EXAMINATION OF OPERATIONAL PARAMETERS FOR VTC WOOD PRODUCTION

Master Thesis

Submitted by
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Acknowledgments

Abstract

Wood densification has been studied intensively for several decades. Recently, the economic utilization of fast grown and low value wood has received a lot of attention. Viscoelastic thermal compression, invented by Dr. Frederick A. Kamke and H. Sizemore, is a densification process that takes advantage of lignin transition at elevated temperatures. The process takes place in a sealed chamber containing a press apparatus with heating, cooling, and steam boiler components. After placing samples with 4 to 5mm x 240mm x 600mm dimensions into the internal press, the chamber is closed by elevating the external press unit. A clamp mechanism is used to assure pressure tightness. The process begins with a conditioning phase where wood is softened by the assistance of saturated steam. After steam venting, a pressing stage follows. In this phase wood is compressed to a specified thickness. The process ends with a combination cooling and compression phase to solidify the lignin. Different treatment combinations of conditioning time, compression rate and compression time were applied and modulus of elasticity (MOE), modulus of rupture (MOR), and glue-bond shear strength parallel to the grain with different adhesive loading rates were examined. The results show that increasing conditioning time elevates MOE, MOR and shear strength. The rate of compression did not show a significant trend. However, MOE and MOR both declined at the highest compression rate. Increasing compression time improves MOR slightly, whereas there was no impact to MOE. Shear strength was not influenced in any systematic trend. A phenol formaldehyde solid resin loading rate of $25g/m^2$ shows significantly lower glue line shear failure stresses compared to a loading rate of 50 and 70 g/m^2.

Zusammenfassung

Das von Dr. Frederick A. Kamke und H. Sizemore entwickelte Holzverdichtungsverfahren „Viscoelastic thermal compression" beruht auf Nutzbarmachung der Plastifizierung von Lignin. In einer dampfdruckdichten Kammer mit integrierter heiz- und kühlbaren Presseinheit wird Lignin mithilfe von gesättigtem Dampf bei Temperaturen um 170°C in den elastischen Zustand transformiert.

Der darauffolgende Pressvorgang bei Umgebungsdruck verdichtet das Holz entsprechend. Hohlräume werden verringert und Zellwände aufeinander gelagert. Ein anschließender Kühlvorgang gewährt die Fixierung des Holzes im verdichteten Zustand. Die Dimension der Proben war 240mm x 600mm bei einer Stärke von 4 bis 5mm. Unterschiedliche Kombinationen mit den Prozessparametern Konditionierzeit, Verdichtungszeit und Verdichtungsgeschwindigkeit wurde getestet. Darauffolgend wurden der E- Modul, die Biegefestigkeit sowie die Scherfestigkeit ermittelt. Die Klebeeigenschaften sowie die minimale Klebstoffauftragsmenge des VTC Holzes wurden im Zuge der Scherfestigkeitsprüfungen durchge-

führt um etwaige Auswirkungen des Verdichtungsprozess zu eruieren. Die Konditionierzeit zeigt einen deutlichen Einfluss auf den E- Modul wie auf die Biegefestigkeit. Verdichtungsgeschwindigkeit gibt keine Trends bei den ausgeführten Tests wieder. Verdichtungszeit wirkt sich positiv auf die Biegefestigkeit aus hingegen zeigt es keinen systematischen Einfluss auf den E- Modul zu haben. Die Scherfestigkeit zeigt in allen drei Prozessvariablen keinen systematischen Einfluss. Eine Auftragsmenge von 25g/m^2 (Feststoffharzgehalt) ergibt signifikant niedrigere Festigkeiten verglichen mit den Auftragsmengen von 50 und 70 g/m^2 welche keinen signifikanten Unterschied zueinander aufweisen.

Keywords: Wood modification, densification, Viscoelastic thermal compression, high pressure compression, hybrid poplar

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1   Introduction

Wood, a fascinating material at the macroscopic and microscopic level, has had great historical importance. Since the beginning of the 20th century, wood has been displaced by synthetic materials in multiple applications. The concept of an Eco-social market economy of the present time gives wood as a renewable resource for building materials more weight and popularity. Specific applications of wood materials require optimized or modified properties. Additionally, the shortage of natural resources and wood use restrictions leads to the necessity of its efficient use. Mechanical properties such as hardness, bending and durability are essential parameters for these specific applications.

The scientific work, carried out by the author for his master thesis, describes a wood densification process to improve hardness, bending strength and bonding quality of low density hybrid poplar (*Populus deltoids x Populus trichocarpa*). Viscoelastic thermal compression is a densification process conducted in a sealed chamber with a controlled environment. Temperatures in the chamber range from 150° to 180°C. Saturated steam is introduced during the process to elevate the wood above the glass transition temperature. After releasing steam pressure, a compression force up to 5 MPa is applied. Due to densification of wood in the viscous range, limited failure within the cell wall occurs. Cavities such as vessels are reduced depending upon the percentage of densification. The densified specimen is kept under compression until the temperature is reduced below 100° C, at which point the lignin hardens.

The process was patented as U.S. process patent #7404422 (Kamke and Sizemore 2008). The technology was further developed at Oregon State University, Department of Wood Science and Engineering, headed by Dr. Frederick A. Kamke. The influence of the different press parameters are partly known from previous research on a prototype press. The newly installed press possesses larger capacities regarding pressing area and pressure. Additionally, the whole process can be controlled via computer.

The original purpose of this work was to develop a suitable pressing schedule for processing wet veneer at the newly installed VTC machine and evaluate the impacts of process parameters on the mechanical properties of wood along with bonding quality and the lowest possible amount of resin loading. This was the first project to utilize the new VTC press. One objective of the project was to learn how to use the new equipment and test the process control system. Because of technical difficulty and time schedule constraint, wet veneer could not be

processed. Instead, air-dried hybrid poplar veneer was used for sample fabrication.

2 Possible Applications

The Inventors describe the possible VTC applications below.

> *"Viscoelastic thermal compression (VTC) wood can be further processed in numerous ways for further use, including but not limited to: cutting and shaping the sheets or panels into various desired lengths or shapes; attaching multiple layers of the sheets together with similar or different materials to form a multi-layered laminate material of the desired thickness, "cosmetic" processing such as coloring, staining, etching, and overlaying. The high-density, dimensionally stable lamina produced by VTC process are of a quality that is suitable for use in laminated composites for structural inside and outside applications, as flooring and underlying materials, siding and roofing material, materials for constructing walls, etc."* (Kamke, et al., 2005)

Viscoelastic thermal compressed wood can be applied to products where density and hardness play a critical role. A survey of forest products engineers and architects was undertaken (Macias, 2006). It showed that the most appropriate VTC applications are for LVL, plywood, concrete forms, transportation components, flooring and the outer layer of beams. Wood hardness can be increased according to the degree of densification up to a maximum density of approximately 1.4 g/m^3, at which point almost all voids are compressed. Eastern cottonwood was tested by (Kamke, 2007) and the original density of 0.4 g/m^3 was compressed threefold.

However, as long as parameter influence on physical properties and color change are not known, designing an industrial VTC machine and producing large scale VTC products might not be feasible. Suitable applications such as flooring require specific standards and qualities.

3 Economical aspects

According to the Food and Agriculture Organization (FAO) (2010), primary forests (forests with native species and no significant disturbance through human influence such as rain forests) make up 36% of the forest area (global) while naturally regenerated forests make up 57 percent. Primary forests decreased by 40 million ha. since the year 2000. On the other hand, planted forest area increased by about 5 million hectare each year.

The wood used in this project is hybrid poplar (*Populus deltoides x Populus tri-chocarpa*). Hybrid poplar is heavily grown on agricultural land in the United States and Canada, mainly in Oregon, Washington, Idaho and British Columbia. Total hybrid poplar land area reaches about 55,000 to 60,000 acres total (Chastagner, et al., 1999). Annual harvesting areas in the years before 2000 were approximately 3,000 acres per year for pulp with an estimated value of $ 8.1 million dollars. (Chastagner, et al., 1999). Stanton (2007) reported an annual hybrid poplar harvest of 34,000 ha (~ 84,000acres) in the Pacific Northwest, North Central and Mississippi River Valley regions for pulp and paper production. The pulp and paper production peaked in 1995 and 1996. The production of pulp and paper in North America has declined since then (FAO, 2004). The stagnation in the pulp and paper industry has forced the development of different applications for plantation wood originally grown for the pulp industry. Hybrid poplar as a fast grown material can be used as biomass for bioenergy or for engineered wood composites such as oriented strandboard (OSB) (Kenney, et al., 1990). According to Goetzl, et al. (2007), United States of America wood products consumption, including structural panels, composite panels and engineered wood products, was estimated at approximately 220 million m^3. A huge demand for structural wood exists and new materials such as VTC wood might create new wood applications due to advanced strength characteristics. The annual consumption of hardwood for pulp and composite panels are about 50 million m^3 (Goetzl, et al., 2007). The United States consumes 160 million m^3 (33% of the world's total) sawnwood annually where softwoods account for approximately 83%. In 2006, the hardwood sawn wood was 24 million m^3 (Goetzl, et al., 2007).

The value of US hardwood flooring, including domestic shipments and imports, in 2005 was estimated at US$ 22.6 billion and represented an area of 102 million m^2. About 80% of the hardwood flooring is produced by 100 companies (Goetzl, et al., 2007). Laminate flooring has overtaken hardwood flooring in this area. Production was estimated to be 120 million m^2 in 2005. Sales of hardwood flooring, including laminates, represent about 1/5 of the US $24 billion floor market (Goetzl, et al., 2007). The wood and energy supply greatly depends on world financial markets due to the capital intensive nature of large projects (Howard, et al., 2011). The current market situation asks for new products with enhanced properties and a high degree of sustainability.

In 2009, 17 million m^3 of structural panel products were produced (Howard, et al., 2011). The private and public construction market has been declining steadily since the financial crisis 2008 (Howard, et al., 2011). Wood flooring imports represent more than 30% of US- flooring consumption. U.S.'s most important importing partners are China (50%), Brazil (21%), Canada (12%), and all others

with 12% (Floor Daily, 2011). High density surfaces produced from domestic wood has the opportunity to replace tropical wood imports.

The four major hardwood flooring manufactures in North America are Armstrong, Shaw, Mohawk and Mannington. Current challenges in the flooring market involve developing alternative durable covering (49%), meeting consumer demand (33%), developing new products (22%) and providing installation services (22%) (FP Innovations, 2009). VTC wood matches well to the challenges mentioned above. The economic impact of this new product development may be not negligible for the wood products industry. The flooring industry could greatly benefit since wood properties and appearance can be adjusted in an efficient modification process. The economic feasibility of this modification process has not been discussed yet.

Coping with juvenile and low density wood and converting it into high performance products require fundamental research to succeed with new developments and products. Overall, a trade off for a 50 to 75 year decrease in rotation times has to be taken into account (Lenth, 1999) and should stimulate further research.

4   Literature Review

4.1   Wood modification

The strength properties of wood relate proportionally to wood density (Kollmann, et al., 1968). Many applications require increased properties in regard to hardness and bending strength (e.g. flooring, joineries). High prices and limited availability of highly dense species enforce the development of modification techniques to improve the properties of lower value, faster growing wood species. Successful research has produced high resistance particle boards, whereas the densification of solid wood has been less economically successful due to high costs and technical problems (Wingate- Hill, 1983). Since the last decades research focuses on upgrading low quality, fast growing wood. Recently developed processes that address this need are the Thermo hydro mechanical treatment (THM), TimTek, Viscoelastic thermal compression (VTC), CaLignum. Several bulking methods achieve anti-swelling effiency (ASE) of a material which is a good indication of dimensional stability. Most of the wood modification processes result in altering the absorption behavior of moisture and thus lowering the equilibrium moisture content (Hill, 2006). Since many properties are influenced by decreasing moisture content (Kollmann, et al., 1968), altering the EMC is an efficient way to enhance certain wood properties.

4.1.1 Definition

(Hill, 2006) provided and all- encompassing definition for wood modification.

