016). Additionally, the method to measure the surface roughness may not illustrate the actual changes properly (Rohumaa et al. 2016). Therefore, Rohumaa et al. (2016) studied the surface integrity of veneer and reported that larger particles disengage from the surface of veneer that is peeled from logs soaked at 20 °C in comparison with veneer from logs soaked at 70 °C. A larger amount of small particles on the surface of veneer peeled from logs soaked at the higher temperature induces a hairier but more integrated surface, which also has most likely a great effect on reduced contact angle (Rohumaa et al. 2014, 2016). Furthermore, a correlation between wettability and bond strength on synthetic adhesives has been reported (Aydin et al. 2006, Rohumaa et al. 2014).

3 MATERIALS AND METHODS

3.1 Veneer Preparation and Soaking

The wood material used in this study was Finnish silver birch (*Betula pendula*). Two logs, approximately 1.2 m in length, were cut from the same trunk and soaked in water for 48 hours. Water in the soaking tank was heated for one log to 70 °C and for the other log, the temperature was kept at room temperature (20 °C). After soaking, the logs were debarked and then peeled on an industrial-scale rotary lathe (Model 3HV66, Raute Oyj, Lahti, Finland). The veneers for ABES testing were peeled into thickness of 0.8 mm and the veneers for plywood manufacture were 1.5 mm thick. Then the veneer mat was cut into sheets that were as uniform and as knot-free as possible. The sheets were dried in a convective laboratory scale dryer at 160 °C for approximately 1.5 minutes. After drying the sheets were stored in a room with temperature of 25 °C and (30 ± 5) % RH before further processing.

3.2 Moisture Conditioning

Veneers were conditioned in two different conditioning chambers. The conditions in the chambers were the aforementioned 25 °C and (30 ± 5) % RH and 20 °C and (60 ± 5) % RH. The veneers were grouped into these two chambers and they were equilibrated allowing the moisture to stabilize for one week. Also after hot pressing with ABES the specimens were conditioned in the chambers for one week.

3.3 Moisture Content Measurement

Moisture content and mass change of the veneers was measured in order to investigate the duration that it would take for the samples to reach the required EMC in both conditioning chambers. Figure 6 shows the arrangement of the moisture control tests.

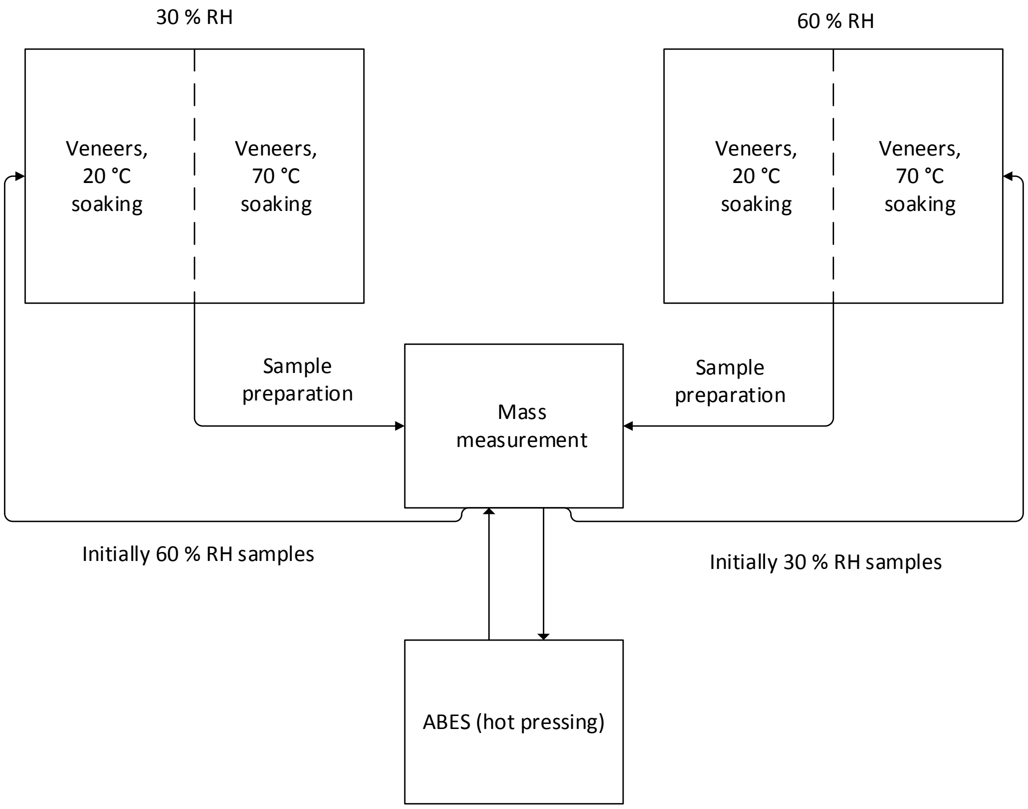


Figure 6. Schematic picture of moisture content measurement prior to moisture conditioning.

Five pairs of samples from each of the soaking temperatures and relative humidity condition combinations were prepared for weighing. The size of the samples was the same as with ABES testing, (117 x 20 x 0.8) mm. After the mass was measured the pairs of samples were hot pressed with ABES. Pressing time was 300 s with a pressing temperature of 220 °C and pressing pressure of 5 MPa. After hot pressing the samples were weighed again and then placed into the other room from which they were taken initially. Consequently the samples that were in the chamber with 30 % RH were placed into the chamber with 60 % RH and vice versa to maximize the difference in moisture contents of the samples prior to and after conditioning. The mass of the specimens was measured for a few days according to standard EN 14251:2003 Chapter 5 at intervals of 6 h until the mass change was under 0.1 % (Equation 2). Mass of the specimens was measured with a Precisa XM60 scale with a precision of 0.001 g.

After the specimens were conditioned, they were dried in a convection oven for 24 h in (103 ± 1) °C to find out the dry mass. Then the specimens were weighed again to determine moisture content with equation 1

$$MC = \frac{m_1 - m_0}{m_0} \cdot 100 \tag{1}$$

where m_1 is the mass of the test piece prior to drying,
m_0 is the mass of the oven dry test piece,
MC is the moisture content in percent.

Mass change is calculated with equation 2

$$W = \frac{m_a - m_b}{m_b} \cdot 100 \tag{2}$$

where m_a is the present mass of the test piece after each measurement,
m_b is the mass of the test piece of previous measurement,
W is the mass change in percent.

3.4 Veneer Hot Pressing and Bond line Testing with ABES

3.4.1 Sample Preparation

0.8 mm thick veneer sheets were cut into smaller pieces in order to prepare the ABES samples with special cutter designed for ABES. The dimensions of the ABES samples were 117 mm x 20 mm and by cutting the samples with the special cutter it is ensured that all the samples were exactly the same size and shape. When cutting the samples, it was ensured that the grain direction was parallel. Also it was ensured that all of the samples were free from irregular parts such as knots.

After cutting the samples, they were placed in sealable plastic bags to keep the moisture content of veneers as near as possible to the level that it was in the conditioning rooms. Furthermore, the samples were stored in the plastic bags for a maximum of one h prior to hot pressing with ABES.

3.4.2 Veneer Bonding with ABES

The ABES testing machine (Adhesive Evaluation Systems, Inc., Corvallis, Oregon, USA) consists of a mini scale hot press and grips with platforms that keep the samples in place. The system operates with pneumatics and is controlled by computer. (Wescott et al. 2007) The ABES testing machine is shown in Figure 7.

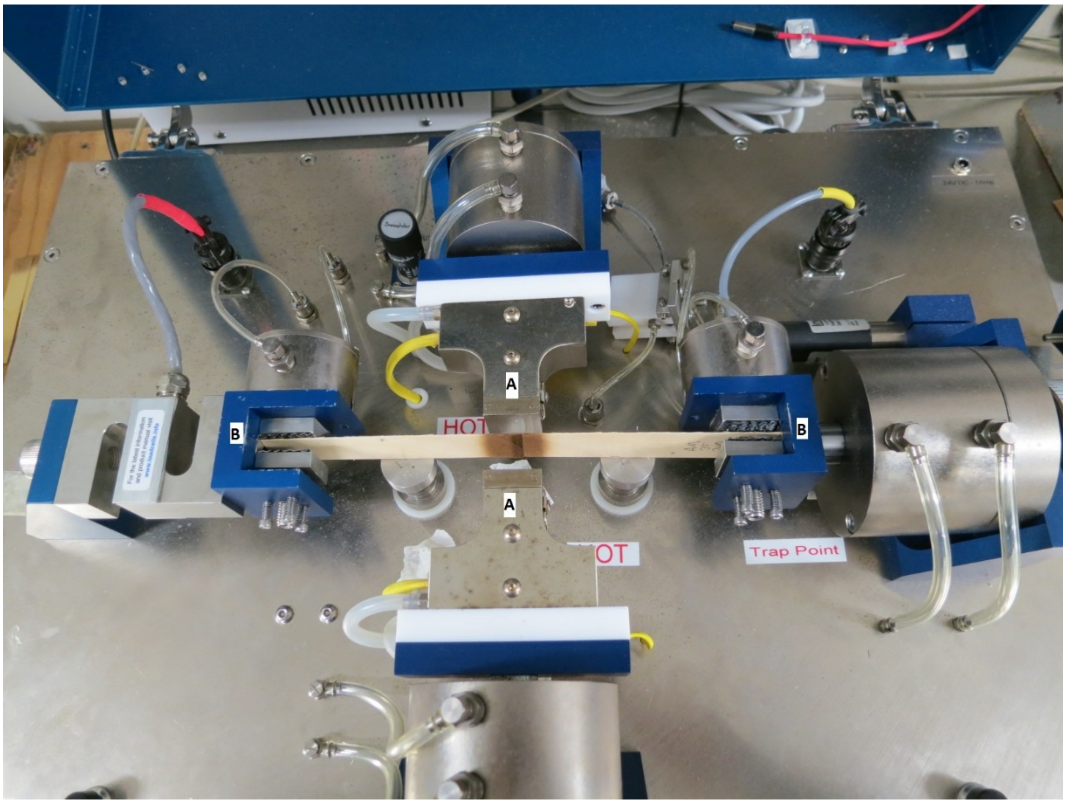


Figure 7. Automated Bonding Evaluation System (ABES). A: heated press plates, B: veneer grips.

This thesis involves a study of the effects of certain process parameters on the self-bonding ability of wood. Therefore pressing time and pressing temperature were varied during the tests. Pressing times that were used were 60 s, 120 s, 180 s, 240 s, 300 s, 360 s, 420 s, 480 s, 540 s, 600 s, 660 s, 720 s, 780 s, 840 s, and 900 s. Pressing temperatures were 180 °C, 200 °C, 220 °C, and 240 °C. Deviation in temperature was a maximum of ± 1 °C. Additionally, a thermocouple was used to determine the temperature in bond line with different pressing temperatures. Pressing pressure was held constant at 5 MPa. Each pressing and measurement with every parameter combinations were replicated seven times.

Regular size and shape of the samples enables the same bond area with every bonding. 4 mm overlapping of the veneers makes the bond area relatively small: 4 mm x 20 mm = 80 mm^2. However, the length of the overlap was only controlled with the ABES stopper and no control measurements were performed, which possibly cause slight variation in the results. Moreover, the side of the veneer with lathe checks was always bonded to the side which had no checks. Layout of the veneer bonding arrangement is shown in Figure 8.

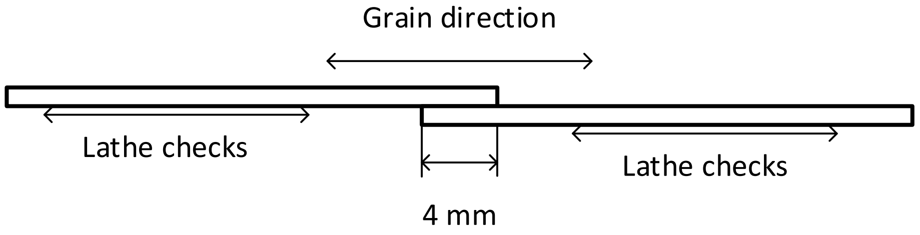


Figure 8. Veneer lathe checks and veneer orientation in ABES testing.

3.4.3 Tensile Shear Strength Testing of Bond line with ABES

After bonding, the bonded pairs of veneers were stored in the conditioning rooms for a week (Section 3.2). Then the conditioned samples were tested for tensile shear strength in ABES. The samples were placed between the grips and then the grip at one end quickly pulls the sample apart from the grip at the other end that is fixed. The process stresses the bond to fracture while a sensor measures the maximum force that is reached.

The actual tensile shear strength of the bond is calculated with equation 3

$$\sigma = \frac{F}{A} \tag{3}$$

where σ is tensile shear strength of the bond (N/mm^2),
F is maximum force (N),
A is the bond area of the veneers (mm^2).

3.4.4 Statistical Analysis

The results of ABES strength tests were analyzed using a one-way analysis of variance with Bonferroni correction. The tests were done to compare the means of the results of different raw material characteristics. All the comparisons were done for every pressing time and for 220 °C and 240 °C of pressing temperature separately. Significance level was set to 5 %, consequently the difference is considered to be statistically significant when $p < 0.05$.

3.5 Manufacture of Self-bonded Plywood and Testing

3.5.1 Plywood Manufacture

The dimensions of the veneers used in the laboratory scale plywood manufacturing were (88 x 88) mm and thickness 1.5 mm. Veneers were cut with a hydraulic clipper and then brought to the hot press. Only veneers that were peeled from logs soaked at 20 °C were used.

Plywood with five and seven veneers was manufactured by using a material test system (MTS) 810 (MTS Systems Corporation, Minneapolis, Minnesota). Parallel and cross lamination of veneers were used. Pressing pressure was held at 5 MPa. However, there were three different combinations to release the pressure at the end of the pressing cycle. Quick release: all the pressure was released at once when the time was complete. Gradual pressure release: when 2 or 3 minutes of pressing time was left, pressure was started to release step by step by halving the pressure, until there was only approximately 0.1 MPa of pressure for the last 30 seconds prior to the hot press being completely opened. These three options are presented more detailed in Table 1.

Table 1. Three different options that was used to release the pressure of the hot press in the last three minutes of the pressing cycle during plywood manufacturing. Press was controlled manually and therefore the times and pressures are not accurate.

Time Remaining	***Quick Release***	***Gradual Release, 2 min***	***Gradual Release, 3 min***
180 s	*5 MPa*	*5 MPa*	*2.5 MPa*
120 s	*5 MPa*	*2.5 MPa*	*1.2 MPa*
90 s	*5 MPa*	*1.2 MPa*	*1.2 MPa*
75 s	*5 MPa*	*1.2 MPa*	*0.6 MPa*
60 s	*5 MPa*	*0.6 MPa*	*0.6 MPa*
45 s	*5 MPa*	*0.6 MPa*	*0.3 MPa*
30 s	*5 MPa*	*0.3 MPa*	*0.3 MPa*
15 s	*5 MPa*	*0.1 MPa*	*0.1 MPa*
0 s	*0 MPa*	*0 MPa*	*0 MPa*

Pressing time ranged from 7.5 minutes to 30 minutes depending on the temperature and the lamination of the veneers. Pressing temperature in this case was not very accurate, because the lower plate of the hot-press was approximately 20 °C warmer than the upper plate. Therefore, the temperature in the center part of the plywood was measured. The temperature varied between 210 °C and 240 °C.

Temperature in the bond lines was once measured with thermocouples that were placed in the middle of each of the veneer layers. When changing the temperature, only the middle bond line was monitored. The same thermometer was used in this case as with the temperature measurement of the bond line of the ABES samples.

3.5.2 Sample Preparation

For shear strength testing, the samples were prepared according to standard EN 314. However, the boards were only tested for shear without any treatments, i.e. boiling in water. Two cuts, deep enough to break three bond lines, were sawn on both sides of the sample. Because of the small size of the plywood boards, only two samples were made from the center part of each board.

3.5.3 Shear Strength Test of Self-bonded Plywood

Prior to testing the samples were stored in conditioning room with 35 % RH and 25 °C. Also the dimensions of the bond area were measured. The shear strength of the bond line was tested using a Zwick 1475 testing machine. After the bond was broken, the quality and position of the fracture was visually evaluated.

A load cell in testing machine measured the maximum breaking force, and the tensile shear strength was calculated with equation 4

$$f_v = \frac{F}{l \cdot b} \tag{4}$$

where f_v is the shear strength of the test piece (N/mm^2)

F is the failing force of the test piece (N)

l is the length of the shear area (mm)

b is the width of the shear area (mm).

4 RESULTS AND DISCUSSION

4.1 Moisture conditioning

The level of moisture conditioning was assessed by mass change %. Acceptable mass change % within measurements of 6 h interval is less than 0.1 %.

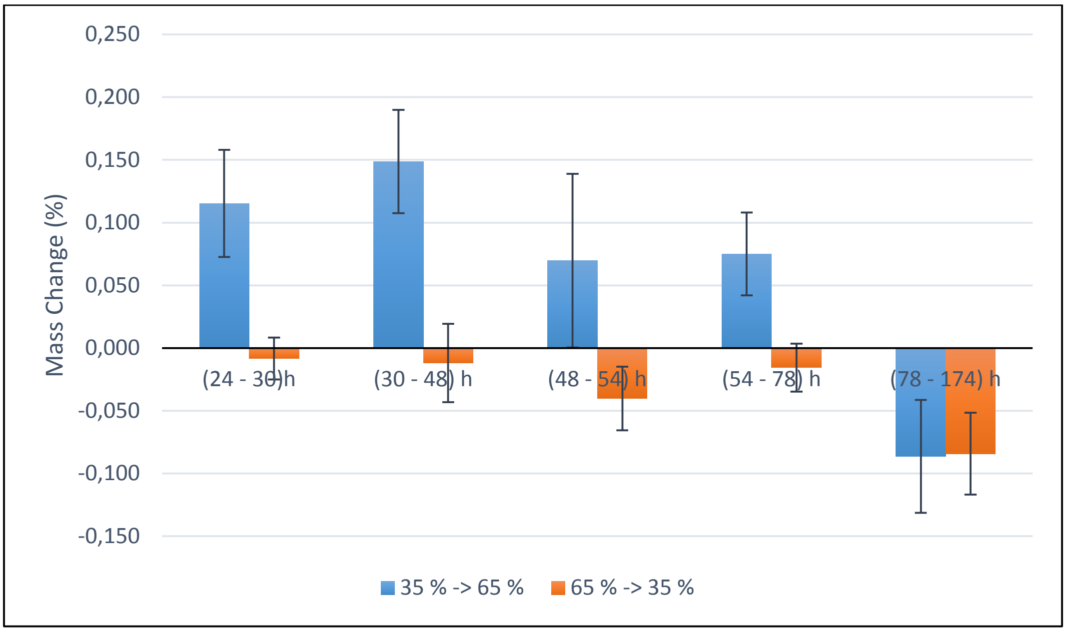


Figure 9. Mass change % of samples that were conditioned in 35 % RH prior to hot pressing with ABES and moved to 65 % RH after hot pressing and samples that were conditioned in 65 % RH and moved to 35 % RH after hot pressing. Times are hours after hot-pressing. Error bars are standard deviations.

Figure 9 shows that conditioning at 65 % RH induces too large a mass change within 6 h during the first day after hot-pressing. Also the mass continues to rise until 48 h, when the mass change has reached its required level. Conditioning at 35 % RH after hot pressing is less critical in terms of mass change % and these samples reach the required moisture content in 24 h. Furthermore, conditioning for one week does not affect mass change of either of the groups too much. Therefore it was decided to condition all the samples for one week prior to tensile shear testing.

The small changes in mass, even after longer conditioning times than required, may be mostly caused by the variation in relative humidity in conditioning chambers. Moreover the standard deviations are relatively high compared to the actual averages of the results. High standard deviations can be explained by the small size of the samples (the weight of two ABES samples was approximately 2.5 g) and the precision of the scale (0.001 g).

The moisture contents of the ABES samples in different parts of the process are shown in Figure 10. Moisture content reduces relatively quickly during ABES hot pressing, although only a small area of the samples is heated. Also the drier samples that are conditioned in higher relative humidity after hot pressing undergo the greatest change in moisture content.

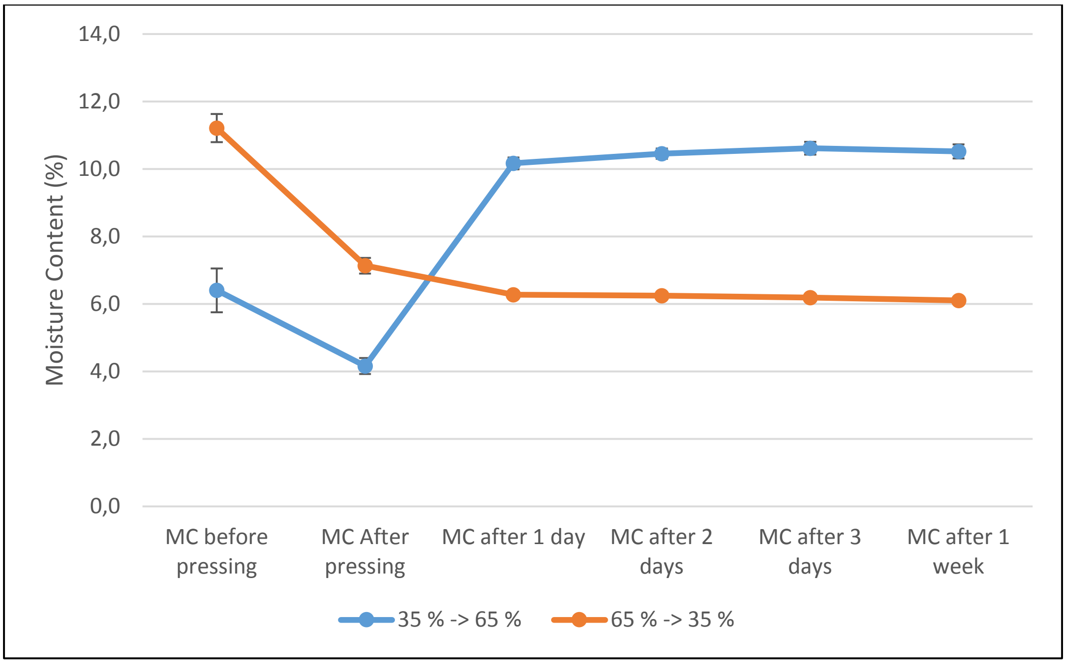


Figure 10. Moisture content of ABES samples during moisture conditioning. Error bars are standard deviations.

Based on these results the moisture content of the samples that are conditioned at 35 % RH prior to hot pressing is approximately 6.4 %, and of the samples that are conditioned in 65 % RH approximately 11.2 %. The moisture content after hot pressing and one week conditioning is slightly lower and it is also dependent on the severity of the treatment.

4.2 ABES Results

Table 2 shows the coding for the parameters in ABES testing. The coding is used to facilitate the presentation of the results of tensile shear tests with ABES and statistical analysis.

Table 2. Coding for the parameters that were used in ABES testing.

Coding	Explanation
A	Log soaking temperature 70 °C
B	Log soaking temperature 20 °C
D	Drier veneer that is conditioned in maximum of 35 % RH before bonding
M	Moister veneer that is conditioned in maximum of 65 % RH before bonding
35	Veneer that is conditioned in maximum of 35 % RH after bonding
65	Veneer that is conditioned in maximum of 65 % RH after bonding
200	Pressing temperature 200 °C
220	Pressing temperature 220 °C
240	Pressing temperature 240 °C

Figure 11 shows tensile shear strength as a function of pressing time for all the combinations of the parameters that were used. Each of the points is an average of seven replicates. If the veneers delaminated either immediately after the press opened or prior to strength testing the value of measurement was marked as 0. The colors of the curves are the same with each similar parameter combinations pressed at different temperatures. For example BM 65 220 and BM 65 240 are both marked with yellow.

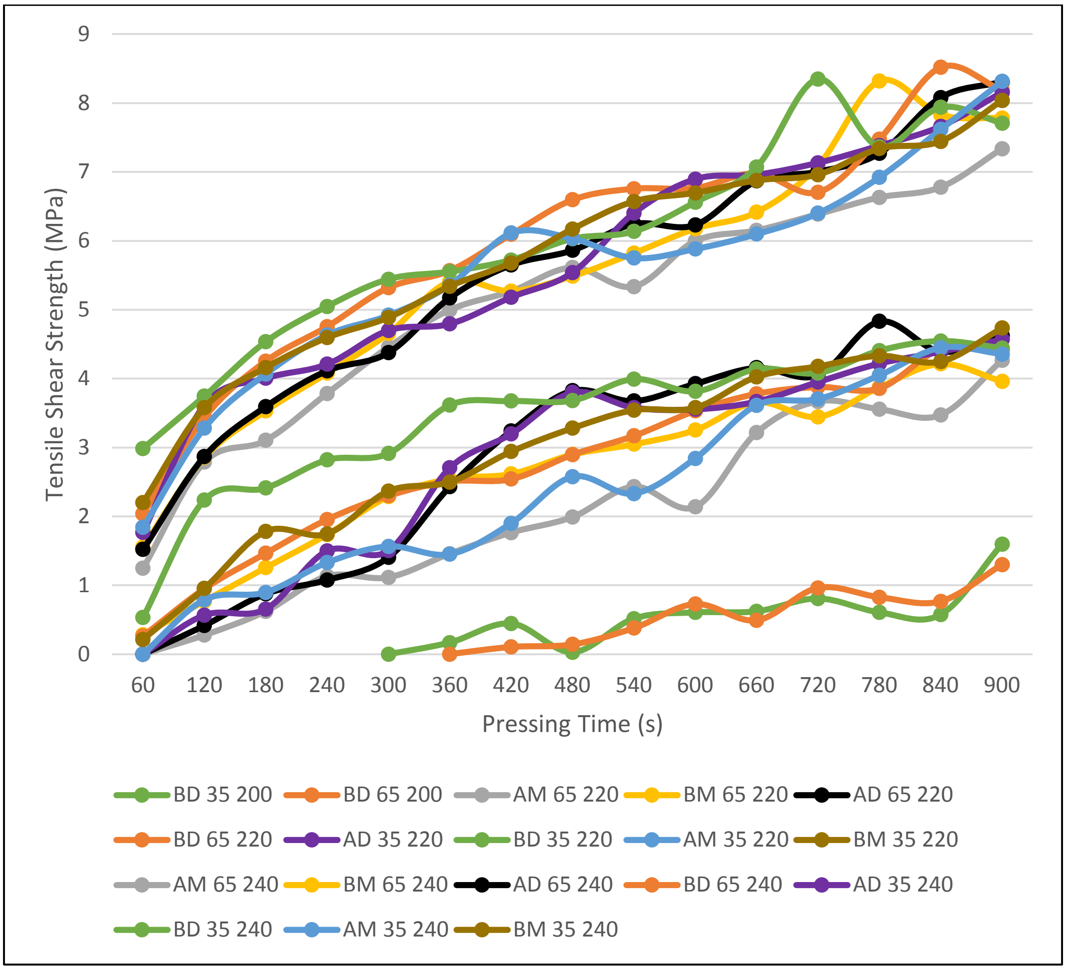


Figure 11. Tensile shear strength of self-bonded birch veneers in function of pressing time. Coding corresponds the explanation that is presented in Table 2.

4.2.1 Effect of Pressing Time and Temperature

Pressing time and temperature have a major impact on the self-bonding ability of veneer as seen in Figure 12. The rise in temperature from 220 °C to 240 °C has a relatively strong influence on bond formation especially with short pressing time. Furthermore, the graphs in Figure 11 are clearly separated from each other with respect to temperature, although there is variation in the results. It was not possible to form any bonds when pressing at 180 °C within the time frame that was used.

A longer pressing time also increases the tensile shear strength of self-bonded veneer. However, the rise is steepest with a short pressing time and it starts to flatten out near 900 s. With a pressing temperature of 200 °C the bond formation is very weak below 900 s, but the tensile strength values begin rising slightly more rapidly. Nevertheless, it is unlikely that the values will rise within a reasonable time due to the fact that only BD-veneer (log soaking temperature 20 °C, drier veneer conditioned in 35 % RH before bonding) was able to form a bond within these conditions.

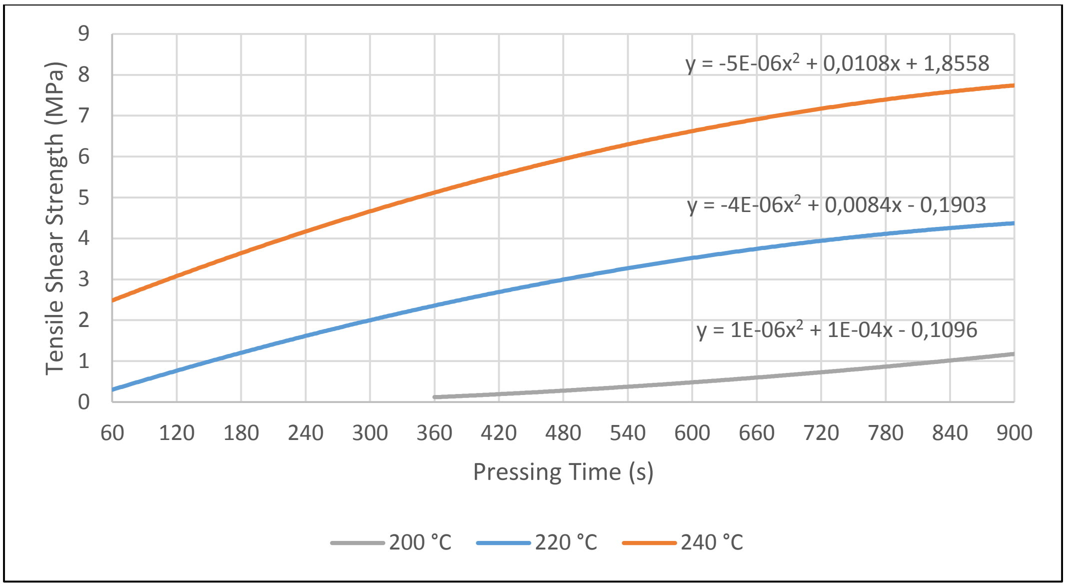


Figure 12. Effect of pressing time on tensile shear strength of self-bonded veneer.

Figure 13 shows the dependence of proportional standard deviation on pressing time and temperature. The graphs highlight the uncertainty of bonding at low temperature and short pressing time. Due to the fact that the delamination of the veneers results in tensile shear strength of 0, delamination increases standard deviation. High proportional standard deviation reveals the fact that bond formation is occasional, and therefore, especially pressing samples at lower temperatures cause that it takes a longer time to reach even moderate standard deviation.

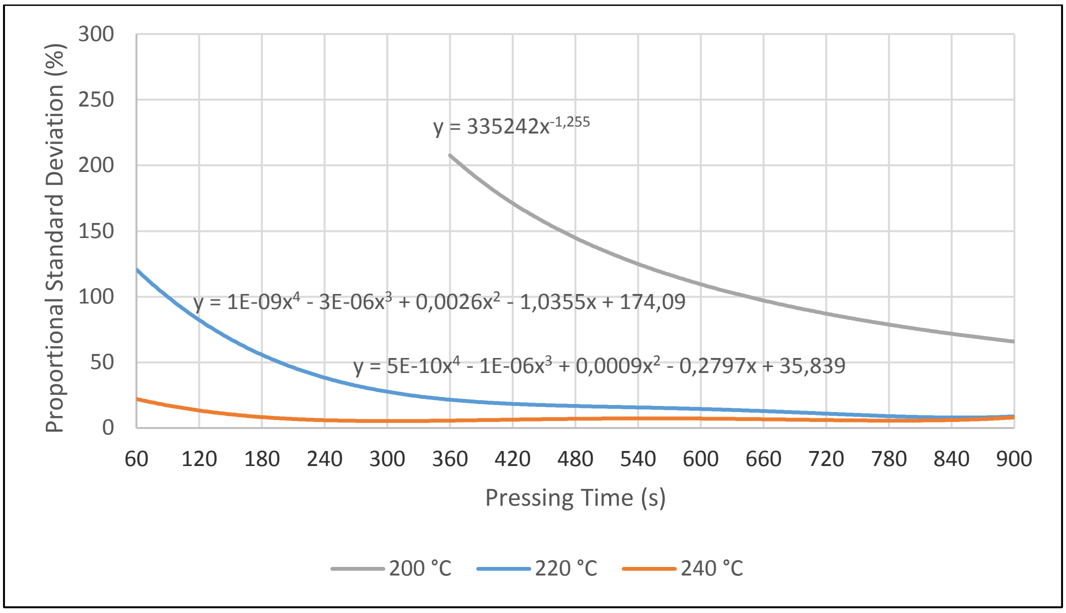


Figure 13. Proportional standard deviation for tensile shear strength of self-bonded veneer pressed in different temperatures as a function of time.