> "*Wood modification involves the action of a chemical, biological, or physical agent upon the material resulting in a permanent change to the polymeric chemical composition; with such a change leading to a desired property enhancement. The modified wood should itself be nontoxic under service conditions and furthermore, there should be no release of any toxic substances during service, or at end of life following disposal or recycling of the modified wood.*"

Hill, (2006) describes the different wood modification methods in detail. He divides modification into three groups; chemical, thermal, and surface and impregnation modification. Wood modification involves altering wood properties to ameliorate certain disadvantages such as dimensional stability, weather performance, durability and others. Surface modification is used to improve wood's ultraviolet

stability, change its surface energy, and improve bonding between wood surfaces (Hill, 2006). Norimoto, et al. (1993) exemplifies three mechanisms important for shape fixation. Those are (1) the formation of cross- linkages between matrix molecules, (2) relaxation of the stresses within the microfibrils and the matrix, and (3) the formation of polymers from hydroxylic cell wall constituents, particularly hemicelluloses, to avoid resoftening under moist environments.

Wood modification

Hydro thermo mecahanical treatments

Chemical treatments affecting the crystalline and non- crystalline structure

Densifictation:
- TM densification
- THM densification
- VTC and moulding

Wood dimmensional stability:
- Torrefied wood
- Thermo wood
- Plato- process

- Wood welding
- Wood surface densification
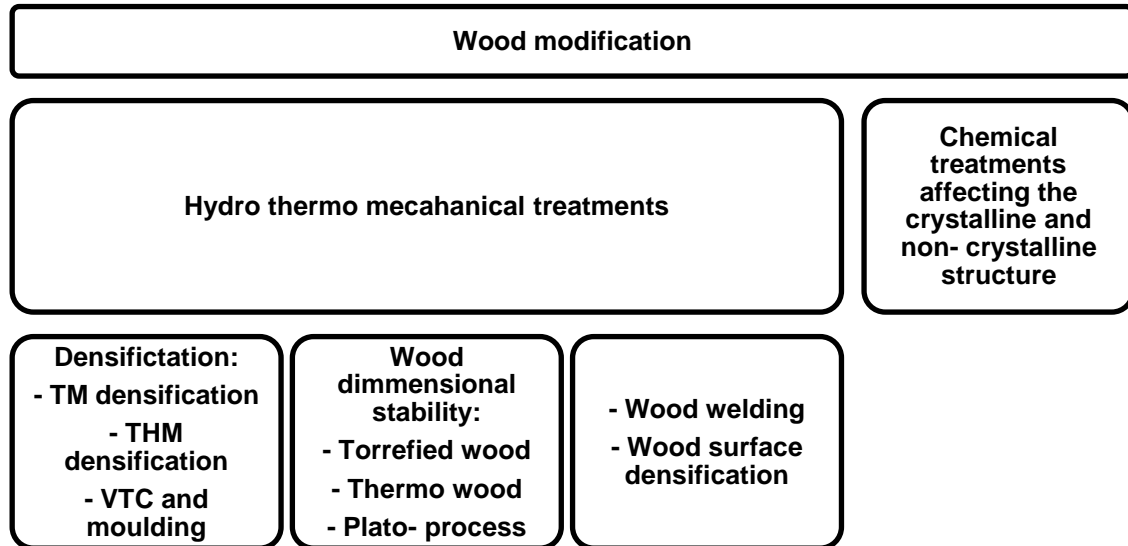
Figure 1: Wood modification focused on hydro thermo mechanical treatments

Figure 1 shows a division of wood modification specifically in regard to hydro thermo mechanical treatments.

4.1.2 Different methods of wood modification

4.1.2.1 Chemical modification

Chemical modification methods mostly involve the chemical reaction of a reagent with the cell wall polymer hydroxyl groups. It either forms a single chemical bond with one OH- group or a cross- linking between two or more OH- groups (Hill, 2006). This changes the chemical nature of the cell wall polymers and hence alters the properties.

4.1.2.2 Thermal modification

Thermal modification of wood happens in the temperature range of 180°C to 260° C. Treatment temperatures below 140°C alter the material properties just slightly and temperatures beyond 300°C result in severe degradation of the material. High temperature treatments also change the macromolecular constituents thus the physical and biological properties of the wood. Mostly, these changes are improvements in dimensional stability, reduced hygroscopicity, higher resistance to

microbiological attack, reduced impact toughness, modulus of rupture and work to fracture. The properties of the thermally modified wood are highly influenced by the treatment conditions employed to the wood. The chemical substances are affected differently by thermal heat treatment. Hemicelluloses degrade to a greater extent than the other macromolecular components. A significant loss of polysaccharide materials such as lignin or celluloses occurs at temperatures above 180°C (Hill, 2006).

4.1.2.3 Surface modification

Due to the difficulty of equalization of the reagent throughout the wood material, surface modification has the aim to alter the ultraviolet (UV) stability of wood, to change the surface energy and (or) improve the compatibility with coatings or matrix materials as well as to elevate bonding between different surfaces (Hill, 2006). Higher UV stability can be achieved by acetylation where phenolic hydroxyl functionalities are altered. The hydrophobicity of the wood surface can be influenced by exposing the wood surfaces with silicone in order to get Si- O- C linkages formed by a hydrolysis reaction.

4.1.2.4 Impregnation modification

The main principle is to impregnate the cell wall of the wood with a chemical which reacts and is locked into the cell wall. Swollen cell walls ensure better accessibility for penetration. Molecular size of the chemical plays an important role. Smaller molecular components gain more easy access to the cell wall interior. Mainly two mechanisms are responsible for fixation of the impregnation. Those are monomer impregnation with subsequent polymerization within the cell wall and diffusion of a soluble material into the cell wall. Second mechanism is followed by a treatment to make the material insoluble (Hill, 2006).

The impregnation process with various resins is discussed by Hill (2006). He noted that a high ASE is feasible if certain criteria are considered such as molecular size of resin molecules, the degree of polymerization, solubility in polar solvents, and a sufficient polarity to exhibit a high affinity with cell wall macromolecular components. Resin increases dimensional stability by penetrating and swelling the cell wall. Phenol formaldehyde resin was found to be the most promising resin. This modification also reduces the hygroscopicity (Hill, 2006).

4.1.3 Modification by mechanical compression

The goal of wood densification is to improve mechanical properties such as Young's modulus, surface hardness, transverse shear strength and dimensional stability. There are many methods described in the literature that involve some

form of mechanical compression of wood with the intent to improve properties. Typically, the wood is first exposed to high temperature to soften the cell wall prior to compression. High moisture content may be used to reduce the required softening temperature. The wood is then compressed in the radial or tangential direction to collapse the cell walls and reduce void volume. Softening the wood prior to compression reduces or eliminates fracture of the cell walls. Depending on the temperature and duration of exposure, some degree of heat treatment is also imposed on the wood. Classification of the wood densification methods is difficult. Therefore, this section will discuss a few of the more prominent methods.

Overall, wood densification techniques try to fulfill the three main mechanisms described by Norimoto, et al., (1993) - (1) formation of cross-linkages between polymers within the cell wall matrix, (2) relaxation of the stresses present in the microfibrils and the matrix, and (3) the formation of polymers from hydroxylic cell wall constituents, particularly hemicelluloses, to avoid a possible shape recovery under moist environments.

Historically, compressed solid wood was produced in Germany in 1930 under the trade name "Lignostone". A similar product under the trade name "Lignofol" was developed at the same time. These products were compressed at approximately 30MPa and 140°C and resulted in product density of about 0.8g/cm^3. The modulus of elasticity reached beyond 11000 MPa (Röchling, 2000). Similar materials, with product names Jicwood and Jablo, have been produced in England at this time, but no information regarding process or properties are available. Although Jicwood and Jablo were used in the manufacture aircraft propellers. In the United States, patents on wood densification methods date back to 1900, although they have not been widely used in industry (Kollman, et al., 1975). Other methods such as Compreg (Stamm, et al., 1941) and Staypak (Seborg, et al., 1962a) were developed in the United States at the US Forest Products Laboratory. Compreg is veneer impregnated with phenolic resin and compressed. Thickness and specific gravity are controlled by pressure along with the amount of resin introduced to the wood. The percentage of resin goes up to 30% depending on the species. Pressure applied to the veneer specimens are between 7 MPa and 8.3 MPa at approximately 150°C (Seborg, et al., 1962a). The increase in strength properties, except the impact strength of Compreg wood, is proportional to the increase in density. The recovery, also referred to as springback, of untreated compressed wood is much higher compared to treated wood (Seborg, et al., 1962a). Staypak involved compression of non-impregnated wood using pressures of 9.6 MPa to 17.2 MPa while in a temperature range of 150°C to 180°C (Seborg, et al., 1962a). Compressed wood must be cooled in the compressed state below the T_g of lignin, between 80°C and 120°C.

Densification of wood, using the compliant properties at high temperature under saturated steam increases the efficiency on the strength of densified wood (Haygreen, et al., 1969; Tabarsa, et al., 1997). This method uses elevated temperatures, in the range from 140°C to 200°C to transfer the lignin into the compliant state. The transition process is supported by saturated steam. Softened wood is than compressed by mechanical compression in order to densify the material and improve mechanical properties. Currently there are two established methods using thermo- hydro- mechanical modification.

Navi, et al., (2000) described a THM process for producing densified wooden blocks. The densification process was carried out under saturated steam temperature of 150°C and compressive force of 12.7 MPa. Under these conditions wood flows plastically and after cooling, can be fixed permanently. Multiple wood species specimens, with and without knots, were used. Densification occurred in the radial, tangential and transverse direction. Densified wood showed significant mechanical performance. Shear strength in longitudinal direction was increased by a factor of ten. Additionally, hygroscopicity is reduced making wood more stable and with almost no shape memory (Navi, et al., 1997).
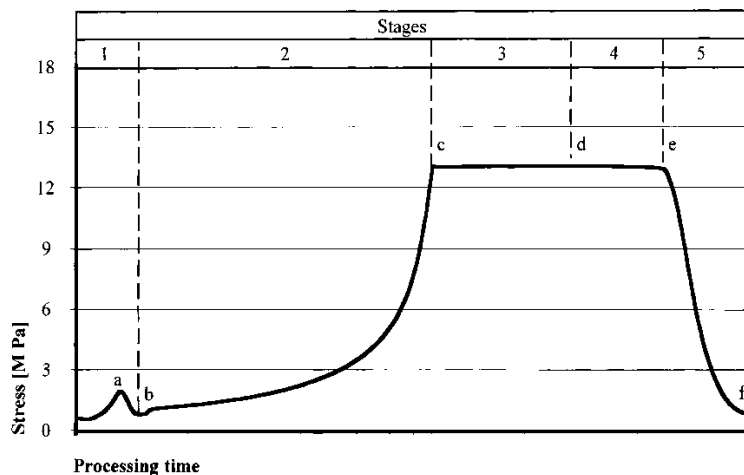
Figure 2: Schematic diagram of THM densification process

The process was carried out in five stages shown in Figure 5. The first stage was softening under saturated steam at 150°C for approximately 10 min. The densification stage followed (2) until the desired pressure was achieved. Stage three is considered constant compression where stage four represents the drying stage where steam injection has stopped. Stage five was the unloading time. Overall processing time is about three hours (Navi, et al., 2000). Species used in testing were spruce and pine. Shear strength was increased from 10 N/mm^2 to 110 N/mm^2, Brinell hardness was increased from 10 N/mm^2 to 65 N/mm^2 in the radial and tangential direction. The adsorption isotherm showed a moisture content e.g. at a relative humidity of 60 percent, almost half of normal undensified wood. Swelling decreased by almost 80%. The examination of early and latewood densification showed more closed wood cells in latewood than earlywood, weaker

parts, such as vessels, are more densified compared to stronger parts. An increase in process temperature also led to a higher pressure of the absorbed water in the specimen. Densification with 25MPa did not compress the wood to the maximum degree and lumens were still open. Nevertheless, the thermo hydro mechanical process yields significantly higher densification compared to the thermo- mechanical process (Navi, et al., 2000).

The basic principles of the VTC process are similar to the process developed and described by Navi, et al. (2000). The main differences are the VTC process is limited to thin wood (< 10 mm), incorporates transient environment conditions, and has a short process time (< 20 min). All experiments treated with the VTC densification process were veneers ranging from 3 to 10mm. The three main steps of the process are conditioning, pressing and cooling. At low temperatures wood has naturally brittle and stiff characteristics. The deformability of wood polymers is elevated at higher temperatures and moisture contents (Navi, et al., 1997). Compression and plastic deformation, depending on wood polymers viscoelastic behavior, can cause cell wall fracture (Navi, et al., 2000). The transformation from the plastic to the compliant state occurs when temperature and moisture are applied. Between these two phases the glass transition temperature is reached (Wolcott, et al., 1994). The T_g of lignin and hemicelluloses changes drastically with moisture content (Irvine, 1984). Figure 3 displays the VTC process schematic. Conditioning of the wood with saturated steam softens the wood. Temperatures most commonly used during this step range from 150°C and 180°C. Softened wood can then be densified without significant cell wall fracture due to the transformation from the plastic to the elastic state. This allows for utilization of the mechanosorptive nature of wood by manipulating the release of moisture during compression (Kamke, et al., 2010a). Cellular collapse occurs by elastic buckling or plastic yielding, and avoidance of brittle crushing, depending on the nature of the cell wall material (Lenth, et al., 2007, Kamke, et al., 2010a). The chamber was vented of steam before a compression stress of approximately 5MPa was applied. After the cellular collapse region, stress increases significantly with the strain, which occurs after the majority of the cell walls have collapsed. Mechanical properties of the densified wood are highly influenced by behavior (Kutnar, et al., 2008b; Kutnar, et al., 2009). The degree of densification is controlled by applied force or via distance between the internal press platen. Cooling took place by connecting water hoses to the press platen. Platen temperature should drop to 100°C before compressive force is released to avoid springback. Technical limitations are the gas pressure and the temperatures are currently 1MPa and 250°C (Kamke, et al., 2010b). With this compression process the wood was densified up to 1.4g/cm^3. The anisotropic nature and the different compressibility of the wood tissues have an effect on density distribution over the cross-section and thus in-

fluence the mechanical properties (Lenth, et al., 2007). Initial moisture content of the wood used may range from 5% up to complete saturation. Final moisture content after processing was between 5% and 8%.
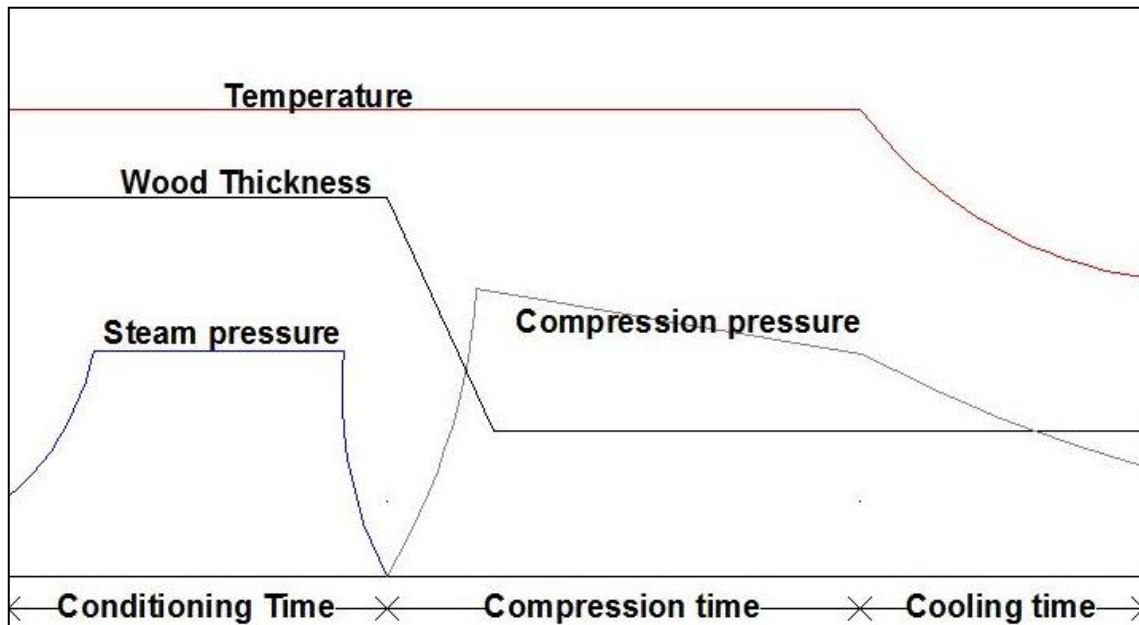
Figure 3 Schematic diagram of VTC process

The apparatus previously used for research on the viscoelastic thermal compression process is shown in Figure 4. The process was performed in a custom- built pressurized vessel equipped with an electrical jacket heater. The detachable lid includes the separately controlled press platen with the hydraulic press. Steam was supplied with a dedicated boiler. Process control elements such as thermocouples are connected to the computer. This prototype press was operated manually and the connected computer was used for controlling temperature and data collection. Steam pressure was monitored with a pressure transducer and controlled via mechanical gauge. A linear variable differential transformer (LVDT) (Macro Sensors, Model HSTAR 750-500, _0.031 mm) controlled the distance between the two press platens.
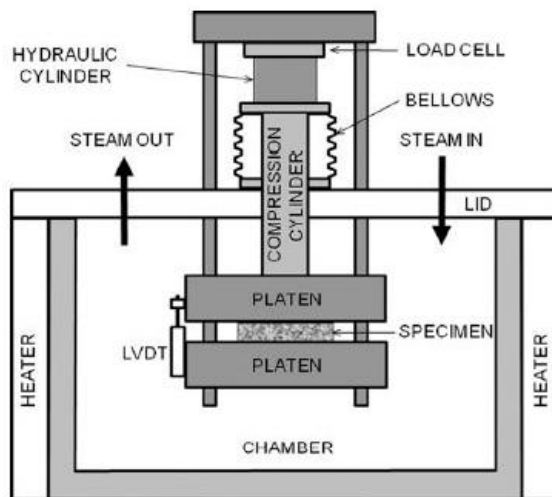
Figure 1. Schematic drawing of the pressure vessel used to compress specimens.
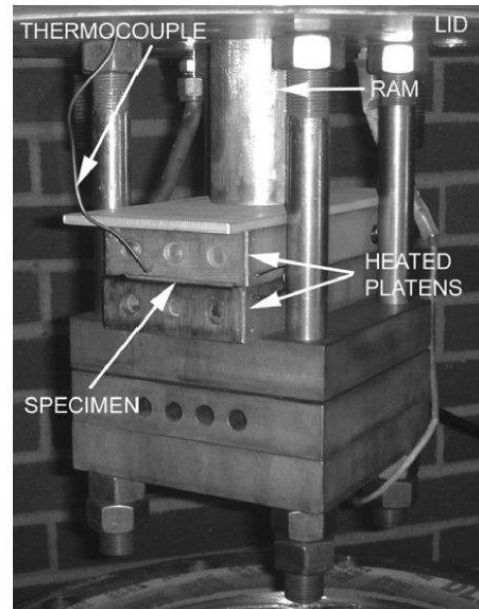
Figure 4 View of the prototype press extracted from VTC apparatus showing single specimen arrangement with independently heated platens schematic (left), real (right) (Kamke, 2006).

The degree of densification, as well as prevention of moisture related springback, is improved by steam treatment. Non-densified wood shows a strong relationship between strength properties and density (Kollmann, et al., 1968). Properties of the wood are highly influenced by moisture, temperature, and time of treatment (Morsing, 2000).

Blomberg, et al., (2005) showed the influence of density of untreated wood to the compression strength in the different directions as well as the bending strength and Brinell hardness. A linear correlation exists between axial compression strength and bending strength. Radial and tangential compression strength and Brinell hardness show an exponential relationship. The relationship between strength properties and density also occurs in VTC- wood (Kamke, 2007). The densification treatment has a major impact on cell wall properties and the relationship between strength and density.

Elevated temperatures at high moisture content soften wood components and show an increase in strength properties. An increase of temperature or moisture content decreases the compressive modulus of wood (Kunesch, 1961). A hydro-thermal treatment has a strong influence on the mechanical behavior of wood during compression. Steam treatment induces cross- linking reactions in the matrix substances and crystallization of microfibrils, (Dwianto, 1999) and hence improves the fixation of densified wood (Norimoto, et al., 1993).

Inoue, et al., (1993a) reported a compression process using steam with temperatures of 180°C or 200°C. The modified wood did not indicate thickness recovery during subsequent water soaking tests. Mechanical properties in addition to

hardness decreased. Darkening occurred because of elevated temperatures during the process.

Ito, et al., (1998) developed a process to transform the shape of the wood with compression and steam fixation. The results show that a steaming temperature of 180°C for 8 minutes is required to prevent recovery. Norimoto, et al., (1993) used microwave heating for bending. The specimens were water- saturated, irradiated with a microwave, and bent over a semi-circular wooden mold. They found that specimens with a heat treatment at 140°C for 2 hours had a significant set recovery. The cell geometry of each region has a high influence to the initiation of the compression. The term relative density defines the ratio of the apparent density and the real density of the solid wood it is made. It has a significant impact to the compression behavior of cellular materials. The anisotropic nature of wood results in a different response to radial and tangential compression (Kollmann, et al., 1968).

The softening or glass transition temperature (T_g) is an important value in densifying wood. When the temperature approaches T_g of the lignin, modulus declines rapidly until a compliant phase is reached (Figure 9). Usually the stiffness properties are reversible by lowering the temperature. Lignin has the most influence on this change, although amorphous cellulose has also been reported to contribute to this behavior (Kamke, et al., 2010a). The T_g depends upon temperature and moisture content of the wood and can be achieved above 50°C. In the time domain of 1- 100 seconds, under water saturated conditions, T_g of wood is approximately 70° C (Kelley, et al., 1987).

Differences between species exist, although significant differences generally appear between hardwoods and softwoods (Militz, 2002). The main components of wood, cellulose, hemicelluloses and lignin, degrade differently under heat treatment. Hemicelluloses is more affected at higher temperatures compared to cellulose and lignin. The differences might be due to higher oxygen contents in hemicelluloses. Extractives within the wood degrade easily and evaporate as volatile compounds (Finnish Thermowood Association, 2003). Loss of polysaccharide material becomes notable above 180°C depending on treatment conditions (Hill, 2006). Lignin has the largest resistance to heat. The mass of lignin starts to decrease at 200°C. Beyond this temperature ether bonds begin to break and the methoxy content decreases. In the temperature range of 120 to 220°C diphenyl-methane- type condensation reactions typically occur. Due to this lignin reaction properties such as color, reactivity and dissolution change. Wood extractives constitute less than 5% of wood material. Extractives exist as fats, waxes, terpenes and phenols. Most of these compounds evaporate easily during heat treatment. Substantial bending strength loss can be witnessed beyond 220°C although the modulus of elasticity is not significantly affected. For this research

pine was used and results may vary depending upon the species (Finnish Ther-mowood Association, 2003). The following figure, Figure 5 shows the reaction mechanism of heat treated wood.
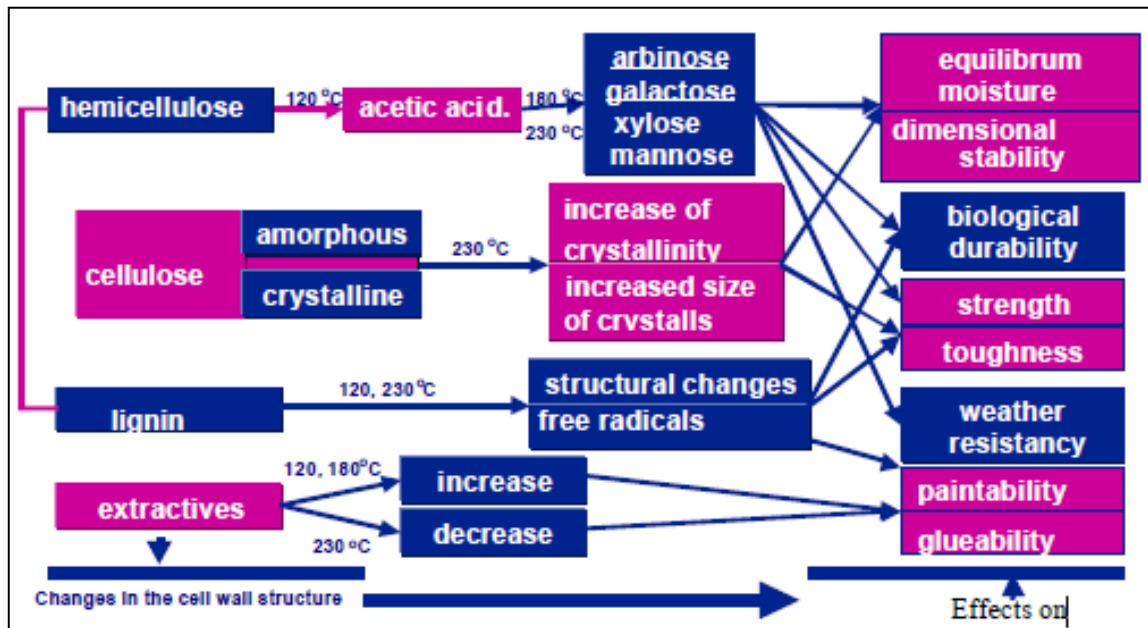
Figure 5 Reaction mechanism of heat- treated wood (Finnish Thermowood Association, 2003)

The presence of water or steam affects reaction chemistry. Organic acids, pri-marily acetic acid, are generated and catalyze the hydrolysis of hemicelluloses and, to a certain extent, the amorphous cellulose (Mitchell, 1988).