Figure 14 shows the color changes of veneers that are self-bonded with ABES. Darkening of veneers pressed at 240 °C appears to be drastic. The color also seems to become darker along with the severity of treatment, i.e. with longer hot-pressing time and higher temperature.

As discussed in Section 2.4.3 there is a connection between color and mechanical properties of self-bonded veneer joint. The results in this thesis also suggest that there may be a correlation between bonding strength and the color of the bond zone. Although this observation is not confirmed, it could be worth of further studying with ABES.

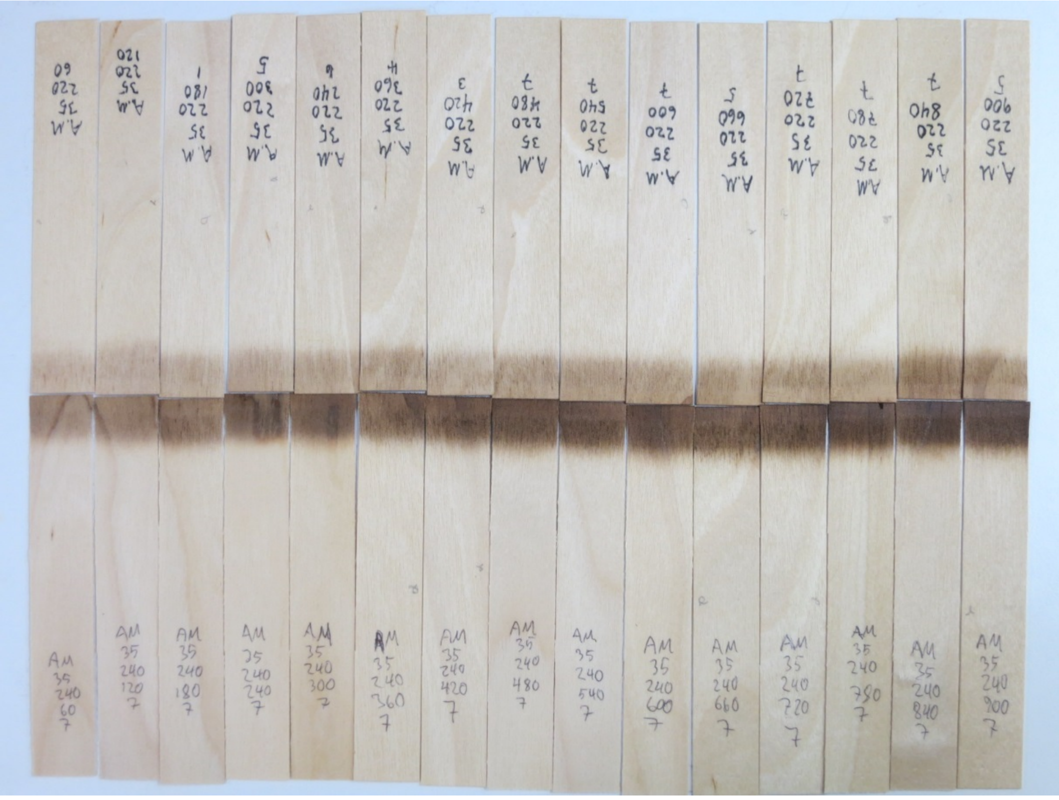


Figure 14. ABES-pressed veneers at different temperatures and times. From left to right: (60 - 900) s. Above: 220 °C. Below: 240 °C.

The bonding and strength tests with ABES provide uniform results with previous studies in terms of pressing time and temperature: higher temperature and longer time enables stronger bonds, and moreover the effect of temperature is substantial (Cristescu 2008). As the temperature rises to 240 °C the chemical changes in hemicelluloses and lignin occur more rapidly and effectively in order form a bond within a relatively short period of time. For example pressing at 240 °C for 300 s provides higher strength on average than pressing at 220 °C for 900 s. Additionally, there is reason to assume that bonding strength does not improve significantly after 900 s based on the shape of the graphs in Figure 12.

Proportional standard deviation appears to be a good indicator of overall bonding quality since the delamination of veneers increases standard deviation significantly. Proportional standard deviation over 100 % means that standard deviation is greater than average, and therefore the average values are very approximate and the formation of the bond is very uncertain because the delamination of the veneers causes the greatest deviation in the results. However, there is also dispersion in the data caused by other factors such as log soaking temperature and the moisture content of veneers. The effects of these factors with respect to bond formation are discussed within following sections. Additionally, possible changes in the size of the bond area and irregularity in the veneers may slightly increase the standard deviation.

4.2.2 Effect of Log Soaking Temperature on Bonding Ability

Log soaking temperature significantly affects the self-bonding ability of birch veneers at low pressing times as shown in Figure 15. After 360 s the difference is insignificant. B-veneer (log soaking temperature of 20 °C, explanation in Table 2) clearly has a greater ability to form a self-bonded joint than A-veneer. Pressing temperature of 200 °C was enough to form bond between BD-veneers (Figure 11) while A-veneers delaminated, except one 900 s AD-sample resulting average of 7 samples 0.39 MPa in comparison to graphs in Figure 11. Moreover, pressing at 220 °C for 60 s was not enough to form a bond with A-veneers, while bonding occurred within every group of B-veneers (Figure 11, Figure 15).

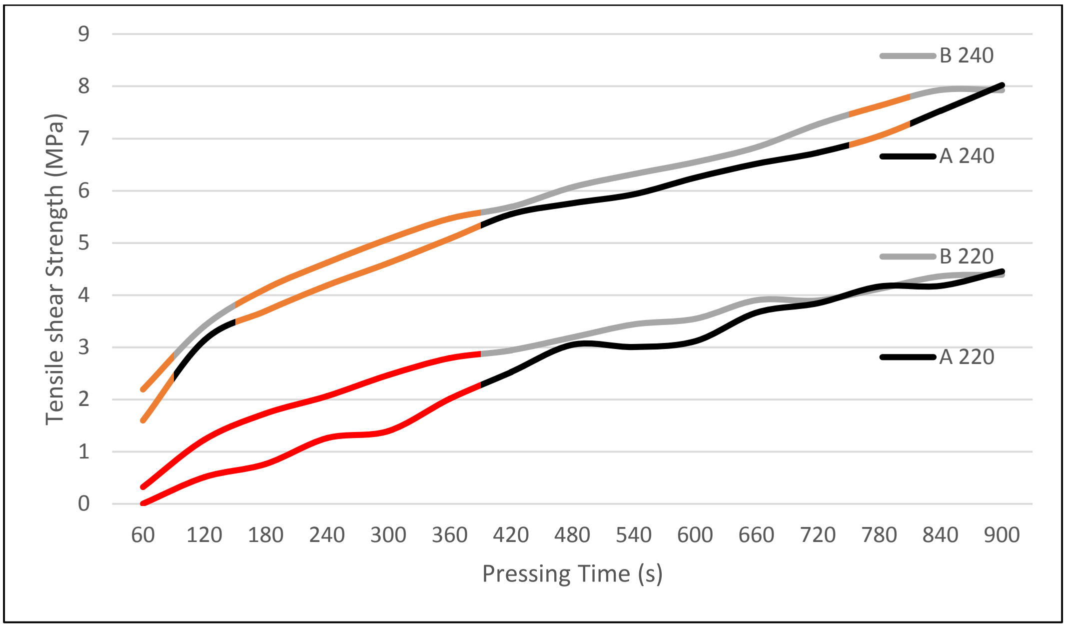


Figure 15. Effect of log soaking temperature on tensile shear strength of birch veneer. The same colored parts (red and orange) show where the graphs differ statistically significantly ($p < 0.05$) (Appendices 1 – 2). Coding corresponds the explanation that is presented in Table 2.

To conclude, the results indicate that veneer soaking temperature influences the self-bonding ability of birch veneer. Especially when a bond is starting to form it can be decisive whether the veneer is peeled from a log that is soaked at 20 °C or 70 °C. Birch logs soaked at 20 °C are reported to contain nearly three times more proanthocyanidins than those soaked at 70 °C (Yamamoto et al. 2015b). Considering the aforementioned cross-linking properties in Section 2.6 and its use as an ingredient in glues may suggest that proantocyanidins also contribute to self-bonding process. Additionally, the phenolic content of self-bonded veneer is reported to be highest at the bond line in 200 °C hot pressing (Cristescu & Karlsson 2013). With higher temperatures, the phenolic content slowly decreases (Cristescu & Karlsson 2013). This may also indicate the significance of phenolic proanthocyanidins in bond formation.

4.2.3 Effect of Veneer Moisture Content on Bonding Ability

Drier veneers form significantly stronger bonds than moister veneers especially with pressing times of 360 – 600 s and at 220 °C. The differences near 60 s and 900 s are smaller and statistically significant only at two single points. The results of the tensile shear tests with ABES and statistical differences are presented in Figure 16.

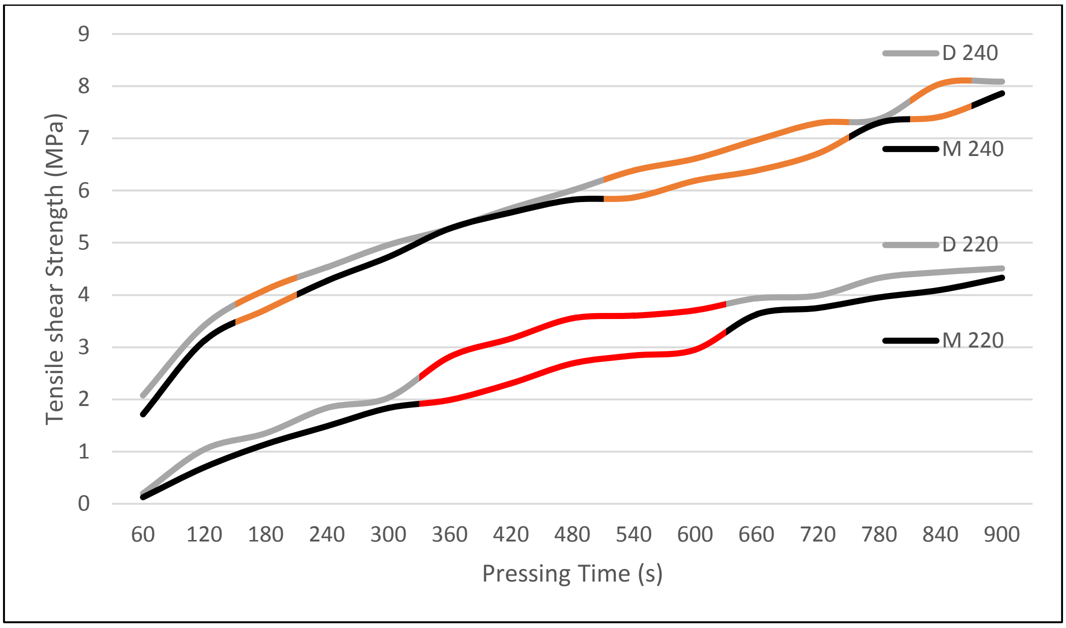


Figure 16. Effect of relative humidity prior to bonding on tensile shear strength of birch veneer. The same colored parts (red and orange) show where the graphs differ statistically significantly ($p < 0.05$) (Appendices 1 – 2). Coding corresponds the explanation that is presented in Table 2.

According to the literature a moisture content below 7 % is reported to be optimal regarding self-bonding and a rise in moisture content can relatively quickly lower the bonding ability of veneer (Pinitiaux et al. 2015). Alternatively, completely dry veneer increases the softening temperature needed, which may also decrease bond strength.

The small differences in the results with low pressing time may be influenced by the dominant effect of log soaking temperature. Especially at 60 s with 220 °C where the A-veneer did not bond at all while the BD-veneer formed a relatively strong bond causing a large gap between the bonding ability of veneers AD and BD. Although the effect of the moisture content of the veneer may not be as great as the effect of log soaking temperature on bond formation, their joint effect can be decisive. As can be seen in Figure 11 all the strongest bonds at pressing temperatures of 220 °C and 240 °C at short pressing time are gained with BD-veneer. Additionally, a pressing temperature of 200 °C was enough to form a bond only with BD-veneers. Therefore, the influence of the moisture content of the veneers in the middle of the curve (significant difference, Figure 16) may be greater than the influence of the other veneer characteristics, even though the influence of moisture content when the bond starts to form could be more important.

4.2.4 Moisture Resistance of Self-bonded Veneer

Figure 17 shows that with 240 °C pressing temperature there is a significant difference in tensile shear strength whether the bonded veneer is being stored at 65 % RH or at 35 % RH until 240 s of hot pressing. With 220 °C no significant difference is noticed based on these results.

None of the bonds delaminated during the storage in 65 % RH. All the delamination in the weakest bonds that were formed (and did not break immediately after opening the press) occurred only when the samples were placed on the platforms or from the impact of closing the grips of ABES.

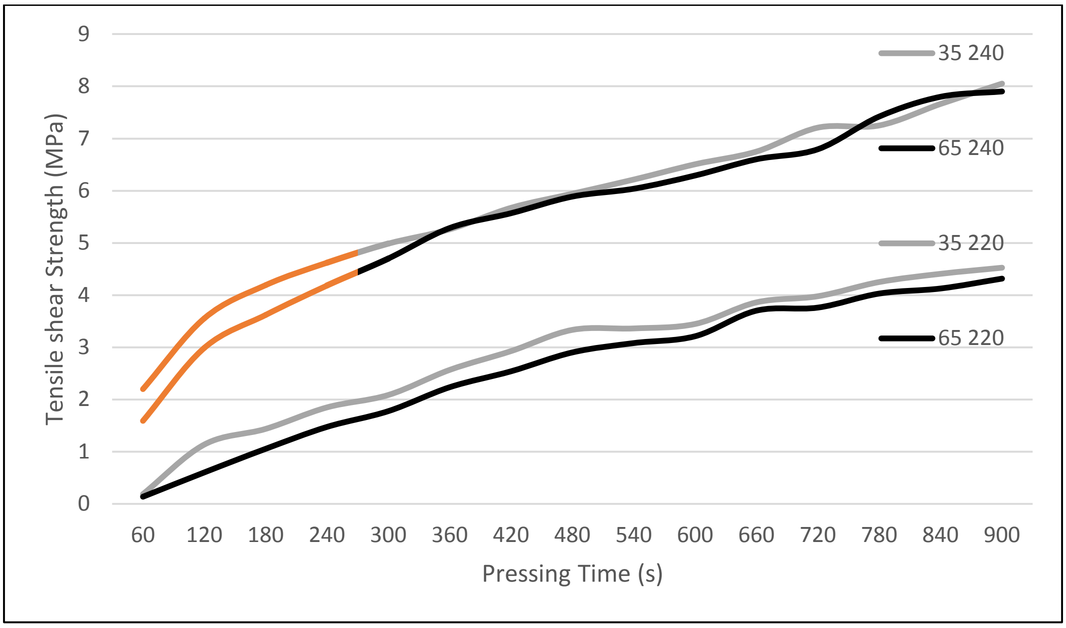


Figure 17. Effect of relative humidity after bonding on tensile shear strength of birch veneer. The same colored parts (orange) show where the graphs differ statistically significantly ($p < 0.05$) (Appendices 1 – 2). Coding corresponds the explanation that is presented in Table 2.

Small difference in strength results of 220 °C hot pressing may be caused by greater effect of log soaking temperature and moisture content of veneer prior to pressing. Nevertheless, there are signs of some kind of difference in frame of 120 – 480 s for 220 °C, although the difference is not significant. Additionally, delamination inflicted by other factors than moisture conditioning after bonding with 220 °C hot pressing are not taken into account.