Bonding quality of thermally modified wood changes due to the reduction of sur-face energy (reduction in OH- content), which affects the wettability of the ma-terial (Chang, et al., 1978). UF resin glue line tests with aspen, beech, maple and elm species from different wood heat treatments showed a reduction in shear strength by increased time and temperature of the treatment. Aspen performed rather better than the other wood tested (Chang, et al., 1978). On the other hand, VTC wood has shown equal or superior adhesive bond strength (Jennings 2003; Jennings et al 2005; Kutnar et al 2008a). This contradiction of adhesive bond performance may be due to densification of VTC wood, reduced adhesive pene-tration into the porous wood, and the complex stress transfer pattern across the bonded interface.

The CaLignum process is a novel approach to wood modification. It uses a Quin-tus press commonly used for sheet metal fabrication (Johanisson, 1994). Wood beams are placed onto the Quintus press table and stabilized with rubber mate-rials. A rubber membrane is placed on top of the beams. The rubber membrane enfolds when oil pressure is applied and compression begins. The oil pressure

reaches 110 MPa in two minutes. Evenly distributed pressure ensures a homogenous compression of the wood.
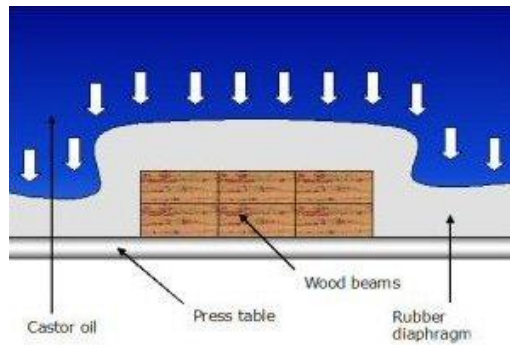
Figure 6 Schematic CaLignum densification process (Calignum, 2010)

Depending upon species, an increase in bending strength up to 40% and about 35% smaller dimension can be achieved. For example, aspen (*Populus sp.*) was densified to 50% of its original thickness. Density was increased from 490 kg/m^3 to 890 kg/m^3 and bending strength from 80 MPa to 104 MPa respectively (CaLignum, 2010).

5 Viscoelastic Thermal Compression VTC

5.1 Properties of Viscoelastic thermal compressed wood

The viscoelastic thermal compression process was chosen for this study. Consequently, a more complete discussion of VTC is included. VTC has a great impact on strength properties, bonding quality, surface energy, adsorption and desorption, equilibrium moisture content, fracture behavior, and dimensional stability. The alteration of wood properties due to the heat treatment of wood, with or without compression, is well described. (Hill, 2006, Tabarsa, et al., 1997, Conner, 2007, Kamke, et al., 2010). Process conditions have a high influence on wood deformation (Dogu, et al., 2010). Examination of compressed wood shows that the deformation of the wood throughout the growth rings was not uniform with the highest deformation occurring in the earlywood regions which control elastic and plastic stress strain response (TABARSA, et al., 2000). It is noted that wood responds differently to radial and tangential compression due to its anisotropic nature (Kunesch, 1961).
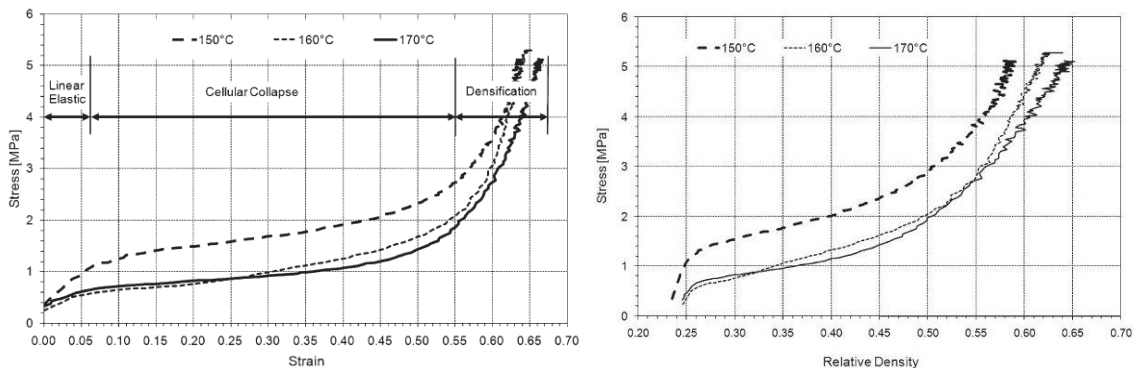
Figure 7(left) Average stress- strain curves of wood in transverse compression under saturated steam conditions at 150, 160, and 170 °C (Kamke, et al., 2010b).
Figure 8 (right) Relative density response of specimens compressed at constant rate of stress under saturated steam at 150, 160, and 170° C (Kamke, et al., 2010b)

As seen in

Figure 8 and Figure 7 a higher degree of densification after cell wall buckling requires higher compression loads. Densification was done up to 1.4g/cm^3 (Conner, 2007; Kamke, et al., 2010b). When densifying wood up to 1.4 g/cm^3 almost all of the void space is removed and cell wall remains. The specific gravity of the cell wall is about 1.5 g/cm^3. Kutnar, et al., (2008) examined VTC wood from hybrid poplar, with different degrees of densification, and found that morphology strongly depends upon the degree of densification. Voids are reduced respectively and cells are deformed without cell wall fracture. The vessel collapsed and flattened in the direction compression was applied whereas wood rays buckled. Hybrid poplar has minor differences between early- and latewood (Balatinecz, et al.,

2010). Morphological differences between early and latewood after VTC processing could not be distinguished (Kutnar, et al., 2008b).

Temperature and degree of compression has a major influence on densified wood recovery. Set recovery was evaluated on different treatments at 150°C, 160°C and 170°C and significant differences were found between wood treated at 150°C compared to 160°C and 170°C. A breakdown of the intermolecular linkages at higher temperatures is responsible for the less memory effect in wood, possibly along with a slight flow of lignin and covalent bonding in the deformed position (Inoue et. al. 2008).

High pressure steam pretreatment caused partial hydrolysis of hemicelluloses (Hsu, et al., 1988). This applies to softwoods as well as for hardwoods, resulting in higher wood compressibility. A steam treatment at 200°C for 1 to 4 minutes showed that hemicelluloses hydrolyzed into low molecular weight compounds, whereas lignin and cellulose did not decompose. Under such treatment, hemicelluloses and lignin were significantly removed from the wood (Ito, et al., 1998).The difference in transition between juvenile and mature wood has the greatest influence on processed wood final properties. At moisture levels up to 12%, yellow poplar juvenile wood showed a lower T_g compared to mature wood (Lenth, 1999). The glass transition temperature (T_g) is considered one of the most significant factors when densifying wood using temperature. In water saturated conditions the glass transition (T_g) of lignin ranges from 60- 90°C (Figure 9). Dry lignin softens at about 200 °C (Irvine, 1984). The average lignin content of hybrid poplar clones is 19% and varies between hybrid species (Blakenhorn, et al., 1985a).
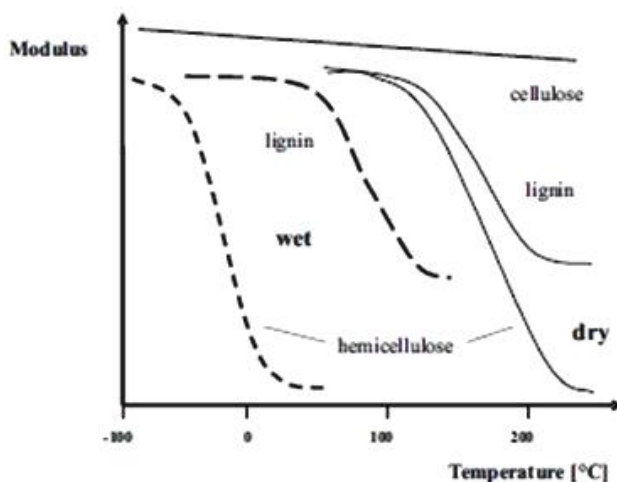
Figure 9: The glass-transition temperature of lignin as a function of moisture content and the temperature (Morsing, 2000)

5.1.1 Modulus of elasticity (MOE), modulus of rupture (MOR)

The modulus of elasticity is defined as the slope of the stress-strain curve in the elastic deformation region, mostly determined from bending or axial testing. Modulus of rupture expresses the maximum load- carrying capacity of a member in bending, which is proportional to the maximum moment borne by the specimen. Although it is a commonly accepted criterion of strength, it is not the true stress value because the computation formula is valid only within the elastic limit (Kollmann, et al., 1968).

The main motivation for densifying wood is improving wood mechanical properties (Irvine, 1984). In general, past research shows an almost linear correlation between degree of densification and the improvement in MOE and MOR (Kamke, et al., 2005, Kamke, et al., 2010b, Kamke, et al., 2009, Kutnar, et al., 2008b, Conner, 2007). As an example, densification of sweetgum (*Liquidambar styraciflua*) and eastern cottonwood (*Populus deltoides*) were densified up to 178%. MOE and MOR increased by 254 and 156% respectively, which is almost proportional to the increase in wood density (Conner, 2007). Thermal degradation of amorphous wood components causes wood weight loss. Subsequently, the mechanical properties are influenced by the treatment temperature (Jennings, 2003; Kamke, et al., 2010b). Calculation of the cell wall modulus (E_{wc}) showed the highest E_{wc} of 1.4 GPa in specimens compressed at 150°C (Kamke, et al., 2010b). Higher temperatures (160 and 170°C) lowered the E_{wc} to 600 MPa and 580MPa.

Three layer laminated composites with densified wood in the outer layers and untreated yellow-poplar (*Liriodendron tulipifera*) in the core layer were tested for modulus of elasticity and modulus of rupture in flatwise bending. MOE and MOR were elevated by 130% and 91% respectively (Kamke, et al., 2005).

5.1.2 Shear strength and compression properties

Shear strength parallel to grain describes the ability to resist internal slipping along the grain. Values presented are average strengths in radial and tangential shear planes (Kretschmann, 2010). Kutnar, et al., (2007) investigated the lap shear tension strength of densified yellow poplar with urea formaldehyde (UF) adhesive, phenol formaldehyde (PF) adhesive and polyvinyl acetate (PVAc). Results showed the same strengths compared to non densified veneers for the first two adhesives whereas polyvinyl acetate (PVAc) samples showed higher strengths as compared to control samples. The compression properties are dependent on various anatomical features of the wood specimen such as density, percentage of latewood material, ray volume and loading direction (Nairn, 2006).

It has been noted that the initiation of compression failure occurs from the buckling of rays in the earlywood (Kunesh, 1968).

5.1.3 Bonding quality

Adhesive bonding potential of wood depends upon surface energy and surface morphology, such as roughness. Non-densified hybrid poplar bonds easily with common commercial adhesives, although some adjustments to the viscosity of adhesives may need to be made because of high porosity and excessive adhesive penetration in to the wood. (Balatinecz, et al., 2010). Mechanical interlocking of the resin with the wood substrate is believed to be an important bonding mechanism. Adhesive penetration also promotes secondary bonding forces between the cured adhesive and the internal surfaces of wood.

Adhesive penetration and wood surface energy are important considerations with VTC wood. Aqueous adhesive systems show little penetration in VTC wood (Kutnar et al 2008a). Surface energy of VTC wood was significantly lower than untreated hybrid polar wood, yet the adhesive bond performance of VTC specimens was superior to the untreated specimens (Kutnar et al 2008c). Surface energy and bond performance of yellow-poplar (*Liriodendron tulipifera*) VTC wood was studied (Jennings 2003; Jennings et al 2005). Surface energies of control, hydro-thermal treated and VTC densified samples were compared. The results showed that hydro-thermal treatment significantly reduced the surface energy of yellow-poplar compared to control specimens. Whereas, densification with the same hydro-thermal exposure exhibited only a slight decrease in surface energy when compared to hydro-thermally treated wood. This final conclusion indicated the major cause of the surface energy reduction of densified wood is due to the hydro-thermal conditioning during the densification process. The bond performance of VTC wood was found to be similar, and in some cases even better, than that of the untreated yellow-poplar.

6   Methods and Materials

6.1.1 Hybrid poplar

Low quality, fast growing hybrid poplar (*Populus deltoids x Populus trichocarpa*) was used for this project. Hybrid poplar clones exhibit a wide range of density between hybrid species as well as in the tree themselves. The density of Belgian poplar hybrids (*P. trichocarpa x deltoides*) ranges between 0.3 and 0.37 g/cm^3 (Balatinecz, et al., 2010).

The veneer was grown in Clatskenie, Oregon and harvested in June 2010. The veneer was peeled from four logs and mixed together. It was cut to 240 mm x

600mm dimensions, sealed in plastic packs, and stored in a coldroom at 4°C. "Moth Ice Crystals" chemical, which contains paradichlorobenzene (99%) in the amount of 10g/ kg wet wood, was added to prevent mold of the wet veneer. Samples were removed from cold room in Mid- August, air dried, taken and sorted accordingly. In some cases, white rot and fungi were found. Those samples were excluded. Due to a shortage of veneer, borderline cases were taken but documented as such.

The anatomy and characteristic of hybrid poplar (*Populus deltoids x Populus trichocarpa*) wood is principally the same throughout all species. The sapwood usually appears as white to light yellow, heartwood more whitish- gray to gray brown. The growth rings are easily visible to the naked eye. Poplar is diffuse to semi- diffuse porous with broad growth rings, often exceeding 2cm. Fresh poplar wood has a characteristically disagreeable odor which disappears after drying. Poplar has about 50 to 65% supportive tissue or fibers. The vessels make up 30% and the parenchyma about 10%.
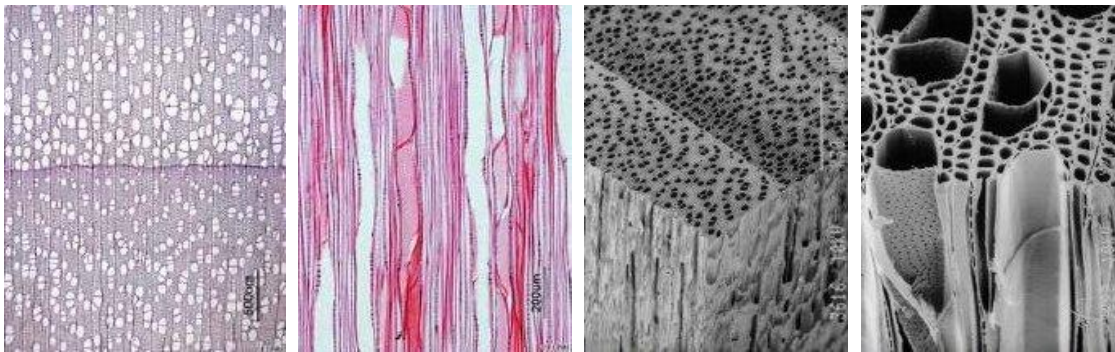
Figure 10: From left: transverse surface, tangential surface, and SEM mocrographs. of *Populus. maximowiczii*. **(Balatinecz, et al., 2010)**

The average density of the wood was 0.3g/cm^3. Poplar is characterized by high polysaccharide (approximately 80% holocellulose) and low lignin content, roughly 20% (Balatinecz, et al., 2010).

6.2 Experimental design

The research design was based on a partial factorial design examining 4 different manufacturing parameters with five levels each. Ten replications from the different treatments, as well as control specimens, were performed as listed below. Table 1 shows the respective values. Twelve different treatment combinations were processed as represented by the specimen codes. The first letter describes the conditioning time prior to compression, the middle digit shows the speed of compression and the last letter determines the compression time (Table 1).

Combinations carried out are: A-3-C; B-3-C; C-1-C;C-2-C;C-3-A; C-3-B;C-3-C; C-3-D; C-3-E; C-5-C; D-3-C; E-3-C.

Table 1: Parameters used in the different treatments

Conditioning prior to compression	sec	Rate (speed) of compression	Compression speed (mm/min)	Compression time level	sec
A	90	1	61	A	120
B	120	2	40	B	150
C	180	3	29	C	180
D	240	4	14	D	210
E	30	5	7	E	240

6.3 Sample preparation

6.3.1 Wood samples

6.3.1.1 VTC- samples

Air dried samples with a dimension of 240mm by 600mm and a thickness of 4.5mm were processed at the VTC- press with different process schedules. Measurements of weight and dimensions were taken before and after the VTC-treatment. After processing, samples were stored in conditioning at 20°C and 65% relative humidity for three weeks and moisture content was traced until steady EMC was reached.
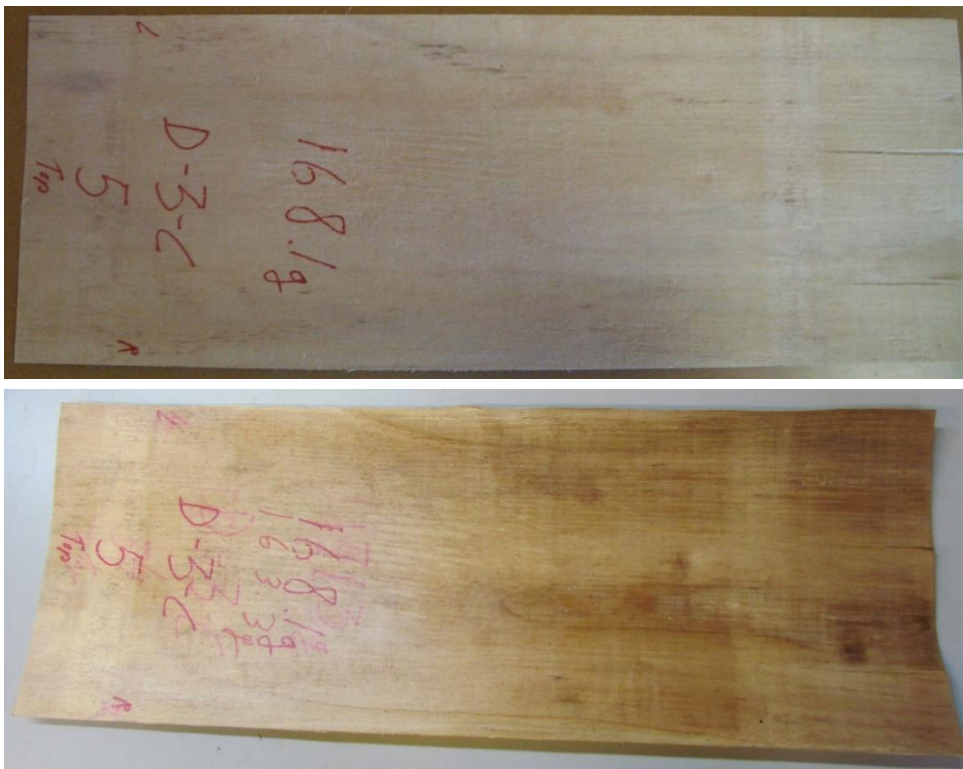
Figure 11: Sample D-3-C, 5 before (top) and after (bottom) pressing

6.3.1.2Control- samples

In order to compare results control samples were separated and not processed. These samples were stored in the conditioning room until EMC was achieved.

6.3.2 Sample cutting

Samples were cut to size for the bending and the shear block test. The dimensions for the bending test sample were 50mm by 200 mm and 50mm by 50mm for the shear block samples. Defects, wet spots, cracks, and visibly destroyed wood were removed (e.g. Figure 12). Usable samples were marked with the process schedule identification and with "B" for bending and "S" for shear block test.

Figure 12 Sizing of VTC samples for bending and shear block test

Between cutting and testing, samples were stored in the conditioning room. Each bending test specimen was tested for modulus of elasticity separately before being glued together to create five layer laminae. The laminated composites were subsequently tested to failure in bending.

6.3.3 Adhesive preparation

Two types of adhesive were used for this experiment. Phenol formaldehyde (PF), GP 3110 RESI- STRAN®; oriented strand board Resin from Georgia Pacific Chemicals LLC, with a solid content of 53%, was used to laminate the 5 layer laminated composites and the shear block samples. This PF resin was chosen because of its low molecular weight and potential to penetrate into the high density VTC. A plywood mixture consisting of 29% PF solid resin, 7.5% extender (Hard Wheat Flour), 7.5% filler (Walnut Shell 100), and 58% water based on percent weight was chosen (Figure 13).

DAP Weldwood Marine Resorcinol two component glue consisting of resin liquid and a catalyst powder was used to laminate the substrate to the shear block test VTC laminae.
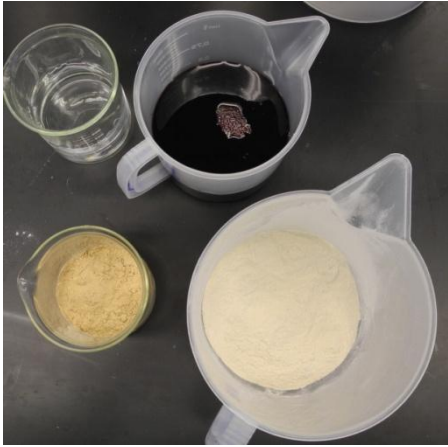
Figure 13: Components for the PF- adhesive mixture

The viscosity of the adhesive was measured with a Brookfield DV- II + Viscometer (Serial number: RT 65840) at 20°C. Viscosity was 500 centerpoise (cP) equates (500 mPa*s).

6.3.4 Adhesive application

A metal roller was used to apply the adhesives. The samples were placed on a covered metal plate and placed on the balance. After the balance was tared, resin was applied as droplets (Figure 14). Due to the high density and the more hydrophobic surface of the VTC wood, the resin did not soak into the wood immediately and equal distribution with the metal roller was possible (Figure 15).

Figure 14: Phenol formaldehyde resin application to VTC- shear test samples (top left); distribution with a metal roller (top right); Adhesive droplets (bottom)

Figure 15 Applied adhesive after allocation

6.3.5 Lamination bending test samples

The 5- layer laminates were produced on an automatic hydraulic hot- press, Model: Auto "M". Pressure applied to the specimens was 7bar and 160°C (top and bottom platen) for five minutes. Aluminum plates for an easy handling and aluminum foil for cleanness were used (Figure 16). Pre-pressing was conducted on a mechanical press with a pressure of 0.5bar for three minutes to allow the resin to contact both sides of the adhesive bond line. The EMC of VTC was between 5% and 7%. Due to water in the adhesive and the slow rate of water adsorption by VTC wood, steam blows in the middle of the laminates occurred (Figure 17), therefore the veneers were dried to zero percent MC.