Pressing at 240 °C caused no delamination while pressing at 220 °C caused several delamination with short hot-pressing time and the moisture variation after the bond is formed is not the reason for delamination, as stated earlier. Strength values of BD-veneer pressed in 200 °C (Figure 11) showed no significant difference (Appendix 3) whether they had been conditioned at 35 % RH or at 65 % RH. In this case it was also possible to inspect merely the effect of moisture conditioning after pressing with fixed variables and without delamination caused by other factors, because only one type of veneers were successfully bonded.

In conclusion, the results considering moisture resistance are somewhat contradictory: pressing at higher temperature causes more significant difference on the results of strength tests. As shown in Figure 5 the water absorption should decrease within more severe conditions (Cristescu 2015). However, the difference in relative humidity that was used in this thesis causes approximately 4 percentage point difference in moisture content of veneers (Figure 10). Greater difference in moisture content could produce clearer results. Additionally, it is not known how strong the bond needs to be in order to survive the opening of the press, where the internal gas pressure could exceed the bond strength. The weakest bonds delaminate immediately as the press open and stronger ones are presumably less affected by humidity. However, the results suggest that the moisture resistance of self-bonded birch veneers is sufficient within the conditions used in this thesis, but minor lowering of bond strength indicates that greater moisture content may reduce moisture resistance significantly.

4.3 Heat Evolution and Measurement in Bond Line

Figure 18 shows that heat reaches acceptable level in less than 15 s with ABES. However, the temperature in the bond line stays a few degrees below desired temperature until 50 s possibly due to higher energy consumption as the moisture in veneer evaporates.

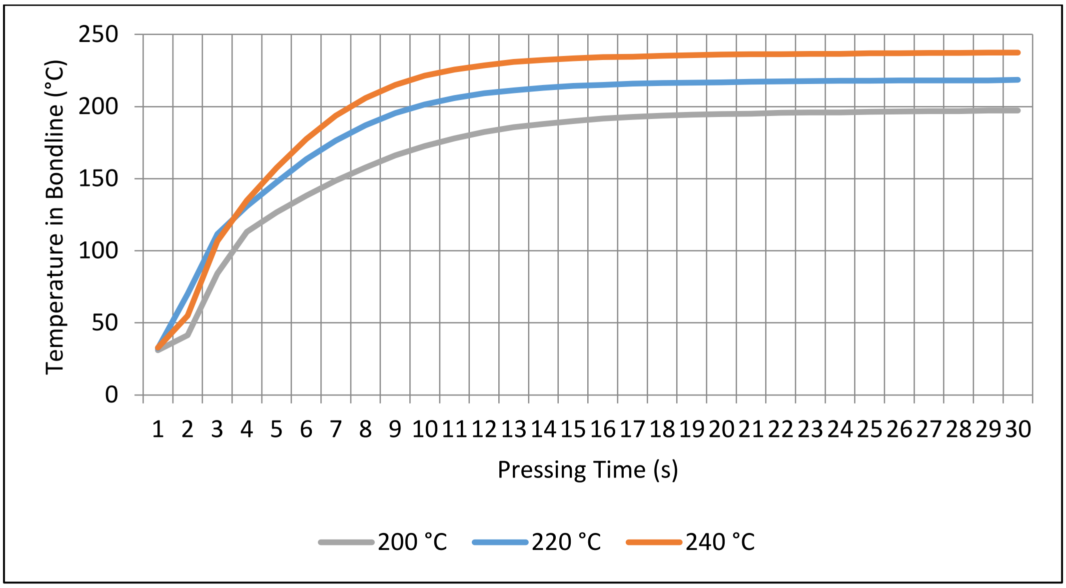


Figure 18. Heat evolution in bondline of ABES samples pressed with different temperatures.

Based on the results, heat reaches the bond line so rapidly that the effect on the strength results is minor. Nevertheless, the effect of temperature rising time on 60 s pressing time is proportionally the greatest.

The results in Figure 19 show that the layers closer to surface warm up more rapidly than the inner layers. As the maximum temperature is reached, the warmest layer is the lowest one and the coolest is topmost bond line. This is caused by the approximately 20 °C higher temperature on the lower pressing plate as mentioned in Section 3.5.1.

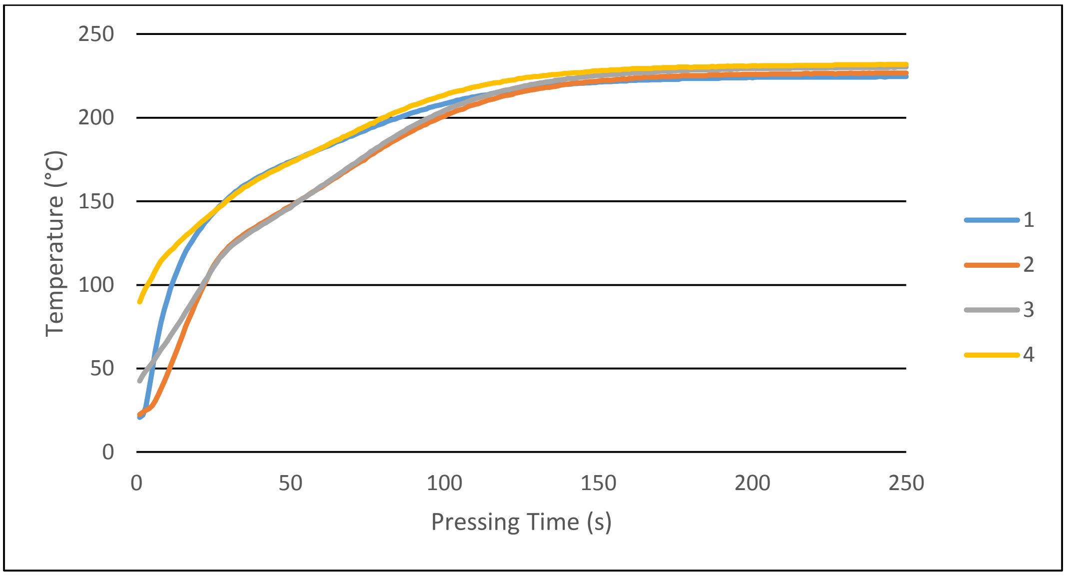


Figure 19. Heat evolution in bond line of parallel laminated 5-ply plywood. Numbers 1 - 4 represent the bond lines from top to bottom respectively.

The inner parts of plywood reach the plateau in approximately 150 s and this was the basis for choosing the pressing times for the plywood tests. Additionally, a similar shoulder can be seen at approximately 120 °C in the curves of the inner bond lines that has been reported by Cristescu et al. (2015a). The difference between outer and inner layers is probably caused by steam that has difficulties to escape inside the board and also mass loss of wood starts at approximately the same temperature (Cristescu 2015, Wannapeera et al. 2011). However, the curves of ABES samples in Figure 18 show a similar type of behavior, especially with pressing temperatures of 200 °C and 220 °C. 240 °C curve continues rising with the same slope. Hot pressing with ABES allows steam to evaporate quite freely because only two veneers are bonded, and further the small bonding area decreases the formation of steam compared to plywood. Therefore, there are signs that temperature growth at 120 °C could be affected by the prevailing temperature outside the veneer. Nonetheless, these are results of only one measurement and needs to be studied more thoroughly prior to jumping to conclusions.

4.4 Plywood Results

Results in this chapter are approximate and no statistical analysis is made due to experimental nature of testing. Parameters were chosen based on ABES testing in order to scale up and compare the results. All the veneers were peeled from log a that was soaked at 20 °C, since they yielded greater strength results in ABES tests.

A pressing time of 7.5 minutes seems to produce stronger bonds within plywood than a press time 10 minutes longer, as shown in Figure 20. Furthermore, 220 °C with 7.5 min was the only group that was able to form a lasting bond and showed no delamination or failure of the bonds prior to testing. However, all the failures in shear strength testing occurred along bond line not within the wood. Longer pressing time resulted in slight opening of the seams causing low shear strength properties. Using veneers with higher moisture content caused explosion when pressed at 220 °C. Therefore, the temperature was reduced to 210 °C and 2 minutes gradual release of the pressure (Table 1) was used. Gradual release was used in order to release the steam more slowly from inside the plywood so as to not cause explosion. Even though the explosion was avoided the actual bonding remained weak. With 7 ply plywood pressing time was elevated to 25 minutes in order to ensure a long enough time for heating up the inner bond lines, additionally gradual press release of 3 minutes was used.

All the plywood samples that were tested consisted of parallel laminated veneers. Cross laminated plywood delaminated almost immediately after the press was opened.

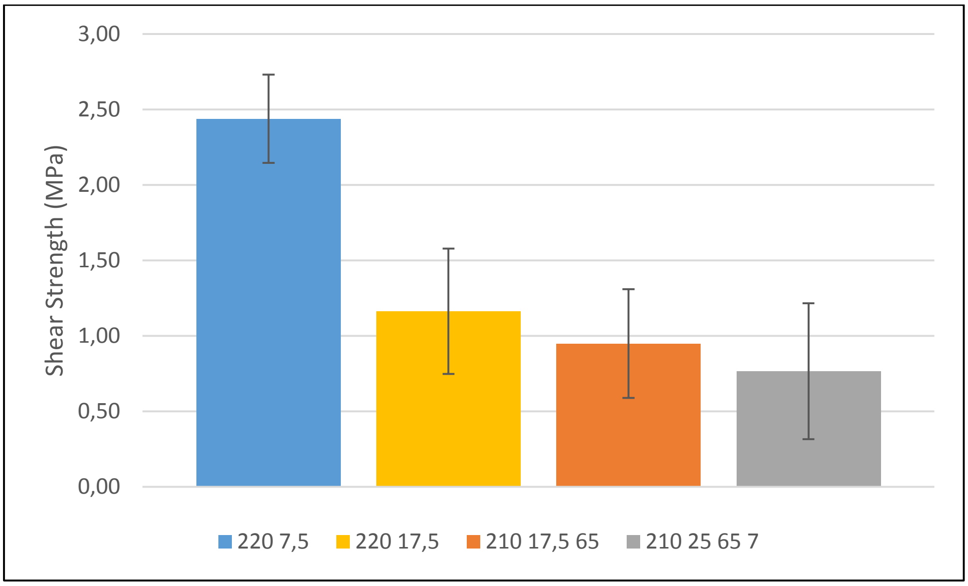


Figure 20. Shear strength of self-bonded, parallel laminated plywood. Process parameters: 5 MPa, 210 °C, 220 °C, 7.5 minutes, 17.5 minutes, 25 minutes. 65: veneers conditioned 65 % RH. 35 % RH conditioning is used if not mentioned. Last column is plywood manufactured from 7 veneers. Error bars are standard deviations. Number of the samples for each of the columns from left to right respectively is 13, 14, 6, 6.

Comparing the results in Figure 20 to the results from ABES testing revealed that the results are not fully comparable. ABES results suggest that a longer pressing time improves bonding. However, pressing 5 sheets of veneer together caused more delamination as pressing time was increased. Uneven distribution of heat inside the board may have influenced the results as the lower side of the board was nearly charred while the upper side appeared more evenly colored. Opening of the seams occurred within lower veneers.

Explosion of moister veneers and opening the seams indicate though that higher moisture content lowers the bonding ability of veneer in plywood as well. Additionally, long pressing time combined with high temperature may result in partial delamination of veneers with a moisture content of 6 %. Ruponen et al. (2015) also reported that the internal gas pressure rises towards the end of hot pressing causing delamination. Furthermore, the internal gas pressure of wood composites reportedly start rising rapidly after 500 s of hot pressing, also increase in the pressing temperature increases the gas pressure (Thomen 2000).

5 CONCLUSIONS

The effect of moisture content and log soaking temperature on the self-bonding properties of birch veneer were studied in this thesis using ABES. Based on the results there are statistically significant differences in the ability of veneers to form durable bonds under different conditions. When bonding veneers at 200 °C, only the drier veneers with log soaking temperature of 20 °C formed proper bonds. Veneers that were manufactured after soaking at 70 °C or conditioned at 65 % prior to hot-pressing delaminated instantly as the press opened.

The best combination of veneer properties in order to form a strong bond between two veneers without using any adhesive based on this study is approximately 6 % of moisture content and log soaking temperature of 20 °C. A higher moisture content appears to distract bond formation and cause too high a gas pressure within the plywood. A log soaking temperature of 70 °C significantly decreases the bonding ability of veneer in conditions where bonding is uncertain. As pressing conditions are more severe the effect of moisture content and log soaking temperature becomes more insignificant.

The moisture resistance of self-bonded veneer is enough to withstand a change in relative humidity from 35 % to 65 %. However, significant weakening of bonds was also discovered, which questions the ability of the bond to withstand more severe moisture conditions.

Pressing temperature was noted to be influential factor in the bond formation: hot pressing at 240 °C was enough to form stronger bond faster than pressing at 220 °C. Considering the internal gas pressure that may increase too high in plywood within longer pressing times, it might essential to be able to form the bond as quickly as possible. Additionally, the ABES results suggest that the bond strength of the veneer joint does not increase excessively after the certain level of bonding is attained. Therefore, based on the results of ABES and plywood testing shorter pressing time could produce relatively good bonding within the veneers of plywood if the pressing temperature is high enough to form a strong bond.

ABES proved to be a reliable method in order to evaluate mechanisms considering the bond formation of self-bonded veneer. Especially, it enables relatively accurate examination of the bonding properties of veneer. However, the wood material especially near bond line should be as uniform as possible within every sample in order to avoid distraction of unwanted properties, since irregularities can cause major error in the results because the small bond area emphasizes minor differences. Additionally, it is important considering self-bonding to ensure that the grain direction is exactly parallel within the bonded veneers.

Results considering parallel laminated plywood suggest that the results from ABES testing are not fully applicable at larger scale. As the bond area grows larger also the effect of internal gas pressure and other factors become increasingly significant. Therefore, for example optimization of pressing parameters with ABES considering large scale plywood is not accurate.

6 FURTHER RESEARCH

Because of the general nature of the thesis it was not possible to study reliably the effects of the factors independently nor evaluate the joint effect of the parameters. Therefore, a more detailed study of the effects would be useful.

Closer study on the chemical properties of self-bonded wood in order to understand better the mechanism should be conducted. For example the effects of proanthocyanidins and generated extractives are not fully known. Additionally, a proper understanding of the chemical changes in log soaking could aid comprehension of the self-bonding phenomenon.

High internal gas pressure when several layers of veneers are bonded quickly becomes too high and causes delamination. Methods to reduce gas pressure should be investigated.

Studying more extreme moisture conditions would be useful with ABES in order to find out how bonds can resist moist conditions. ABES would also be a convenient tool to determine an optimal moisture content for self-bonded veneer.

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APPENDIX 1

Table 1. ANOVA with Bonferroni correction. 60 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1,566	5	,313	2,800	,019
Within Groups	18,117	162	,112		
Total	19,683	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 220	B 220	-,32230*	,08938	,006	-,5886	-,0560
	D 220	-,19813	,08938	,420	-,4644	,0682
	M 220	-,12417	,08938	1,000	-,3904	,1421
	35 220	-,18608	,08938	,584	-,4524	,0802
	65 220	-,13622	,08938	1,000	-,4025	,1301
B 220	A 220	,32230*	,08938	,006	,0560	,5886
	D 220	,12417	,08938	1,000	-,1421	,3904
	M 220	,19813	,08938	,420	-,0682	,4644
	35 220	,13622	,08938	1,000	-,1301	,4025
	65 220	,18608	,08938	,584	-,0802	,4524
D 220	A 220	,19813	,08938	,420	-,0682	,4644
	B 220	-,12417	,08938	1,000	-,3904	,1421
	M 220	,07397	,08938	1,000	-,1923	,3403
	35 220	,01206	,08938	1,000	-,2542	,2783
	65 220	,06191	,08938	1,000	-,2044	,3282
M 220	A 220	,12417	,08938	1,000	-,1421	,3904
	B 220	-,19813	,08938	,420	-,4644	,0682
	D 220	-,07397	,08938	1,000	-,3403	,1923
	35 220	-,06191	,08938	1,000	-,3282	,2044
	65 220	-,01206	,08938	1,000	-,2783	,2542
35 220	A 220	,18608	,08938	,584	-,0802	,4524
	B 220	-,13622	,08938	1,000	-,4025	,1301

	D 220	-,01206	,08938	1,000	-,2783	,2542
	M 220	,06191	,08938	1,000	-,2044	,3282
	65 220	,04985	,08938	1,000	-,2164	,3161
65 220	A 220	,13622	,08938	1,000	-,1301	,4025
	B 220	-,18608	,08938	,584	-,4524	,0802
	D 220	-,06191	,08938	1,000	-,3282	,2044
	M 220	,01206	,08938	1,000	-,2542	,2783
	35 220	-,04985	,08938	1,000	-,3161	,2164

*. The mean difference is significant at the 0.05 level.