Figure 16 VTC- laminates layup and gluing; Layup (top left); Layup- adhesive applied (top right); pre-pressing (bottom left); pressing with temperature control (bottom middle); press platen in press position with thermocouple wire (bottom right)

Figure 17 Blows in the middle of the laminate due trapped moisture

6.3.6 Lamination shear test samples

The two layer shear test laminates were pressed in the same press as bending test samples. It was possible to achieve an adequate bond without drying the VTC- veneers. Press time was five minutes at 160 °C with a pressure of 7bar.
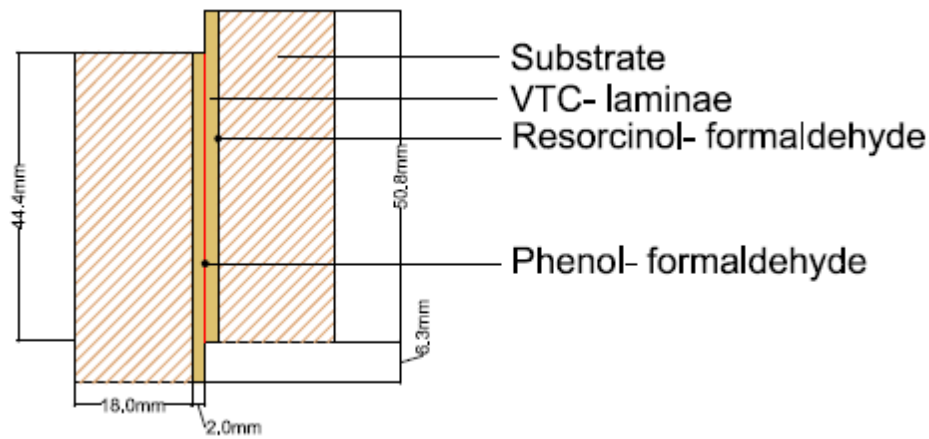
Figure 18: Shear block dimensions according ASTM D 905- 98

Laminates were then produced according ASTM D 905- 98. Due to the availability, oak substrates were used (Figure 19).The substrate was glued with resorcinol adhesive and pressed in a cold press for 12 hours. Sizing of the samples was done on a standard circular saw.

Figure 19 Shear test samples glued on oak substrates after pressing (left) and after cutting (right)

6.3.7 Bending test

The bending test was carried out with a modified form of ASTM D 1037- 99 *"Standard Test Methods for Evaluating Properties of Wood- Base Fiber and Particle Panel Materials"* Span length and speed were adjusted due to thin specimens. The standard requires a width of 50 mm if thickness is less than 6mm. Speed is calculated as follows: $N = zL^2/6d$ where N is the rate of motion in inch per min or mm per min, z is defined as the unit rate of fiber strain (inch per inch;

mm per mm) of outer fiber length per minute (0.005), L is the span length and d the depth (thickness) of specimen in inch or mm. Individual VTC lamina were tested for MOE using a non- destructive method prior to lamination into the 5-layer composites. Span length was 59.2mm and load rate was 0.86mm/ min. After lamination, MOE and MOR were carried out in destructive testing with a span length of 150mm and a loading rate of 2.08mm.

Figure 20: Bending test set up for individual VTC veneer samples (left) and for the 5 layers laminates (middle and right)

6.3.8 Resin loading rates

Former research on VTC processed wood used resin loading rates around 200 g/m^2. A goal of this research was to determine the minimum amount of adhesive that needed to be applied to VTC laminates. VTC specimens were bonded with 85, 170, and 238 g/m^2 for shear and 204 g/m^2 for bending respectively. The mixture was done as described under adhesive preparation. Untreated veneer specimens for the shear test were laminated with the increments listed in Table 2 except 25 g/m^2. Bending test samples for untreated wood were loaded with 50- 80- 100 g/m^2 and VTC bending test samples were bonded with a loading rate of 60 g/m^2.

Table 2: Resin solids loading rates (g/m^2) for bending and shear test laminates

Resin solids loading rates (g/m^2) for bending and shear test laminates			
VTC- shear	VTC- bending	Control- beding	Control- shear
25	-	-	-
50	-	50	50
-	60	-	60
70		-	70
-	-	80	80
-	-	-	90
-	-	100	100

6.3.9 Shear block test

The shear block test was performed according to ASTM D 905- 98 *"Standard Test Methods for Strength Properties of Adhesive Bonds in Shear by Compression Loading"* on a universal testing machine. Speed of compression speed was 5mm/ min.
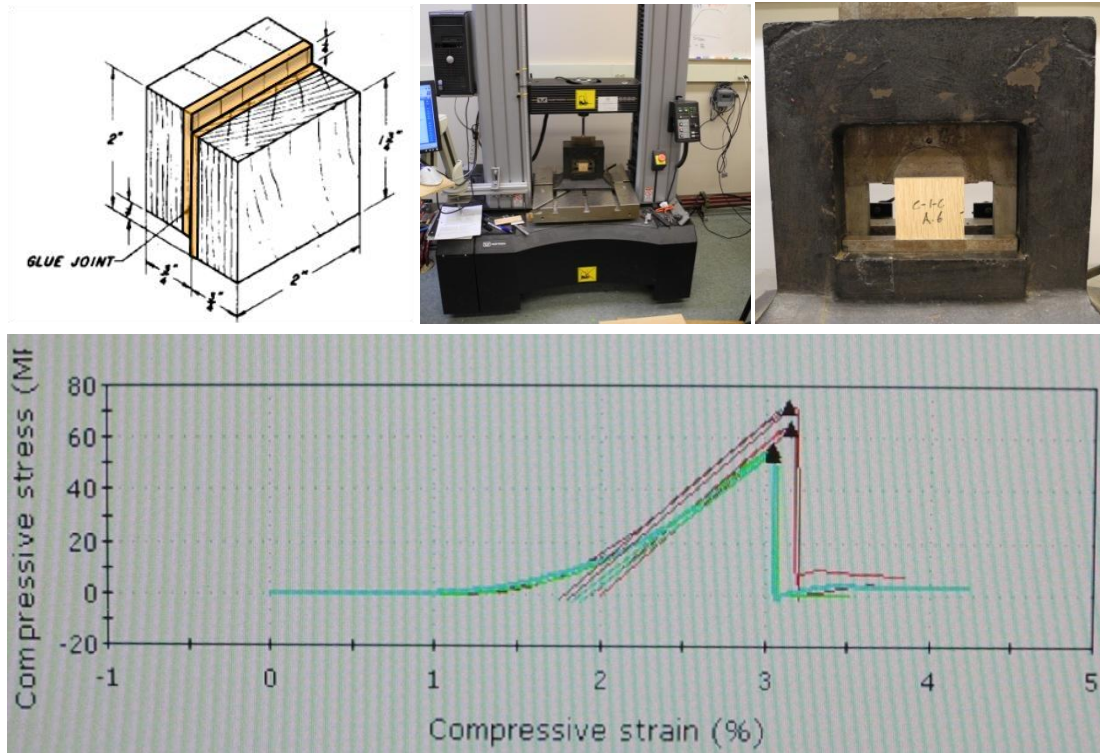
Figure 21: Shear block sample dimensions (top left), set up in the universal testing machine (top middle and right), and screen shot of the shear test figures (bottom)

6.3.10   Determining moisture content

Determining moisture content was done using the oven dry method according to standard ASTM D 4442 (2007). A "Mettler Toledo" PB 1502-S balance was used to measure sample weights. The amount of moisture in wood is ordinarily expressed as a percentage of wood mass when oven-dried.

6.4 VTC process equipment

Oregon State University, together with Oregon Built Environment & Sustainable Technologies Center and Corvallis Tool Company (CTC), invested in a universal hot press adjusted for the novel viscoelastic thermal compression process. The internal press mounted on the upper platen of the large press as well as the chamber sitting on the bottom platen were originally designed and used in a smaller hot press owned by Oregon State University (Kamke and Rathi 2010). Dedicated to the press is a boiler for the steam supply and a connected control

panel equipped with Siemens industrial automation program. The following chapter describes the different parts of the equipment used for the VTC- process.

6.4.1 Universal hot press

The press table includes the table support and the 65 mm thick steel platens with heating elements. The heated steel platens are divided from the table support by a non-compressible high density 20mm insulation material. The table is guided by 4 posts which also hold the non-moveable upper press section. Table area is 1200 mm by 1000 mm and the diameter of the main ram is 580mm, supported by two small rams with a diameter of 76mm arranged diagonally. Heating plates are powered with 480V current and reach a maximum temperature of 500°F.

Figure 22: VTC press unit

6.4.2 Chamber and internal press

The chamber, including the internal press as such, is well described by (Kamke, et al., 2010a):

"*The VTC device was constructed as such to process samples that were 61 cm (24 inch) in length and 25 cm (10 inch) in width. The device mainly consists of three parts: the lid, bellows and an internal press Figure 23. The lid contains the various input and output ports for water, steam, electrical cables and temperature sensors. Also, the lid supports the internal press assembly, transfers compres-*

sion force, and enables the insertion and removal of test specimens. The bellows is a cylindrical stainless-steel structure that consists of a flange, corrugated wall and bottom plate, which together with the lid forms a sealed chamber.
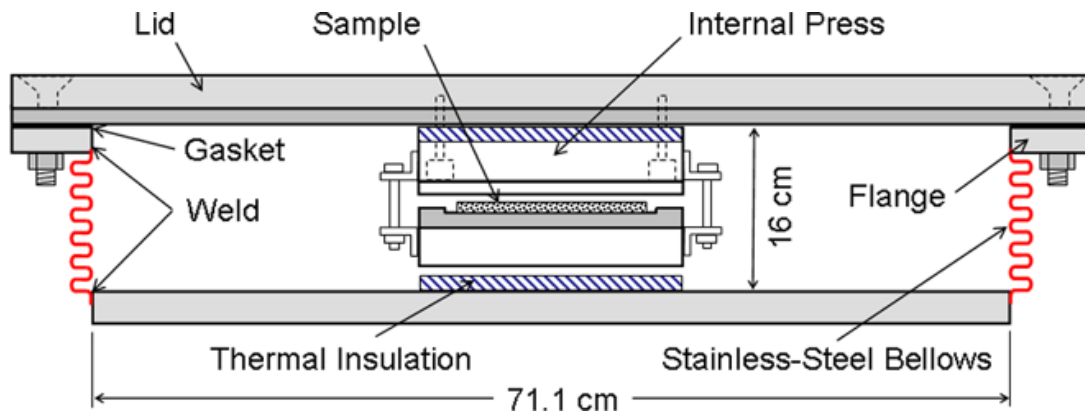
Figure 23 Front view of the VTC device illustrating movable bellows; shown with internal press in open position

*The bellows were designed for axial compression and expansion of approximately 25 mm. The internal press supports the test specimen, imparts direct compression on the specimen, and is independently controlled for heating and cooling. As shown in Figure 23, the top platen of the internal press is attached to the lid while the bottom platen is suspended from the top platen on four rods. Rigid insulation (ZRCI, Florida NY, model RSLE-57), 12 mm thick, thermally isolates the internal press from the lid and the bottom of the bellows. The lid was constructed from a layer of stainless-steel (6 mm, T304) and a layer of mild steel (32 mm, A36), which were then welded together along the circumference. The stainless-steel surface is exposed to the pressurized steam inside the vessel. All access ports were drilled into the edge of the machine steel layer to a depth of 16 cm. These holes intersected with holes drilled perpendicular from the interior of the lid (*Figure 24*). All ports were threaded and sealed to accommodate the mechanical fittings. The electrical lines and thermocouple wires were installed with soft-sealant, feed-through connectors (Conax, Buffalo NY). All water and steam lines were connected via stainless-steel, compression style fittings, with flexible lines to permit movement of the internal press and bellows. Quick-disconnect fittings were used on all steam and water lines.* The lid is fastened to the bellows with a clamp device which is mounted on the VTC- press and secured with two bolts. A synthetic O- ring gasket between the two flanges is used to seal the chamber. *The bellows was obtained from DME Incorporated (Santa Fe CA). It consists of a bottom plate and top flange that are welded to a flexible bellows. The bottom plate is made of T304 stainless-steel, has a diameter of 71 cm (28 inch) and is 32 mm (1.25 inch) thick. The bellows has an internal diameter of 71 cm (28 inch)*

and is made of 1.3 mm (0.05 inch) thick T321 stainless-steel. The bellows is rated for 1 MPa (150 psi) at 200°C (392°F).

The top flange has an outside diameter of 93 cm (36.5 inch), an "internal diameter" of 71 cm (28 inch). The bellows was originally designed as an industrial pipe expansion joint, but bottom flange was removed and replaced with a solid plate. Details of the internal press are shown in Figure 25.
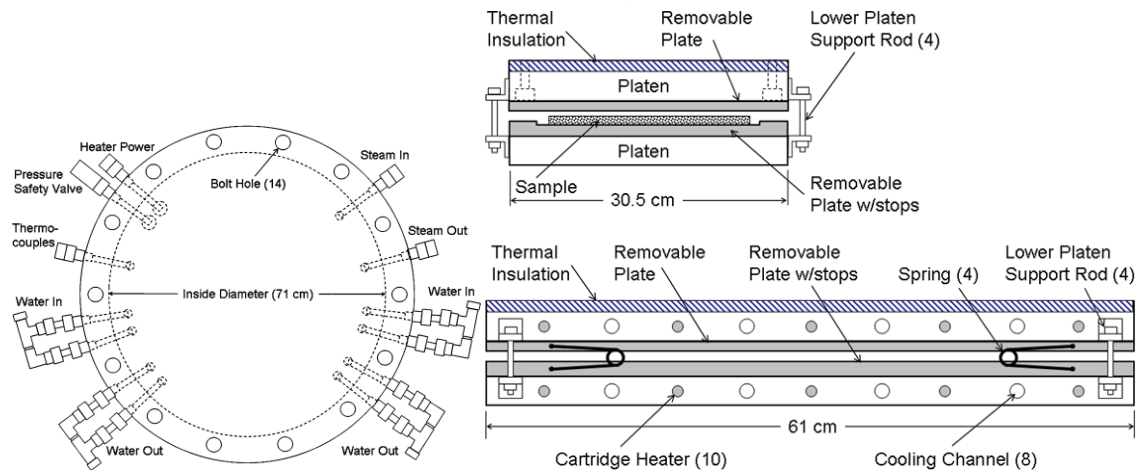
Figure 24 Top view of lid showing ports for water, steam, and electrical connections

Figure 25 Front view (*top*) and side view (*bottom*) of internal press

The platens were made from aluminum (alloy 6061) of dimensions 3.8 cm by 30.5 cm by 61.5 cm. Each platen was bored (13 mm diameter) through the width to create five channels for heater cartridges and four channels for cooling water. A 3 mm diameter hole, centered upon the thickness and width of each platen, was used for insertion of a thermocouple. Five cartridge heaters are installed in each platen. The temperature of the internal top and bottom platens is independently controlled by a proportional-integral-derivative (PID) device (Omega Engineering, Model CN9000A). Removable aluminum plates separate the specimen from the platens. The lower plate is machined to create mechanical stops for thickness control.
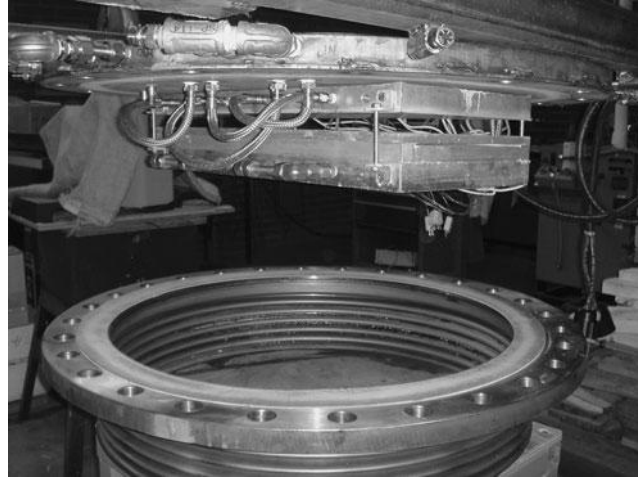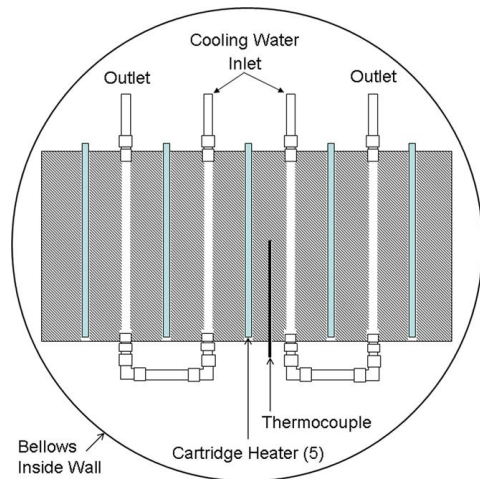
Figure 26 (left): Top view of internal press platen showing cooling and heating apparatus; outline illustrates position of platen within the cylindrical stainless-steel bellows

Figure 27 (right): VTC device shown with the lid open; top platen of internal press is attached to the bottom of the lid and lower platen is suspended by four rods

Although the external press controller could be used to control specimen thickness, the mechanical stops provide more precise thickness control. The removable plates also provide the option to emboss the specimen with a pattern. Four torsion springs support the weight of the upper removable plate. The purpose of the springs is to lift the removable plate and expose the surface of the sample to the environment when the internal press is in the open position. Cooling water lines for the internal press are shown in Figure 26. To increase cooling capacity the inlet line for each platen is split (Figure 24) before it enters the lid, each line supplying cooling water to one platen. Cooling water is then directed to the center of the platen, makes one pass through the platen and then redirected through the outer portion of the platen before exiting the system. At the start of the cooling step the water is flashed to steam when it encounters the hot platen, so it is important to maintain an open exhaust cooling line to avoid build-up of steam pressure inside the cooling lines. A backflow preventer on the inlet cooling water supply line prevents steam from entering the cooling water supply system"

6.4.3 Boiler and water supply

A water boiler, that produces saturated steam, rated to 17.2 bar, is connected to the VTC chamber. Steam input and output is controlled with a high pressure solenoid valve. A manual control needle value is used to restrict steam flow. A manual ball valve may be used to bypass the automatic solenoid valve for venting steam. Cooling water, which is used for the internal platens, comes from the water network available at the location and is also controlled by automatic solenoid valves.

6.4.4 Control panel

The entire process is controlled by a Siemens Industrial Process computer program and is connected as presented in the following schematic (Figure 28).
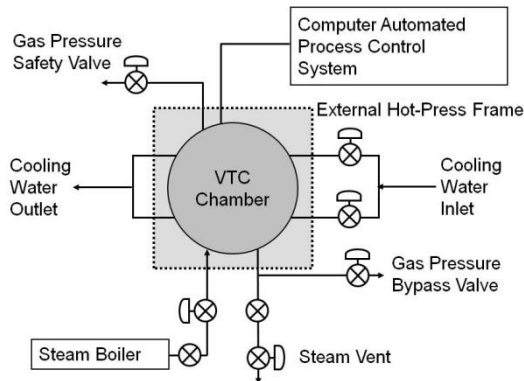
Figure 28 Schematic of control system for VTC device (Kamke, et al., 2010a)

The computer program takes control over the entire process except the steam outlet, which was controlled manually due to technical problems. Heating up of the external platen to 400 °F takes roughly 1 hour and the internal platen takes about 20 min. Figure 29 shows the arrangement of the specimen in the internal press. The internal press platen, which includes the cooling lines, is also shown in Figure 27. Removable platens are used to exchange with different patterns and for easier clean up. These platens are pushed apart with four springs. Stainless aluminum plates were used for better handling. The aluminum foil was necessary to protect the edges of the wood specimen from condensed water.

Arrangement of the specimen in the VTC- press:
- Top internal press platen
- Removable platen
- Stainless steel plate (2mm)
- Aluminium foil
- Wood specimen (~ 3.6mm)
- Aluminium foil
- Stainless steel plate (2mm)
- Removable platen (machined)
- Bottom internal press platen
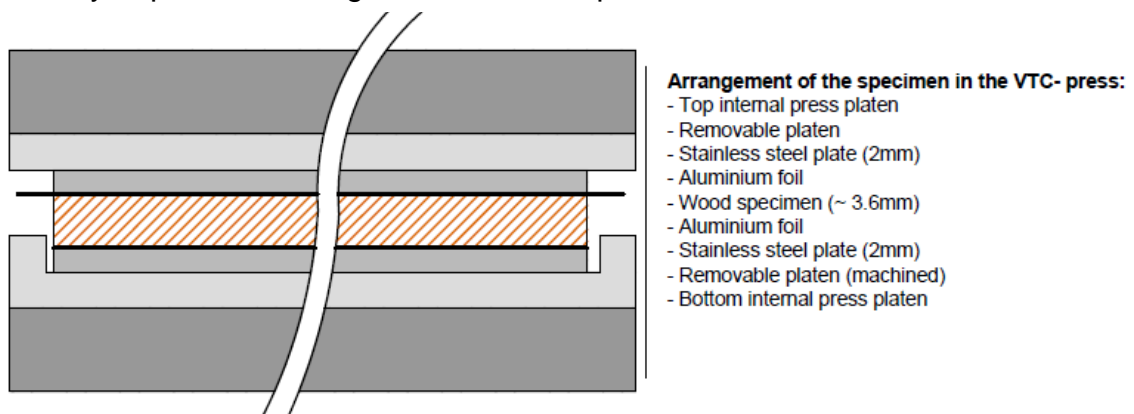
Figure 29: Arrangement of the specimen in the VTC- press (front view)

6.4.5 Process Parameter for operational window

The following chapter describes the parameters used in this experiment. The parameters were chosen based on past research experiences at Oregon state University and published in (Kamke, et al., 2005; Kamke, 2007; Kutnar, et al., 2009; Conner, 2007).

6.4.6 Conditioning time prior to compression

The goal of conditioning is to soften the wood. As described in the literature review, lignin must change from the glassy to the viscous phase in order to compress wood without destroying the cellular structure. Saturated water vapor acts as a conveyer of heat energy into the wood without drying the specimen. Figure 30 shows an ordinary saturated water vapor pressure graph. The red line marks the temperature (170 °C) and pressure (7.9 bar) used in this experiments.
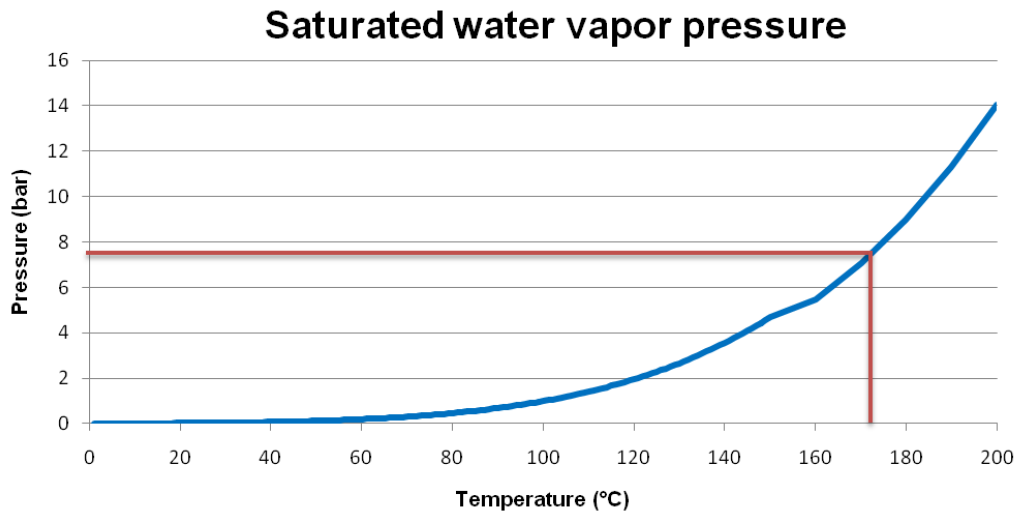
Figure 30: Saturated water vapor pressure graph

6.4.7 Compression time

The necessity of a certain time for relaxation of cell wall polymers in the compressed state, as well as possible thermal degradation reactions, was tested by using five different compression time levels (Table 1). It is defined as the time from closing the press to the final thickness until introducing the cooling step during the compressed state (Figure 3). Depending on the speed of compression, the compression in the final position deviated accordingly due to the slower movement of the press.

6.4.8 Rate (speed) of compression

Wood has a time- dependent relaxation behavior when subjected to load where it changes cell alignment. Compression of wood cells with low speed was expected to result in a similar phenomenon. To verify this phenomenon in the VTC process and how it affects mechanical properties, different levels of compression speed were performed.