Table 2. ANOVA with Bonferroni correction. 120 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	12,831	5	2,566	5,455	,000
Within Groups	76,209	162	,470		
Total	89,040	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 220	B 220	-,71617*	,18331	,002	-1,2623	-,1700
	D 220	-,53045	,18331	,065	-1,0766	,0157
	M 220	-,18572	,18331	1,000	-,7319	,3604
	35 220	-,62490*	,18331	,012	-1,1710	-,0788
	65 220	-,09127	,18331	1,000	-,6374	,4549
B 220	A 220	,71617*	,18331	,002	,1700	1,2623
	D 220	,18572	,18331	1,000	-,3604	,7319
	M 220	,53045	,18331	,065	-,0157	1,0766
	35 220	,09127	,18331	1,000	-,4549	,6374
	65 220	,62490*	,18331	,012	,0788	1,1710
D 220	A 220	,53045	,18331	,065	-,0157	1,0766
	B 220	-,18572	,18331	1,000	-,7319	,3604

	M 220	,34472	,18331	,927	-,2014	,8909
	35 220	-,09446	,18331	1,000	-,6406	,4517
	65 220	,43918	,18331	,266	-,1070	,9853
M 220	A 220	,18572	,18331	1,000	-,3604	,7319
	B 220	-,53045	,18331	,065	-1,0766	,0157
	D 220	-,34472	,18331	,927	-,8909	,2014
	35 220	-,43918	,18331	,266	-,9853	,1070
	65 220	,09446	,18331	1,000	-,4517	,6406
35 220	A 220	,62490*	,18331	,012	,0788	1,1710
	B 220	-,09127	,18331	1,000	-,6374	,4549
	D 220	,09446	,18331	1,000	-,4517	,6406
	M 220	,43918	,18331	,266	-,1070	,9853
	65 220	,53363	,18331	,062	-,0125	1,0798
65 220	A 220	,09127	,18331	1,000	-,4549	,6374
	B 220	-,62490*	,18331	,012	-1,1710	-,0788
	D 220	-,43918	,18331	,266	-,9853	,1070
	M 220	-,09446	,18331	1,000	-,6406	,4517
	35 220	-,53363	,18331	,062	-1,0798	,0125

*. The mean difference is significant at the 0.05 level.

Table 3. ANOVA with Bonferroni correction. 180 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	15,759	5	3,152	5,289	,000
Within Groups	96,533	162	,596		
Total	112,291	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 220	B 220	-,96667*	,20631	,000	-1,5813	-,3520
	D 220	-,58973	,20631	,072	-1,2044	,0249
	M 220	-,37694	,20631	1,000	-,9916	,2377

	35 220	-,67431*	,20631	,020	-1,2890	-,0596
	65 220	-,29236	,20631	1,000	-,9070	,3223
B 220	A 220	,96667*	,20631	,000	,3520	1,5813
	D 220	,37694	,20631	1,000	-,2377	,9916
	M 220	,58973	,20631	,072	-,0249	1,2044
	35 220	,29236	,20631	1,000	-,3223	,9070
	65 220	,67431*	,20631	,020	,0596	1,2890
D 220	A 220	,58973	,20631	,072	-,0249	1,2044
	B 220	-,37694	,20631	1,000	-,9916	,2377
	M 220	,21279	,20631	1,000	-,4019	,8274
	35 220	-,08458	,20631	1,000	-,6992	,5301
	65 220	,29737	,20631	1,000	-,3173	,9120
M 220	A 220	,37694	,20631	1,000	-,2377	,9916
	B 220	-,58973	,20631	,072	-1,2044	,0249
	D 220	-,21279	,20631	1,000	-,8274	,4019
	35 220	-,29737	,20631	1,000	-,9120	,3173
	65 220	,08458	,20631	1,000	-,5301	,6992
35 220	A 220	,67431*	,20631	,020	,0596	1,2890
	B 220	-,29236	,20631	1,000	-,9070	,3223
	D 220	,08458	,20631	1,000	-,5301	,6992
	M 220	,29737	,20631	1,000	-,3173	,9120
	65 220	,38196	,20631	,989	-,2327	,9966
65 220	A 220	,29236	,20631	1,000	-,3223	,9070
	B 220	-,67431*	,20631	,020	-1,2890	-,0596
	D 220	-,29737	,20631	1,000	-,9120	,3173
	M 220	-,08458	,20631	1,000	-,6992	,5301
	35 220	-,38196	,20631	,989	-,9966	,2327

*. The mean difference is significant at the 0.05 level.

Table 4. ANOVA with Bonferroni correction. 240 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	12,779	5	2,556	4,019	,002
Within Groups	103,018	162	,636		
Total	115,797	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 220	B 220	-,80574*	,21313	,003	-1,4407	-,1708
	D 220	-,57864	,21313	,110	-1,2136	,0563
	M 220	-,22710	,21313	1,000	-,8621	,4079
	35 220	-,58992	,21313	,094	-1,2249	,0451
	65 220	-,21582	,21313	1,000	-,8508	,4192
B 220	A 220	,80574*	,21313	,003	,1708	1,4407
	D 220	,22710	,21313	1,000	-,4079	,8621
	M 220	,57864	,21313	,110	-,0563	1,2136
	35 220	,21582	,21313	1,000	-,4192	,8508
	65 220	,58992	,21313	,094	-,0451	1,2249
D 220	A 220	,57864	,21313	,110	-,0563	1,2136
	B 220	-,22710	,21313	1,000	-,8621	,4079
	M 220	,35154	,21313	1,000	-,2834	,9865
	35 220	-,01129	,21313	1,000	-,6463	,6237
	65 220	,36282	,21313	1,000	-,2722	,9978
M 220	A 220	,22710	,21313	1,000	-,4079	,8621
	B 220	-,57864	,21313	,110	-1,2136	,0563
	D 220	-,35154	,21313	1,000	-,9865	,2834
	35 220	-,36282	,21313	1,000	-,9978	,2722
	65 220	,01129	,21313	1,000	-,6237	,6463
35 220	A 220	,58992	,21313	,094	-,0451	1,2249
	B 220	-,21582	,21313	1,000	-,8508	,4192
	D 220	,01129	,21313	1,000	-,6237	,6463
	M 220	,36282	,21313	1,000	-,2722	,9978
	65 220	,37411	,21313	1,000	-,2609	1,0091
65 220	A 220	,21582	,21313	1,000	-,4192	,8508
	B 220	-,58992	,21313	,094	-1,2249	,0451
	D 220	-,36282	,21313	1,000	-,9978	,2722
	M 220	-,01129	,21313	1,000	-,6463	,6237
	35 220	-,37411	,21313	1,000	-1,0091	,2609

*. The mean difference is significant at the 0.05 level.

Table 5. ANOVA with Bonferroni correction. 300 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	17,928	5	3,586	7,355	,000
Within Groups	78,973	162	,487		
Total	96,901	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 220	B 220	-1,07042*	,18660	,000	-1,6264	-,5145
	D 220	-,63384*	,18660	,013	-1,1898	-,0779
	M 220	-,43658	,18660	,308	-,9925	,1194
	35 220	-,69001*	,18660	,004	-1,2460	-,1341
	65 220	-,38042	,18660	,647	-,9364	,1755
B 220	A 220	1,07042*	,18660	,000	,5145	1,6264
	D 220	,43658	,18660	,308	-,1194	,9925
	M 220	,63384*	,18660	,013	,0779	1,1898
	35 220	,38042	,18660	,647	-,1755	,9364
	65 220	,69001*	,18660	,004	,1341	1,2460
D 220	A 220	,63384*	,18660	,013	,0779	1,1898
	B 220	-,43658	,18660	,308	-,9925	,1194
	M 220	,19725	,18660	1,000	-,3587	,7532
	35 220	-,05617	,18660	1,000	-,6121	,4998
	65 220	,25342	,18660	1,000	-,3025	,8094
M 220	A 220	,43658	,18660	,308	-,1194	,9925
	B 220	-,63384*	,18660	,013	-1,1898	-,0779
	D 220	-,19725	,18660	1,000	-,7532	,3587
	35 220	-,25342	,18660	1,000	-,8094	,3025
	65 220	,05617	,18660	1,000	-,4998	,6121
35 220	A 220	,69001*	,18660	,004	,1341	1,2460
	B 220	-,38042	,18660	,647	-,9364	,1755
	D 220	,05617	,18660	1,000	-,4998	,6121

	M 220	,25342	,18660	1,000	-,3025	,8094
	65 220	,30959	,18660	1,000	-,2464	,8655
65 220	A 220	,38042	,18660	,647	-,1755	,9364
	B 220	-,69001*	,18660	,004	-1,2460	-,1341
	D 220	-,25342	,18660	1,000	-,8094	,3025
	M 220	-,05617	,18660	1,000	-,6121	,4998
	35 220	-,30959	,18660	1,000	-,8655	,2464

*. The mean difference is significant at the 0.05 level.

Table 6. ANOVA with Bonferroni correction. 360 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	19,680	5	3,936	6,643	,000
Within Groups	95,991	162	,593		
Total	115,670	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

		Mean Difference			95% Confidence Interval	
(I) Group	(J) Group	(I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 220	B 220	-,78108*	,20573	,003	-1,3940	-,1681
	D 220	-,80447*	,20573	,002	-1,4174	-,1915
	M 220	,02339	,20573	1,000	-,5895	,6363
	35 220	-,55656	,20573	,113	-1,1695	,0564
	65 220	-,22451	,20573	1,000	-,8375	,3884
B 220	A 220	,78108*	,20573	,003	,1681	1,3940
	D 220	-,02339	,20573	1,000	-,6363	,5895
	M 220	,80447*	,20573	,002	,1915	1,4174
	35 220	,22451	,20573	1,000	-,3884	,8375
	65 220	,55656	,20573	,113	-,0564	1,1695
D 220	A 220	,80447*	,20573	,002	,1915	1,4174
	B 220	,02339	,20573	1,000	-,5895	,6363
	M 220	,82786*	,20573	,001	,2149	1,4408

	35 220	,24791	,20573	1,000	-,3650	,8608
	65 220	,57996	,20573	,081	-,0330	1,1929
M 220	A 220	-,02339	,20573	1,000	-,6363	,5895
	B 220	-,80447*	,20573	,002	-1,4174	-,1915
	D 220	-,82786*	,20573	,001	-1,4408	-,2149
	35 220	-,57996	,20573	,081	-1,1929	,0330
	65 220	-,24791	,20573	1,000	-,8608	,3650
35 220	A 220	,55656	,20573	,113	-,0564	1,1695
	B 220	-,22451	,20573	1,000	-,8375	,3884
	D 220	-,24791	,20573	1,000	-,8608	,3650
	M 220	,57996	,20573	,081	-,0330	1,1929
	65 220	,33205	,20573	1,000	-,2809	,9450
65 220	A 220	,22451	,20573	1,000	-,3884	,8375
	B 220	-,55656	,20573	,113	-1,1695	,0564
	D 220	-,57996	,20573	,081	-1,1929	,0330
	M 220	,24791	,20573	1,000	-,3650	,8608
	35 220	-,33205	,20573	1,000	-,9450	,2809

*. The mean difference is significant at the 0.05 level.

Table 7. ANOVA with Bonferroni correction. 420 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	14,860	5	2,972	4,792	,000
Within Groups	100,474	162	,620		
Total	115,334	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

		Mean Difference			95% Confidence Interval	
(I) Group	(J) Group	(I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 220	B 220	-,41800	,21048	,731	-1,0451	,2091
	D 220	-,63818*	,21048	,042	-1,2653	-,0111
	M 220	,22018	,21048	1,000	-,4069	,8473

	35 220	-,40261	,21048	,863	-1,0297	,2245
	65 220	-,01539	,21048	1,000	-,6425	,6117
B 220	A 220	,41800	,21048	,731	-,2091	1,0451
	D 220	-,22018	,21048	1,000	-,8473	,4069
	M 220	,63818*	,21048	,042	,0111	1,2653
	35 220	,01539	,21048	1,000	-,6117	,6425
	65 220	,40261	,21048	,863	-,2245	1,0297
D 220	A 220	,63818*	,21048	,042	,0111	1,2653
	B 220	,22018	,21048	1,000	-,4069	,8473
	M 220	,85836*	,21048	,001	,2313	1,4854
	35 220	,23557	,21048	1,000	-,3915	,8627
	65 220	,62279	,21048	,053	-,0043	1,2499
M 220	A 220	-,22018	,21048	1,000	-,8473	,4069
	B 220	-,63818*	,21048	,042	-1,2653	-,0111
	D 220	-,85836*	,21048	,001	-1,4854	-,2313
	35 220	-,62279	,21048	,053	-1,2499	,0043
	65 220	-,23557	,21048	1,000	-,8627	,3915
35 220	A 220	,40261	,21048	,863	-,2245	1,0297
	B 220	-,01539	,21048	1,000	-,6425	,6117
	D 220	-,23557	,21048	1,000	-,8627	,3915
	M 220	,62279	,21048	,053	-,0043	1,2499
	65 220	,38722	,21048	1,000	-,2399	1,0143
65 220	A 220	,01539	,21048	1,000	-,6117	,6425
	B 220	-,40261	,21048	,863	-1,0297	,2245
	D 220	-,62279	,21048	,053	-1,2499	,0043
	M 220	,23557	,21048	1,000	-,3915	,8627
	35 220	-,38722	,21048	1,000	-1,0143	,2399

*. The mean difference is significant at the 0.05 level.

Table 8. ANOVA with Bonferroni correction. 480 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	13,350	5	2,670	4,926	,000
Within Groups	87,814	162	,542		
Total	101,165	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 220	B 220	-,14259	,19677	1,000	-,7288	,4437
	D 220	-,50335	,19677	,172	-1,0896	,0829
	M 220	,36076	,19677	1,000	-,2255	,9470
	35 220	-,28728	,19677	1,000	-,8735	,2990
	65 220	,14469	,19677	1,000	-,4416	,7309
B 220	A 220	,14259	,19677	1,000	-,4437	,7288
	D 220	-,36076	,19677	1,000	-,9470	,2255
	M 220	,50335	,19677	,172	-,0829	1,0896
	35 220	-,14469	,19677	1,000	-,7309	,4416
	65 220	,28728	,19677	1,000	-,2990	,8735
D 220	A 220	,50335	,19677	,172	-,0829	1,0896
	B 220	,36076	,19677	1,000	-,2255	,9470
	M 220	,86411*	,19677	,000	,2779	1,4504
	35 220	,21607	,19677	1,000	-,3702	,8023
	65 220	,64804*	,19677	,018	,0618	1,2343
M 220	A 220	-,36076	,19677	1,000	-,9470	,2255
	B 220	-,50335	,19677	,172	-1,0896	,0829
	D 220	-,86411*	,19677	,000	-1,4504	-,2779
	35 220	-,64804*	,19677	,018	-1,2343	-,0618
	65 220	-,21607	,19677	1,000	-,8023	,3702
35 220	A 220	,28728	,19677	1,000	-,2990	,8735
	B 220	,14469	,19677	1,000	-,4416	,7309
	D 220	-,21607	,19677	1,000	-,8023	,3702
	M 220	,64804*	,19677	,018	,0618	1,2343
	65 220	,43196	,19677	,444	-,1543	1,0182
65 220	A 220	-,14469	,19677	1,000	-,7309	,4416
	B 220	-,28728	,19677	1,000	-,8735	,2990
	D 220	-,64804*	,19677	,018	-1,2343	-,0618
	M 220	,21607	,19677	1,000	-,3702	,8023
	35 220	-,43196	,19677	,444	-1,0182	,1543

*. The mean difference is significant at the 0.05 level.

Table 9. ANOVA with Bonferroni correction. 540 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	11,971	5	2,394	5,418	,000
Within Groups	71,584	162	,442		
Total	83,555	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

		Mean Difference			95% Confidence Interval	
(I) Group	(J) Group	(I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 220	B 220	-,43487	,17766	,232	-,9642	,0944
	D 220	-,60058*	,17766	,014	-1,1299	-,0713
	M 220	,16571	,17766	1,000	-,3636	,6950
	35 220	-,35772	,17766	,686	-,8870	,1716
	65 220	-,07715	,17766	1,000	-,6065	,4522
B 220	A 220	,43487	,17766	,232	-,0944	,9642
	D 220	-,16571	,17766	1,000	-,6950	,3636
	M 220	,60058*	,17766	,014	,0713	1,1299
	35 220	,07715	,17766	1,000	-,4522	,6065
	65 220	,35772	,17766	,686	-,1716	,8870
D 220	A 220	,60058*	,17766	,014	,0713	1,1299
	B 220	,16571	,17766	1,000	-,3636	,6950
	M 220	,76630*	,17766	,000	,2370	1,2956
	35 220	,24286	,17766	1,000	-,2864	,7722
	65 220	,52344	,17766	,055	-,0059	1,0527
M 220	A 220	-,16571	,17766	1,000	-,6950	,3636
	B 220	-,60058*	,17766	,014	-1,1299	-,0713
	D 220	-,76630*	,17766	,000	-1,2956	-,2370
	35 220	-,52344	,17766	,055	-1,0527	,0059
	65 220	-,24286	,17766	1,000	-,7722	,2864
35 220	A 220	,35772	,17766	,686	-,1716	,8870
	B 220	-,07715	,17766	1,000	-,6065	,4522
	D 220	-,24286	,17766	1,000	-,7722	,2864

	M 220	,52344	,17766	,055	-,0059	1,0527
	65 220	,28058	,17766	1,000	-,2487	,8099
65 220	A 220	,07715	,17766	1,000	-,4522	,6065
	B 220	-,35772	,17766	,686	-,8870	,1716
	D 220	-,52344	,17766	,055	-1,0527	,0059
	M 220	,24286	,17766	1,000	-,2864	,7722
	35 220	-,28058	,17766	1,000	-,8099	,2487

*. The mean difference is significant at the 0.05 level.

Table 10. ANOVA with Bonferroni correction. 600 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	11,333	5	2,267	4,464	,001
Within Groups	82,246	162	,508		
Total	93,579	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
A 220	B 220	-,43012	,19043	,379	-,9975	,1372
	D 220	-,59230*	,19043	,033	-1,1597	-,0249
	M 220	,16219	,19043	1,000	-,4052	,7295
	35 220	-,33257	,19043	1,000	-,8999	,2348
	65 220	-,09754	,19043	1,000	-,6649	,4698
B 220	A 220	,43012	,19043	,379	-,1372	,9975
	D 220	-,16219	,19043	1,000	-,7295	,4052
	M 220	,59230*	,19043	,033	,0249	1,1597
	35 220	,09754	,19043	1,000	-,4698	,6649
	65 220	,33257	,19043	1,000	-,2348	,8999
D 220	A 220	,59230*	,19043	,033	,0249	1,1597
	B 220	,16219	,19043	1,000	-,4052	,7295
	M 220	,75449*	,19043	,002	,1871	1,3219

	35 220	,25973	,19043	1,000	-,3076	,8271
	65 220	,49476	,19043	,154	-,0726	1,0621
M 220	A 220	-,16219	,19043	1,000	-,7295	,4052
	B 220	-,59230*	,19043	,033	-1,1597	-,0249
	D 220	-,75449*	,19043	,002	-1,3219	-,1871
	35 220	-,49476	,19043	,154	-1,0621	,0726
	65 220	-,25973	,19043	1,000	-,8271	,3076
35 220	A 220	,33257	,19043	1,000	-,2348	,8999
	B 220	-,09754	,19043	1,000	-,6649	,4698
	D 220	-,25973	,19043	1,000	-,8271	,3076
	M 220	,49476	,19043	,154	-,0726	1,0621
	65 220	,23503	,19043	1,000	-,3323	,8024
65 220	A 220	,09754	,19043	1,000	-,4698	,6649
	B 220	-,33257	,19043	1,000	-,8999	,2348
	D 220	-,49476	,19043	,154	-1,0621	,0726
	M 220	,25973	,19043	1,000	-,3076	,8271
	35 220	-,23503	,19043	1,000	-,8024	,3323

*. The mean difference is significant at the 0.05 level.

Table 11. ANOVA with Bonferroni correction. 660 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	2,475	5	,495	1,527	,184
Within Groups	52,518	162	,324		
Total	54,994	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

		Mean Difference			95% Confidence Interval	
(I) Group	(J) Group	(I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 220	B 220	-,23777	,15217	1,000	-,6911	,2156
	D 220	-,27299	,15217	1,000	-,7264	,1804
	M 220	,03522	,15217	1,000	-,4182	,4886

	35 220	-,19839	,15217	1,000	-,6518	,2550
	65 220	-,03938	,15217	1,000	-,4927	,4140
B 220	A 220	,23777	,15217	1,000	-,2156	,6911
	D 220	-,03522	,15217	1,000	-,4886	,4182
	M 220	,27299	,15217	1,000	-,1804	,7264
	35 220	,03938	,15217	1,000	-,4140	,4927
	65 220	,19839	,15217	1,000	-,2550	,6518
D 220	A 220	,27299	,15217	1,000	-,1804	,7264
	B 220	,03522	,15217	1,000	-,4182	,4886
	M 220	,30821	,15217	,667	-,1452	,7616
	35 220	,07460	,15217	1,000	-,3788	,5280
	65 220	,23362	,15217	1,000	-,2198	,6870
M 220	A 220	-,03522	,15217	1,000	-,4886	,4182
	B 220	-,27299	,15217	1,000	-,7264	,1804
	D 220	-,30821	,15217	,667	-,7616	,1452
	35 220	-,23362	,15217	1,000	-,6870	,2198
	65 220	-,07460	,15217	1,000	-,5280	,3788
35 220	A 220	,19839	,15217	1,000	-,2550	,6518
	B 220	-,03938	,15217	1,000	-,4927	,4140
	D 220	-,07460	,15217	1,000	-,5280	,3788
	M 220	,23362	,15217	1,000	-,2198	,6870
	65 220	,15902	,15217	1,000	-,2944	,6124
65 220	A 220	,03938	,15217	1,000	-,4140	,4927
	B 220	-,19839	,15217	1,000	-,6518	,2550
	D 220	-,23362	,15217	1,000	-,6870	,2198
	M 220	,07460	,15217	1,000	-,3788	,5280
	35 220	-,15902	,15217	1,000	-,6124	,2944

Table 12. ANOVA with Bonferroni correction. 720 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1,522	5	,304	,826	,533
Within Groups	59,711	162	,369		
Total	61,233	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 220	B 220	-,05313	,16226	1,000	-,5366	,4303
	D 220	-,14750	,16226	1,000	-,6309	,3359
	M 220	,09437	,16226	1,000	-,3891	,5778
	35 220	-,13545	,16226	1,000	-,6189	,3480
	65 220	,08232	,16226	1,000	-,4011	,5657
B 220	A 220	,05313	,16226	1,000	-,4303	,5366
	D 220	-,09437	,16226	1,000	-,5778	,3891
	M 220	,14750	,16226	1,000	-,3359	,6309
	35 220	-,08232	,16226	1,000	-,5657	,4011
	65 220	,13545	,16226	1,000	-,3480	,6189
D 220	A 220	,14750	,16226	1,000	-,3359	,6309
	B 220	,09437	,16226	1,000	-,3891	,5778
	M 220	,24187	,16226	1,000	-,2416	,7253
	35 220	,01205	,16226	1,000	-,4714	,4955
	65 220	,22982	,16226	1,000	-,2536	,7132
M 220	A 220	-,09437	,16226	1,000	-,5778	,3891
	B 220	-,14750	,16226	1,000	-,6309	,3359
	D 220	-,24187	,16226	1,000	-,7253	,2416
	35 220	-,22982	,16226	1,000	-,7132	,2536
	65 220	-,01205	,16226	1,000	-,4955	,4714
35 220	A 220	,13545	,16226	1,000	-,3480	,6189
	B 220	,08232	,16226	1,000	-,4011	,5657
	D 220	-,01205	,16226	1,000	-,4955	,4714
	M 220	,22982	,16226	1,000	-,2536	,7132
	65 220	,21776	,16226	1,000	-,2657	,7012
65 220	A 220	-,08232	,16226	1,000	-,5657	,4011
	B 220	-,13545	,16226	1,000	-,6189	,3480
	D 220	-,22982	,16226	1,000	-,7132	,2536
	M 220	,01205	,16226	1,000	-,4714	,4955
	35 220	-,21776	,16226	1,000	-,7012	,2657

Table 13. ANOVA with Bonferroni correction. 780 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	2,702	5	,540	2,188	,058
Within Groups	40,009	162	,247		
Total	42,711	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

		Mean Difference			95% Confidence Interval	
(I) Group	(J) Group	(I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 220	B 220	,04509	,13282	1,000	-,3506	,4408
	D 220	-,16594	,13282	1,000	-,5617	,2298
	M 220	,21103	,13282	1,000	-,1847	,6067
	35 220	-,08799	,13282	1,000	-,4837	,3077
	65 220	,13308	,13282	1,000	-,2626	,5288
B 220	A 220	-,04509	,13282	1,000	-,4408	,3506
	D 220	-,21103	,13282	1,000	-,6067	,1847
	M 220	,16594	,13282	1,000	-,2298	,5617
	35 220	-,13308	,13282	1,000	-,5288	,2626
	65 220	,08799	,13282	1,000	-,3077	,4837
D 220	A 220	,16594	,13282	1,000	-,2298	,5617
	B 220	,21103	,13282	1,000	-,1847	,6067
	M 220	,37696	,13282	,077	-,0187	,7727
	35 220	,07795	,13282	1,000	-,3178	,4737
	65 220	,29902	,13282	,386	-,0967	,6947
M 220	A 220	-,21103	,13282	1,000	-,6067	,1847
	B 220	-,16594	,13282	1,000	-,5617	,2298
	D 220	-,37696	,13282	,077	-,7727	,0187
	35 220	-,29902	,13282	,386	-,6947	,0967
	65 220	-,07795	,13282	1,000	-,4737	,3178
35 220	A 220	,08799	,13282	1,000	-,3077	,4837
	B 220	,13308	,13282	1,000	-,2626	,5288
	D 220	-,07795	,13282	1,000	-,4737	,3178
	M 220	,29902	,13282	,386	-,0967	,6947

	65 220	,22107	,13282	1,000	-,1746	,6168
65 220	A 220	-,13308	,13282	1,000	-,5288	,2626
	B 220	-,08799	,13282	1,000	-,4837	,3077
	D 220	-,29902	,13282	,386	-,6947	,0967
	M 220	,07795	,13282	1,000	-,3178	,4737
	35 220	-,22107	,13282	1,000	-,6168	,1746

Table 14. ANOVA with Bonferroni correction. 840 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	3,238	5	,648	2,325	,045
Within Groups	45,124	162	,279		
Total	48,362	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

					95% Confidence Interval	
(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 220	B 220	-,18326	,14105	1,000	-,6035	,2370
	D 220	-,26379	,14105	,949	-,6840	,1565
	M 220	,08054	,14105	1,000	-,3397	,5008
	35 220	-,23228	,14105	1,000	-,6525	,1880
	65 220	,04902	,14105	1,000	-,3712	,4693
B 220	A 220	,18326	,14105	1,000	-,2370	,6035
	D 220	-,08054	,14105	1,000	-,5008	,3397
	M 220	,26379	,14105	,949	-,1565	,6840
	35 220	-,04902	,14105	1,000	-,4693	,3712
	65 220	,23228	,14105	1,000	-,1880	,6525
D 220	A 220	,26379	,14105	,949	-,1565	,6840
	B 220	,08054	,14105	1,000	-,3397	,5008
	M 220	,34433	,14105	,236	-,0759	,7646
	35 220	,03152	,14105	1,000	-,3887	,4518
	65 220	,31281	,14105	,420	-,1074	,7331

M 220	A 220	-,08054	,14105	1,000	-,5008	,3397
	B 220	-,26379	,14105	,949	-,6840	,1565
	D 220	-,34433	,14105	,236	-,7646	,0759
	35 220	-,31281	,14105	,420	-,7331	,1074
	65 220	-,03152	,14105	1,000	-,4518	,3887
35 220	A 220	,23228	,14105	1,000	-,1880	,6525
	B 220	,04902	,14105	1,000	-,3712	,4693
	D 220	-,03152	,14105	1,000	-,4518	,3887
	M 220	,31281	,14105	,420	-,1074	,7331
	65 220	,28129	,14105	,717	-,1390	,7015
65 220	A 220	-,04902	,14105	1,000	-,4693	,3712
	B 220	-,23228	,14105	1,000	-,6525	,1880
	D 220	-,31281	,14105	,420	-,7331	,1074
	M 220	,03152	,14105	1,000	-,3887	,4518
	35 220	-,28129	,14105	,717	-,7015	,1390

Table 15. ANOVA with Bonferroni correction. 900 s, 220 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1,139	5	,228	1,294	,269
Within Groups	28,522	162	,176		
Total	29,662	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

					95% Confidence Interval	
(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 220	B 220	,06643	,11214	1,000	-,2677	,4005
	D 220	-,05786	,11214	1,000	-,3920	,2763
	M 220	,12429	,11214	1,000	-,2098	,4584
	35 220	-,07143	,11214	1,000	-,4055	,2627
	65 220	,13786	,11214	1,000	-,1963	,4720
B 220	A 220	-,06643	,11214	1,000	-,4005	,2677

	D 220	-,12429	,11214	1,000	-,4584	,2098
	M 220	,05786	,11214	1,000	-,2763	,3920
	35 220	-,13786	,11214	1,000	-,4720	,1963
	65 220	,07143	,11214	1,000	-,2627	,4055
D 220	A 220	,05786	,11214	1,000	-,2763	,3920
	B 220	,12429	,11214	1,000	-,2098	,4584
	M 220	,18214	,11214	1,000	-,1520	,5163
	35 220	-,01357	,11214	1,000	-,3477	,3205
	65 220	,19571	,11214	1,000	-,1384	,5298
M 220	A 220	-,12429	,11214	1,000	-,4584	,2098
	B 220	-,05786	,11214	1,000	-,3920	,2763
	D 220	-,18214	,11214	1,000	-,5163	,1520
	35 220	-,19571	,11214	1,000	-,5298	,1384
	65 220	,01357	,11214	1,000	-,3205	,3477
35 220	A 220	,07143	,11214	1,000	-,2627	,4055
	B 220	,13786	,11214	1,000	-,1963	,4720
	D 220	,01357	,11214	1,000	-,3205	,3477
	M 220	,19571	,11214	1,000	-,1384	,5298
	65 220	,20929	,11214	,957	-,1248	,5434
65 220	A 220	-,13786	,11214	1,000	-,4720	,1963
	B 220	-,07143	,11214	1,000	-,4055	,2627
	D 220	-,19571	,11214	1,000	-,5298	,1384
	M 220	-,01357	,11214	1,000	-,3477	,3205
	35 220	-,20929	,11214	,957	-,5434	,1248

APPENDIX 2

Table 1. ANOVA with Bonferroni correction. 60 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	12,103	5	2,421	5,763	,000
Within Groups	68,048	162	,420		
Total	80,151	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 240	B 240	-,60060*	,17321	,010	-1,1167	-,0845
	D 240	-,48291	,17321	,089	-,9990	,0332
	M 240	-,11769	,17321	1,000	-,6338	,3984
	35 240	-,60461*	,17321	,009	-1,1207	-,0885
	65 240	,00401	,17321	1,000	-,5121	,5201
B 240	A 240	,60060*	,17321	,010	,0845	1,1167
	D 240	,11769	,17321	1,000	-,3984	,6338
	M 240	,48291	,17321	,089	-,0332	,9990
	35 240	-,00401	,17321	1,000	-,5201	,5121
	65 240	,60461*	,17321	,009	,0885	1,1207
D 240	A 240	,48291	,17321	,089	-,0332	,9990
	B 240	-,11769	,17321	1,000	-,6338	,3984
	M 240	,36522	,17321	,548	-,1508	,8813
	35 240	-,12170	,17321	1,000	-,6378	,3944
	65 240	,48692	,17321	,083	-,0291	1,0030
M 240	A 240	,11769	,17321	1,000	-,3984	,6338
	B 240	-,48291	,17321	,089	-,9990	,0332
	D 240	-,36522	,17321	,548	-,8813	,1508
	35 240	-,48692	,17321	,083	-1,0030	,0291
	65 240	,12170	,17321	1,000	-,3944	,6378
35 240	A 240	,60461*	,17321	,009	,0885	1,1207

	B 240	,00401	,17321	1,000	-,5121	,5201
	D 240	,12170	,17321	1,000	-,3944	,6378
	M 240	,48692	,17321	,083	-,0291	1,0030
	65 240	,60863*	,17321	,009	,0926	1,1247
65 240	A 240	-,00401	,17321	1,000	-,5201	,5121
	B 240	-,60461*	,17321	,009	-1,1207	-,0885
	D 240	-,48692	,17321	,083	-1,0030	,0291
	M 240	-,12170	,17321	1,000	-,6378	,3944
	35 240	-,60863*	,17321	,009	-1,1247	-,0926

*. The mean difference is significant at the 0.05 level.

Table 2. ANOVA with Bonferroni correction. 120 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	6,657	5	1,331	6,746	,000
Within Groups	31,968	162	,197		
Total	38,625	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

		Mean Difference			95% Confidence Interval	
(I) Group	(J) Group	(I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 240	B 240	-,26522	,11872	,403	-,6189	,0885
	D 240	-,27790	,11872	,307	-,6316	,0758
	M 240	,01268	,11872	1,000	-,3410	,3664
	35 240	-,41576*	,11872	,009	-,7695	-,0620
	65 240	,15054	,11872	1,000	-,2032	,5043
B 240	A 240	,26522	,11872	,403	-,0885	,6189
	D 240	-,01268	,11872	1,000	-,3664	,3410
	M 240	,27790	,11872	,307	-,0758	,6316
	35 240	-,15054	,11872	1,000	-,5043	,2032
	65 240	,41576*	,11872	,009	,0620	,7695
D 240	A 240	,27790	,11872	,307	-,0758	,6316

	B 240	,01268	,11872	1,000	-,3410	,3664
	M 240	,29058	,11872	,232	-,0631	,6443
	35 240	-,13786	,11872	1,000	-,4916	,2159
	65 240	,42844*	,11872	,006	,0747	,7822
M 240	A 240	-,01268	,11872	1,000	-,3664	,3410
	B 240	-,27790	,11872	,307	-,6316	,0758
	D 240	-,29058	,11872	,232	-,6443	,0631
	35 240	-,42844*	,11872	,006	-,7822	-,0747
	65 240	,13786	,11872	1,000	-,2159	,4916
35 240	A 240	,41576*	,11872	,009	,0620	,7695
	B 240	,15054	,11872	1,000	-,2032	,5043
	D 240	,13786	,11872	1,000	-,2159	,4916
	M 240	,42844*	,11872	,006	,0747	,7822
	65 240	,56629*	,11872	,000	,2126	,9200
65 240	A 240	-,15054	,11872	1,000	-,5043	,2032
	B 240	-,41576*	,11872	,009	-,7695	-,0620
	D 240	-,42844*	,11872	,006	-,7822	-,0747
	M 240	-,13786	,11872	1,000	-,4916	,2159
	35 240	-,56629*	,11872	,000	-,9200	-,2126

*. The mean difference is significant at the 0.05 level.

Table 3. ANOVA with Bonferroni correction. 180 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	9,145	5	1,829	8,359	,000
Within Groups	35,444	162	,219		
Total	44,589	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

					95% Confidence Interval	
(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 240	B 240	-,42647*	,12501	,012	-,7989	-,0540
	D 240	-,40353*	,12501	,023	-,7760	-,0311

	M 240	-,02295	,12501	1,000	-,3954	,3495
	35 240	-,49893*	,12501	,001	-,8714	-,1265
	65 240	,07246	,12501	1,000	-,3000	,4449
B 240	A 240	,42647*	,12501	,012	,0540	,7989
	D 240	,02295	,12501	1,000	-,3495	,3954
	M 240	,40353*	,12501	,023	,0311	,7760
	35 240	-,07246	,12501	1,000	-,4449	,3000
	65 240	,49893*	,12501	,001	,1265	,8714
D 240	A 240	,40353*	,12501	,023	,0311	,7760
	B 240	-,02295	,12501	1,000	-,3954	,3495
	M 240	,38058*	,12501	,041	,0081	,7530
	35 240	-,09540	,12501	1,000	-,4679	,2771
	65 240	,47598*	,12501	,003	,1035	,8484
M 240	A 240	,02295	,12501	1,000	-,3495	,3954
	B 240	-,40353*	,12501	,023	-,7760	-,0311
	D 240	-,38058*	,12501	,041	-,7530	-,0081
	35 240	-,47598*	,12501	,003	-,8484	-,1035
	65 240	,09540	,12501	1,000	-,2771	,4679
35 240	A 240	,49893*	,12501	,001	,1265	,8714
	B 240	,07246	,12501	1,000	-,3000	,4449
	D 240	,09540	,12501	1,000	-,2771	,4679
	M 240	,47598*	,12501	,003	,1035	,8484
	65 240	,57138*	,12501	,000	,1989	,9438
65 240	A 240	-,07246	,12501	1,000	-,4449	,3000
	B 240	-,49893*	,12501	,001	-,8714	-,1265
	D 240	-,47598*	,12501	,003	-,8484	-,1035
	M 240	-,09540	,12501	1,000	-,4679	,2771
	35 240	-,57138*	,12501	,000	-,9438	-,1989

*. The mean difference is significant at the 0.05 level.

Table 4. ANOVA with Bonferroni correction. 240 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	6,245	5	1,249	4,874	,000
Within Groups	41,512	162	,256		
Total	47,757	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 240	B 240	-,43330*	,13529	,025	-,8364	-,0302
	D 240	-,34563	,13529	,173	-,7487	,0575
	M 240	-,08768	,13529	1,000	-,4908	,3154
	35 240	-,43563*	,13529	,023	-,8387	-,0325
	65 240	,00232	,13529	1,000	-,4008	,4054
B 240	A 240	,43330*	,13529	,025	,0302	,8364
	D 240	,08768	,13529	1,000	-,3154	,4908
	M 240	,34563	,13529	,173	-,0575	,7487
	35 240	-,00232	,13529	1,000	-,4054	,4008
	65 240	,43563*	,13529	,023	,0325	,8387
D 240	A 240	,34563	,13529	,173	-,0575	,7487
	B 240	-,08768	,13529	1,000	-,4908	,3154
	M 240	,25795	,13529	,875	-,1451	,6610
	35 240	-,09000	,13529	1,000	-,4931	,3131
	65 240	,34795	,13529	,165	-,0551	,7510
M 240	A 240	,08768	,13529	1,000	-,3154	,4908
	B 240	-,34563	,13529	,173	-,7487	,0575
	D 240	-,25795	,13529	,875	-,6610	,1451
	35 240	-,34795	,13529	,165	-,7510	,0551
	65 240	,09000	,13529	1,000	-,3131	,4931
35 240	A 240	,43563*	,13529	,023	,0325	,8387
	B 240	,00232	,13529	1,000	-,4008	,4054
	D 240	,09000	,13529	1,000	-,3131	,4931
	M 240	,34795	,13529	,165	-,0551	,7510
	65 240	,43795*	,13529	,022	,0349	,8410
65 240	A 240	-,00232	,13529	1,000	-,4054	,4008
	B 240	-,43563*	,13529	,023	-,8387	-,0325
	D 240	-,34795	,13529	,165	-,7510	,0551
	M 240	-,09000	,13529	1,000	-,4931	,3131
	35 240	-,43795*	,13529	,022	-,8410	-,0349

*. The mean difference is significant at the 0.05 level.

Table 5. ANOVA with Bonferroni correction. 300 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	4,882	5	,976	4,582	,001
Within Groups	34,523	162	,213		
Total	39,405	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

		Mean Difference			95% Confidence Interval	
(I) Group	(J) Group	(I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 240	B 240	-,46290*	,12338	,004	-,8305	-,0953
	D 240	-,34683	,12338	,083	-,7144	,0208
	M 240	-,11607	,12338	1,000	-,4837	,2515
	35 240	-,37393*	,12338	,043	-,7415	-,0063
	65 240	-,08897	,12338	1,000	-,4566	,2786
B 240	A 240	,46290*	,12338	,004	,0953	,8305
	D 240	,11607	,12338	1,000	-,2515	,4837
	M 240	,34683	,12338	,083	-,0208	,7144
	35 240	,08897	,12338	1,000	-,2786	,4566
	65 240	,37393*	,12338	,043	,0063	,7415
D 240	A 240	,34683	,12338	,083	-,0208	,7144
	B 240	-,11607	,12338	1,000	-,4837	,2515
	M 240	,23076	,12338	,949	-,1368	,5983
	35 240	-,02710	,12338	1,000	-,3947	,3405
	65 240	,25786	,12338	,573	-,1097	,6254
M 240	A 240	,11607	,12338	1,000	-,2515	,4837
	B 240	-,34683	,12338	,083	-,7144	,0208
	D 240	-,23076	,12338	,949	-,5983	,1368
	35 240	-,25786	,12338	,573	-,6254	,1097
	65 240	,02710	,12338	1,000	-,3405	,3947
35 240	A 240	,37393*	,12338	,043	,0063	,7415
	B 240	-,08897	,12338	1,000	-,4566	,2786

	D 240	,02710	,12338	1,000	-,3405	,3947
	M 240	,25786	,12338	,573	-,1097	,6254
	65 240	,28496	,12338	,333	-,0826	,6525
65 240	A 240	,08897	,12338	1,000	-,2786	,4566
	B 240	-,37393*	,12338	,043	-,7415	-,0063
	D 240	-,25786	,12338	,573	-,6254	,1097
	M 240	-,02710	,12338	1,000	-,3947	,3405
	35 240	-,28496	,12338	,333	-,6525	,0826

*. The mean difference is significant at the 0.05 level.

Table 6. ANOVA with Bonferroni correction. 360 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	2,101	5	,420	1,998	,082
Within Groups	34,070	162	,210		
Total	36,171	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 240	B 240	-,38656*	,12256	,029	-,7517	-,0214
	D 240	-,19107	,12256	1,000	-,5562	,1741
	M 240	-,19549	,12256	1,000	-,5607	,1697
	35 240	-,18089	,12256	1,000	-,5461	,1843
	65 240	-,20567	,12256	1,000	-,5708	,1595
B 240	A 240	,38656*	,12256	,029	,0214	,7517
	D 240	,19549	,12256	1,000	-,1697	,5607
	M 240	,19107	,12256	1,000	-,1741	,5562
	35 240	,20567	,12256	1,000	-,1595	,5708
	65 240	,18089	,12256	1,000	-,1843	,5461
D 240	A 240	,19107	,12256	1,000	-,1741	,5562
	B 240	-,19549	,12256	1,000	-,5607	,1697

	M 240	-,00442	,12256	1,000	-,3696	,3607
	35 240	,01018	,12256	1,000	-,3550	,3753
	65 240	-,01460	,12256	1,000	-,3798	,3506
M 240	A 240	,19549	,12256	1,000	-,1697	,5607
	B 240	-,19107	,12256	1,000	-,5562	,1741
	D 240	,00442	,12256	1,000	-,3607	,3696
	35 240	,01460	,12256	1,000	-,3506	,3798
	65 240	-,01018	,12256	1,000	-,3753	,3550
35 240	A 240	,18089	,12256	1,000	-,1843	,5461
	B 240	-,20567	,12256	1,000	-,5708	,1595
	D 240	-,01018	,12256	1,000	-,3753	,3550
	M 240	-,01460	,12256	1,000	-,3798	,3506
	65 240	-,02478	,12256	1,000	-,3899	,3404
65 240	A 240	,20567	,12256	1,000	-,1595	,5708
	B 240	-,18089	,12256	1,000	-,5461	,1843
	D 240	,01460	,12256	1,000	-,3506	,3798
	M 240	,01018	,12256	1,000	-,3550	,3753
	35 240	,02478	,12256	1,000	-,3404	,3899

*. The mean difference is significant at the 0.05 level.

Table 7. ANOVA with Bonferroni correction. 420 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	,508	5	,102	,414	,839
Within Groups	39,759	162	,245		
Total	40,267	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

					95% Confidence Interval	
(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 240	B 240	-,13893	,13240	1,000	-,5334	,2555
	D 240	-,10888	,13240	1,000	-,5034	,2856
	M 240	-,03004	,13240	1,000	-,4245	,3644

	35 240	-,12134	,13240	1,000	-,5158	,2731
	65 240	-,01759	,13240	1,000	-,4121	,3769
B 240	A 240	,13893	,13240	1,000	-,2555	,5334
	D 240	,03004	,13240	1,000	-,3644	,4245
	M 240	,10888	,13240	1,000	-,2856	,5034
	35 240	,01759	,13240	1,000	-,3769	,4121
	65 240	,12134	,13240	1,000	-,2731	,5158
D 240	A 240	,10888	,13240	1,000	-,2856	,5034
	B 240	-,03004	,13240	1,000	-,4245	,3644
	M 240	,07884	,13240	1,000	-,3156	,4733
	35 240	-,01246	,13240	1,000	-,4069	,3820
	65 240	,09129	,13240	1,000	-,3032	,4858
M 240	A 240	,03004	,13240	1,000	-,3644	,4245
	B 240	-,10888	,13240	1,000	-,5034	,2856
	D 240	-,07884	,13240	1,000	-,4733	,3156
	35 240	-,09129	,13240	1,000	-,4858	,3032
	65 240	,01246	,13240	1,000	-,3820	,4069
35 240	A 240	,12134	,13240	1,000	-,2731	,5158
	B 240	-,01759	,13240	1,000	-,4121	,3769
	D 240	,01246	,13240	1,000	-,3820	,4069
	M 240	,09129	,13240	1,000	-,3032	,4858
	65 240	,10375	,13240	1,000	-,2907	,4982
65 240	A 240	,01759	,13240	1,000	-,3769	,4121
	B 240	-,12134	,13240	1,000	-,5158	,2731
	D 240	-,09129	,13240	1,000	-,4858	,3032
	M 240	-,01246	,13240	1,000	-,4069	,3820
	35 240	-,10375	,13240	1,000	-,4982	,2907

Table 8. ANOVA with Bonferroni correction. 480 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1,840	5	,368	1,012	,412
Within Groups	58,930	162	,364		
Total	60,770	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 240	B 240	-,30951	,16119	,849	-,7898	,1707
	D 240	-,24509	,16119	1,000	-,7253	,2352
	M 240	-,06442	,16119	1,000	-,5447	,4158
	35 240	-,18223	,16119	1,000	-,6625	,2980
	65 240	-,12728	,16119	1,000	-,6075	,3530
B 240	A 240	,30951	,16119	,849	-,1707	,7898
	D 240	,06442	,16119	1,000	-,4158	,5447
	M 240	,24509	,16119	1,000	-,2352	,7253
	35 240	,12728	,16119	1,000	-,3530	,6075
	65 240	,18223	,16119	1,000	-,2980	,6625
D 240	A 240	,24509	,16119	1,000	-,2352	,7253
	B 240	-,06442	,16119	1,000	-,5447	,4158
	M 240	,18067	,16119	1,000	-,2996	,6609
	35 240	,06286	,16119	1,000	-,4174	,5431
	65 240	,11781	,16119	1,000	-,3624	,5981
M 240	A 240	,06442	,16119	1,000	-,4158	,5447
	B 240	-,24509	,16119	1,000	-,7253	,2352
	D 240	-,18067	,16119	1,000	-,6609	,2996
	35 240	-,11781	,16119	1,000	-,5981	,3624
	65 240	-,06286	,16119	1,000	-,5431	,4174
35 240	A 240	,18223	,16119	1,000	-,2980	,6625
	B 240	-,12728	,16119	1,000	-,6075	,3530
	D 240	-,06286	,16119	1,000	-,5431	,4174
	M 240	,11781	,16119	1,000	-,3624	,5981
	65 240	,05496	,16119	1,000	-,4253	,5352
65 240	A 240	,12728	,16119	1,000	-,3530	,6075
	B 240	-,18223	,16119	1,000	-,6625	,2980
	D 240	-,11781	,16119	1,000	-,5981	,3624
	M 240	,06286	,16119	1,000	-,4174	,5431
	35 240	-,05496	,16119	1,000	-,5352	,4253

Table 9. ANOVA with Bonferroni correction. 540 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	6,244	5	1,249	4,283	,001
Within Groups	47,235	162	,292		
Total	53,479	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
A 240	B 240	-,38777	,14431	,119	-,8177	,0422
	D 240	-,45112*	,14431	,032	-,8811	-,0212
	M 240	,06335	,14431	1,000	-,3666	,4933
	35 240	-,28187	,14431	,788	-,7118	,1481
	65 240	-,10589	,14431	1,000	-,5359	,3241
B 240	A 240	,38777	,14431	,119	-,0422	,8177
	D 240	-,06335	,14431	1,000	-,4933	,3666
	M 240	,45112*	,14431	,032	,0212	,8811
	35 240	,10589	,14431	1,000	-,3241	,5359
	65 240	,28187	,14431	,788	-,1481	,7118
D 240	A 240	,45112*	,14431	,032	,0212	,8811
	B 240	,06335	,14431	1,000	-,3666	,4933
	M 240	,51446*	,14431	,007	,0845	,9444
	35 240	,16924	,14431	1,000	-,2607	,5992
	65 240	,34522	,14431	,268	-,0847	,7752
M 240	A 240	-,06335	,14431	1,000	-,4933	,3666
	B 240	-,45112*	,14431	,032	-,8811	-,0212
	D 240	-,51446*	,14431	,007	-,9444	-,0845
	35 240	-,34522	,14431	,268	-,7752	,0847
	65 240	-,16924	,14431	1,000	-,5992	,2607
35 240	A 240	,28187	,14431	,788	-,1481	,7118
	B 240	-,10589	,14431	1,000	-,5359	,3241
	D 240	-,16924	,14431	1,000	-,5992	,2607
	M 240	,34522	,14431	,268	-,0847	,7752

	65 240	,17598	,14431	1,000	-,2540	,6059
65 240	A 240	,10589	,14431	1,000	-,3241	,5359
	B 240	-,28187	,14431	,788	-,7118	,1481
	D 240	-,34522	,14431	,268	-,7752	,0847
	M 240	,16924	,14431	1,000	-,2607	,5992
	35 240	-,17598	,14431	1,000	-,6059	,2540

*. The mean difference is significant at the 0.05 level.

Table 10. ANOVA with Bonferroni correction. 600 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	4,431	5	,886	3,937	,002
Within Groups	36,467	162	,225		
Total	40,897	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

					95% Confidence Interval	
(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 240	B 240	-,29875	,12680	,295	-,6765	,0790
	D 240	-,36196	,12680	,073	-,7398	,0158
	M 240	,06321	,12680	1,000	-,3146	,4410
	35 240	-,25714	,12680	,663	-,6349	,1206
	65 240	-,04161	,12680	1,000	-,4194	,3362
B 240	A 240	,29875	,12680	,295	-,0790	,6765
	D 240	-,06321	,12680	1,000	-,4410	,3146
	M 240	,36196	,12680	,073	-,0158	,7398
	35 240	,04161	,12680	1,000	-,3362	,4194
	65 240	,25714	,12680	,663	-,1206	,6349
D 240	A 240	,36196	,12680	,073	-,0158	,7398
	B 240	,06321	,12680	1,000	-,3146	,4410
	M 240	,42518*	,12680	,015	,0474	,8030
	35 240	,10482	,12680	1,000	-,2730	,4826

	65 240	,32036	,12680	,187	-,0574	,6981
M 240	A 240	-,06321	,12680	1,000	-,4410	,3146
	B 240	-,36196	,12680	,073	-,7398	,0158
	D 240	-,42518*	,12680	,015	-,8030	-,0474
	35 240	-,32036	,12680	,187	-,6981	,0574
	65 240	-,10482	,12680	1,000	-,4826	,2730
35 240	A 240	,25714	,12680	,663	-,1206	,6349
	B 240	-,04161	,12680	1,000	-,4194	,3362
	D 240	-,10482	,12680	1,000	-,4826	,2730
	M 240	,32036	,12680	,187	-,0574	,6981
	65 240	,21554	,12680	1,000	-,1623	,5933
65 240	A 240	,04161	,12680	1,000	-,3362	,4194
	B 240	-,25714	,12680	,663	-,6349	,1206
	D 240	-,32036	,12680	,187	-,6981	,0574
	M 240	,10482	,12680	1,000	-,2730	,4826
	35 240	-,21554	,12680	1,000	-,5933	,1623

*. The mean difference is significant at the 0.05 level.

Table 11. ANOVA with Bonferroni correction. 660 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	6,495	5	1,299	3,926	,002
Within Groups	53,594	162	,331		
Total	60,088	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

					95% Confidence Interval	
(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 240	B 240	-,31665	,15372	,615	-,7746	,1413
	D 240	-,45062	,15372	,058	-,9086	,0074
	M 240	,13397	,15372	1,000	-,3240	,5920
	35 240	-,23228	,15372	1,000	-,6903	,2257

	65 240	-,08437	,15372	1,000	-,5424	,3736
B 240	A 240	,31665	,15372	,615	-,1413	,7746
	D 240	-,13397	,15372	1,000	-,5920	,3240
	M 240	,45062	,15372	,058	-,0074	,9086
	35 240	,08437	,15372	1,000	-,3736	,5424
	65 240	,23228	,15372	1,000	-,2257	,6903
D 240	A 240	,45062	,15372	,058	-,0074	,9086
	B 240	,13397	,15372	1,000	-,3240	,5920
	M 240	,58460*	,15372	,003	,1266	1,0426
	35 240	,21835	,15372	1,000	-,2396	,6763
	65 240	,36625	,15372	,275	-,0917	,8242
M 240	A 240	-,13397	,15372	1,000	-,5920	,3240
	B 240	-,45062	,15372	,058	-,9086	,0074
	D 240	-,58460*	,15372	,003	-1,0426	-,1266
	35 240	-,36625	,15372	,275	-,8242	,0917
	65 240	-,21835	,15372	1,000	-,6763	,2396
35 240	A 240	,23228	,15372	1,000	-,2257	,6903
	B 240	-,08437	,15372	1,000	-,5424	,3736
	D 240	-,21835	,15372	1,000	-,6763	,2396
	M 240	,36625	,15372	,275	-,0917	,8242
	65 240	,14790	,15372	1,000	-,3101	,6059
65 240	A 240	,08437	,15372	1,000	-,3736	,5424
	B 240	-,23228	,15372	1,000	-,6903	,2257
	D 240	-,36625	,15372	,275	-,8242	,0917
	M 240	,21835	,15372	1,000	-,2396	,6763
	35 240	-,14790	,15372	1,000	-,6059	,3101

*. The mean difference is significant at the 0.05 level.

Table 12. ANOVA with Bonferroni correction. 720 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	11,380	5	2,276	4,744	,000
Within Groups	77,725	162	,480		
Total	89,105	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 240	B 240	-,54478	,18512	,056	-1,0963	,0068
	D 240	-,56598*	,18512	,039	-1,1175	-,0144
	M 240	,02121	,18512	1,000	-,5303	,5727
	35 240	-,47933	,18512	,157	-1,0309	,0722
	65 240	-,06545	,18512	1,000	-,6170	,4861
B 240	A 240	,54478	,18512	,056	-,0068	1,0963
	D 240	-,02121	,18512	1,000	-,5727	,5303
	M 240	,56598*	,18512	,039	,0144	1,1175
	35 240	,06545	,18512	1,000	-,4861	,6170
	65 240	,47933	,18512	,157	-,0722	1,0309
D 240	A 240	,56598*	,18512	,039	,0144	1,1175
	B 240	,02121	,18512	1,000	-,5303	,5727
	M 240	,58719*	,18512	,027	,0356	1,1387
	35 240	,08665	,18512	1,000	-,4649	,6382
	65 240	,50054	,18512	,114	-,0510	1,0521
M 240	A 240	-,02121	,18512	1,000	-,5727	,5303
	B 240	-,56598*	,18512	,039	-1,1175	-,0144
	D 240	-,58719*	,18512	,027	-1,1387	-,0356
	35 240	-,50054	,18512	,114	-1,0521	,0510
	65 240	-,08665	,18512	1,000	-,6382	,4649
35 240	A 240	,47933	,18512	,157	-,0722	1,0309
	B 240	-,06545	,18512	1,000	-,6170	,4861
	D 240	-,08665	,18512	1,000	-,6382	,4649
	M 240	,50054	,18512	,114	-,0510	1,0521
	65 240	,41388	,18512	,401	-,1377	,9654
65 240	A 240	,06545	,18512	1,000	-,4861	,6170
	B 240	-,47933	,18512	,157	-1,0309	,0722
	D 240	-,50054	,18512	,114	-1,0521	,0510
	M 240	,08665	,18512	1,000	-,4649	,6382
	35 240	-,41388	,18512	,401	-,9654	,1377

*. The mean difference is significant at the 0.05 level.

Table 13. ANOVA with Bonferroni correction. 780 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	5,094	5	1,019	2,255	,051
Within Groups	73,171	162	,452		
Total	78,264	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A 240	B 240	-,57402*	,17962	,025	-1,1092	-,0389
	D 240	-,32219	,17962	1,000	-,8573	,2130
	M 240	-,25183	,17962	1,000	-,7870	,2833
	35 240	-,20129	,17962	1,000	-,7364	,3338
	65 240	-,37272	,17962	,593	-,9079	,1624
B 240	A 240	,57402*	,17962	,025	,0389	1,1092
	D 240	,25183	,17962	1,000	-,2833	,7870
	M 240	,32219	,17962	1,000	-,2130	,8573
	35 240	,37272	,17962	,593	-,1624	,9079
	65 240	,20129	,17962	1,000	-,3338	,7364
D 240	A 240	,32219	,17962	1,000	-,2130	,8573
	B 240	-,25183	,17962	1,000	-,7870	,2833
	M 240	,07036	,17962	1,000	-,4648	,6055
	35 240	,12089	,17962	1,000	-,4142	,6560
	65 240	-,05054	,17962	1,000	-,5857	,4846
M 240	A 240	,25183	,17962	1,000	-,2833	,7870
	B 240	-,32219	,17962	1,000	-,8573	,2130
	D 240	-,07036	,17962	1,000	-,6055	,4648
	35 240	,05054	,17962	1,000	-,4846	,5857
	65 240	-,12089	,17962	1,000	-,6560	,4142
35 240	A 240	,20129	,17962	1,000	-,3338	,7364
	B 240	-,37272	,17962	,593	-,9079	,1624
	D 240	-,12089	,17962	1,000	-,6560	,4142
	M 240	-,05054	,17962	1,000	-,5857	,4846

	65 240	-,17143	,17962	1,000	-,7066	,3637
65 240	A 240	,37272	,17962	,593	-,1624	,9079
	B 240	-,20129	,17962	1,000	-,7364	,3338
	D 240	,05054	,17962	1,000	-,4846	,5857
	M 240	,12089	,17962	1,000	-,4142	,6560
	35 240	,17143	,17962	1,000	-,3637	,7066

*. The mean difference is significant at the 0.05 level.

Table 14. ANOVA with Bonferroni correction. 840 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	8,080	5	1,616	2,864	,017
Within Groups	91,407	162	,564		
Total	99,486	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

					95% Confidence Interval	
(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 240	B 240	-,39951	,20076	,724	-,9976	,1986
	D 240	-,51549	,20076	,167	-1,1136	,0826
	M 240	,11598	,20076	1,000	-,4821	,7141
	35 240	-,13129	,20076	1,000	-,7294	,4668
	65 240	-,26821	,20076	1,000	-,8663	,3299
B 240	A 240	,39951	,20076	,724	-,1986	,9976
	D 240	-,11598	,20076	1,000	-,7141	,4821
	M 240	,51549	,20076	,167	-,0826	1,1136
	35 240	,26821	,20076	1,000	-,3299	,8663
	65 240	,13129	,20076	1,000	-,4668	,7294
D 240	A 240	,51549	,20076	,167	-,0826	1,1136
	B 240	,11598	,20076	1,000	-,4821	,7141
	M 240	,63147*	,20076	,030	,0334	1,2296
	35 240	,38420	,20076	,861	-,2139	,9823

	65 240	,24728	,20076	1,000	-,3508	,8454
M 240	A 240	-,11598	,20076	1,000	-,7141	,4821
	B 240	-,51549	,20076	,167	-1,1136	,0826
	D 240	-,63147*	,20076	,030	-1,2296	-,0334
	35 240	-,24728	,20076	1,000	-,8454	,3508
	65 240	-,38420	,20076	,861	-,9823	,2139
35 240	A 240	,13129	,20076	1,000	-,4668	,7294
	B 240	-,26821	,20076	1,000	-,8663	,3299
	D 240	-,38420	,20076	,861	-,9823	,2139
	M 240	,24728	,20076	1,000	-,3508	,8454
	65 240	-,13692	,20076	1,000	-,7350	,4612
65 240	A 240	,26821	,20076	1,000	-,3299	,8663
	B 240	-,13129	,20076	1,000	-,7294	,4668
	D 240	-,24728	,20076	1,000	-,8454	,3508
	M 240	,38420	,20076	,861	-,2139	,9823
	35 240	,13692	,20076	1,000	-,4612	,7350

*. The mean difference is significant at the 0.05 level.

Table 15. ANOVA with Bonferroni correction. 900 s, 240 °C.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	1,146	5	,229	,527	,756
Within Groups	70,514	162	,435		
Total	71,660	167			

Multiple Comparisons

Dependent Variable: MPa

Bonferroni

		Mean Difference			95% Confidence Interval	
(I) Group	(J) Group	(I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
A 240	B 240	,09933	,17633	1,000	-,4260	,6247
	D 240	-,06134	,17633	1,000	-,5867	,4640
	M 240	,16067	,17633	1,000	-,3647	,6860
	35 240	-,02571	,17633	1,000	-,5511	,4996

	65 240	,12504	,17633	1,000	-,4003	,6504
B 240	A 240	-,09933	,17633	1,000	-,6247	,4260
	D 240	-,16067	,17633	1,000	-,6860	,3647
	M 240	,06134	,17633	1,000	-,4640	,5867
	35 240	-,12504	,17633	1,000	-,6504	,4003
	65 240	,02571	,17633	1,000	-,4996	,5511
D 240	A 240	,06134	,17633	1,000	-,4640	,5867
	B 240	,16067	,17633	1,000	-,3647	,6860
	M 240	,22201	,17633	1,000	-,3033	,7473
	35 240	,03562	,17633	1,000	-,4897	,5610
	65 240	,18638	,17633	1,000	-,3390	,7117
M 240	A 240	-,16067	,17633	1,000	-,6860	,3647
	B 240	-,06134	,17633	1,000	-,5867	,4640
	D 240	-,22201	,17633	1,000	-,7473	,3033
	35 240	-,18638	,17633	1,000	-,7117	,3390
	65 240	-,03562	,17633	1,000	-,5610	,4897
35 240	A 240	,02571	,17633	1,000	-,4996	,5511
	B 240	,12504	,17633	1,000	-,4003	,6504
	D 240	-,03562	,17633	1,000	-,5610	,4897
	M 240	,18638	,17633	1,000	-,3390	,7117
	65 240	,15076	,17633	1,000	-,3746	,6761
65 240	A 240	-,12504	,17633	1,000	-,6504	,4003
	B 240	-,02571	,17633	1,000	-,5511	,4996
	D 240	-,18638	,17633	1,000	-,7117	,3390
	M 240	,03562	,17633	1,000	-,4897	,5610
	35 240	-,15076	,17633	1,000	-,6761	,3746

APPENDIX 3

Table 1. ANOVA for BD 35 200 and BD 65 200.

ANOVA

MPa

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	,010	1	,010	,029	,866
Within Groups	17,067	51	,335		
Total	17,077	52			