6.4.9 Adhesive coverage

Compression of the wood changes the surface characteristics drastically (Kutnar, et al., 2007). The change of the surface energy and their characteristics influences glueability and adhesion between the resin and the VTC processed wood. Previous research has concluded that VTC- processed wood has better bonding qualities compared to non- densified wood (Jennings, 2003). Different adhesive loading levels were applied to the shear block test samples and bonded together.

7 Results and Discussion

7.1 Statistics

The statistics were done with the Winds SDA 6 and an independent group t- test (ANOVA) was used. The confidence level used to determine the statistical significance was 95%.

7.2 Mechanical properties

Wood is an anisotropic material and has different mechanical properties in the longitudinal, tangential and radial directions. In this research wood was compressed in the radial direction and properties modified respectively. MOE, MOR, as well as glueline shear strength, were carried out at moisture contents between 3 and 5%.

7.2.1 Modulus of elasticity (MOE)

7.2.1.1 Influence of densification

Kollmann, et al., (1968) describes the influence of density to the modulus of elasticity. As seen in Figure 31, a strong correlation between MOE and density occurs. Control samples were used to determine the density before compressing. An average MOE of untreated samples was found to be 3.9 GPa (s.d. 683 MPa) at an average density of 0.3 g/cm^3 at 12% MC. It was not the intention of this research to compress to different degrees of densification. A cooling problem on one side of the internal platen could not be solved for this study. In addition, a higher than desired end of process moisture content caused a higher thickness recovery. A VTC wood density of 0.9 g/cm^3 means a densification of approximately 200%. The strong linear correlation of VTC densified wood was also found by Kamke, et al., (2005).

Table 3: Description of the data presented in the Figure 31, MOE as a function of density

	Density (g/m^3)	MOE (MPa)	Thickness (mm)	Quantity
mean	0.67	11492.61	2.00	641
s.d.	0.11	3298.23	0.31	
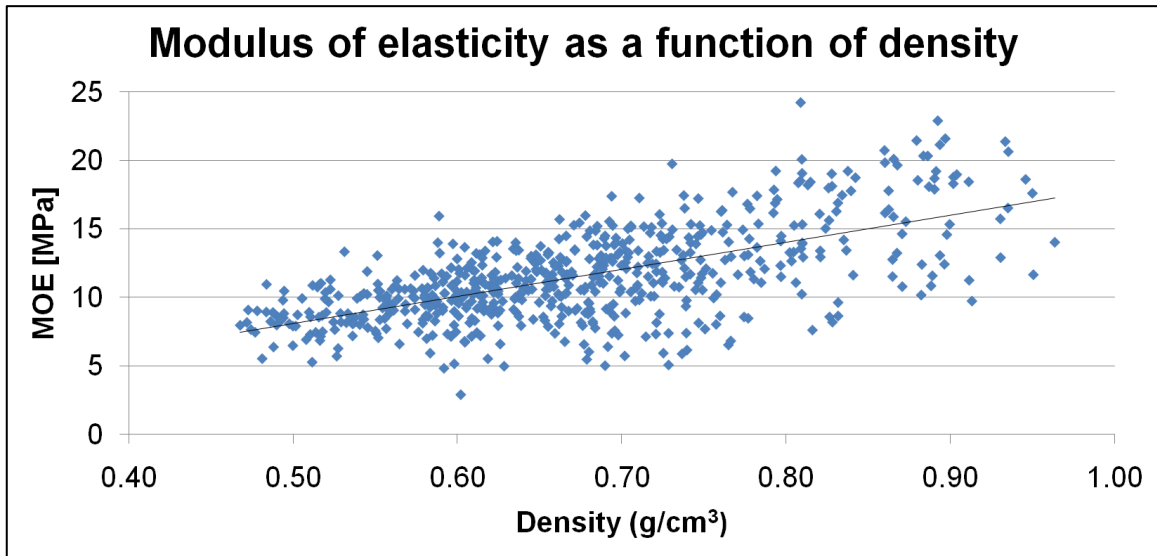
Figure 31: Modulus of elasticity versus density (all separated specimens from all treatments)

7.2.1.2 Influence of conditioning time

The results from the individual specimen presented in Figure 32 show a significant influence of the conditioning time to the modulus of elasticity. According to the statistics, the conditioning time of 30 and 120, as well as the group 180, and 240 seconds, does not show significant differences from each other. However, the two groups are different from each other. The conditioning time of 90 seconds showed a significant lower MOE. One might expect a longer conditioning time would improve MOE, since a greater degree of softening would occur prior to compression, and thus less damage to cell walls would occur. The 30 second conditioning time did not follow the expected trend. The reason for this behavior is not clear. Perhaps the densification in the specimens conditioned for 30 seconds was not uniform, with greater density near the surface. If this were the case, a flatwise bending test would yield higher MOE. Extending the conditioning time increases MOE, which plateaued after 180 seconds. Furthermore, the variability at the higher conditioning time levels is considerably higher than the first three levels. The influence of the conditioning time to MOE on the laminates (Figure 33) does not show a significant difference. A conditioning time of 90s does not differ from the other and thus follows the trend contradictory to Figure 32.

Table 4: Description of the data presented in Figure 32; MOE as a function of conditioning time

Schedule	E-3-C		A-3-C		B-3-C		C-3-C		D-3-C	
Conditioning time (s)	30		90		120		180		240	
Quantity	50		50		52		53		51	
	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)
mean	0.63	11349.18	0.75	8099.63	0.66	11400.77	0.74	13801.98	0.73	13947.39
s.d.	0.05	1833.40	0.10	2386.92	0.07	2838.81	0.12	4323.48	0.09	3293.49
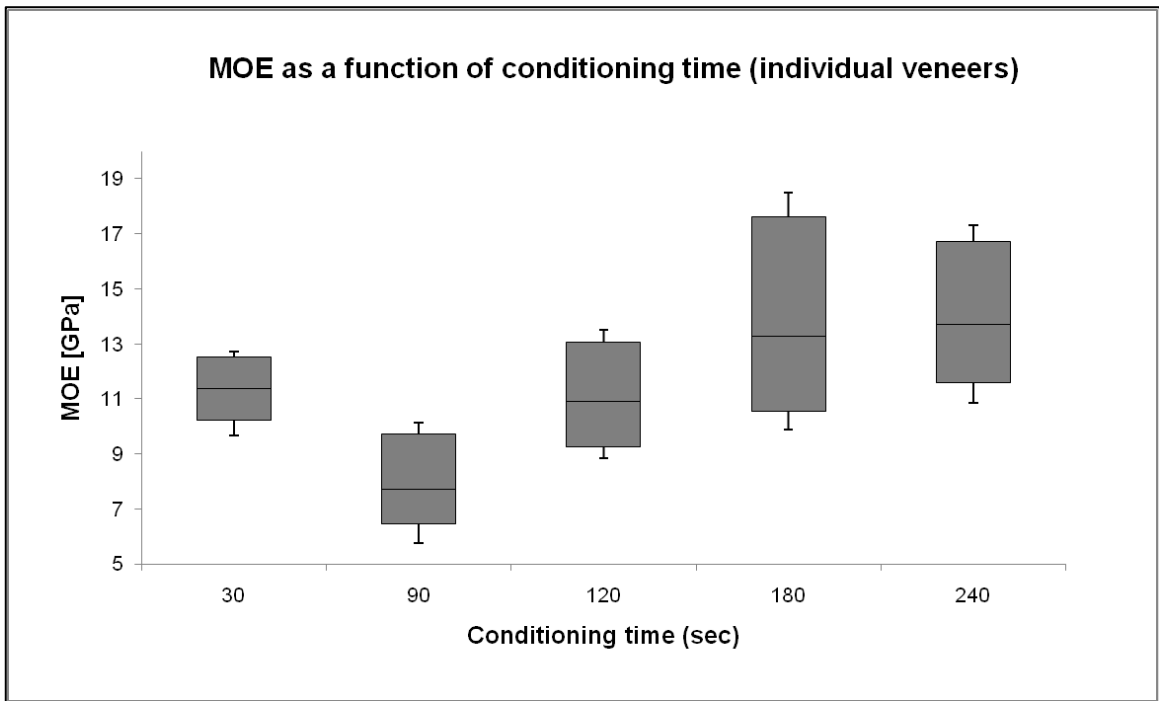
Figure 32: Effect of conditioning time to modulus of elasticity, (compression speed: 29mm/ min; Compression time: 180 seconds)

Table 5: Description of the data presented in Figure 32, Figure 32MOE versus conditioning time (5- layer laminates)

Schedule	E-3-C		A-3-C		B-3-C		C-3-C		D-3-C	
Conditioning time (s)	30		90		120		180		240	
Quantity	10		10		10		10		10	
	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)
mean	0.63	9799.92	0.75	11977.95	0.66	11948.44	0.75	11425.77	0.73	12582.83
s.d.	0.02	1436.74	0.07	2583.03	0.05	1426.29	0.10	4222.24	0.07	2526.20
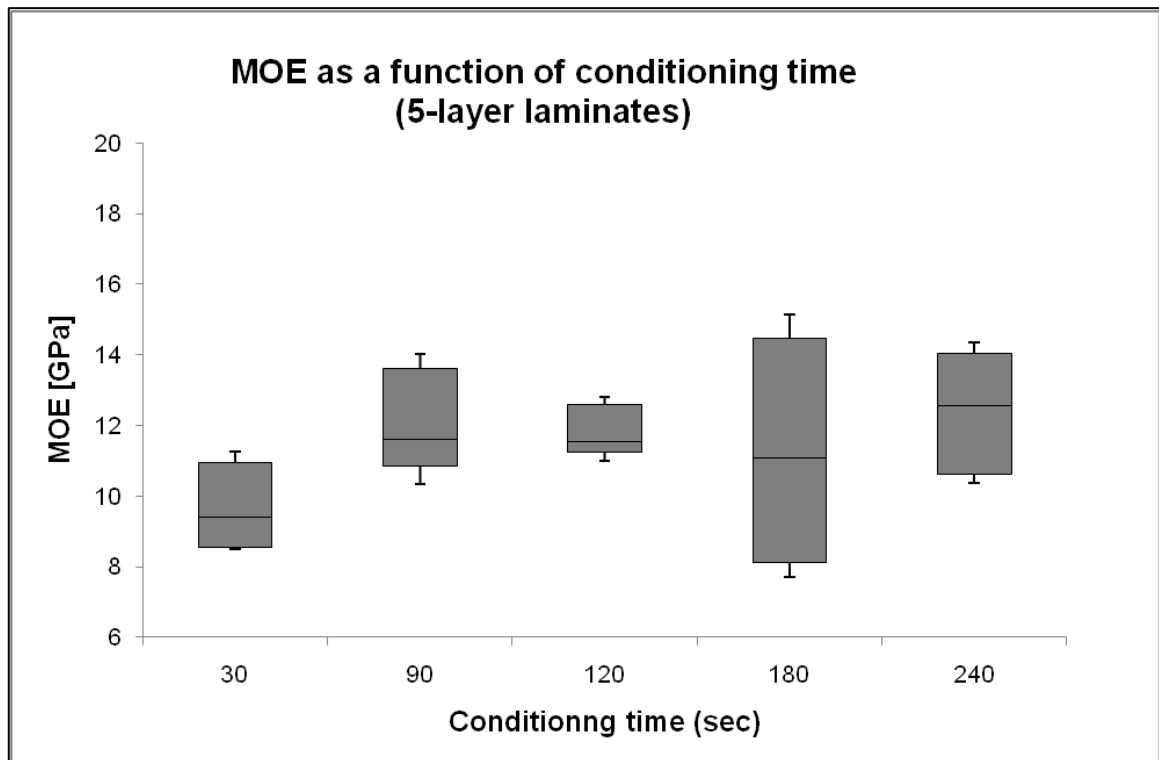
Figure 33: Effect of conditioning time to modulus of elasticity - 5- layer laminates, (compression speed: 29mm/ min; Compression time: 180 seconds)

7.2.1.3 Influence of rate of compression

The following box plots (Figure 34 and Figure 35) present the effect of load rate. Statistical comparisons of the averages with the independent t-test show a significant (95%) difference between 7, 61mm/min and 14 and 29mm/min compression speed levels at the individual specimens. However, the figure, Figure 34 also displays a gradual increase in MOE and an abrupt decline after passing a speed level of 29 mm/min. At higher rate of loading wood exhibits more brittle behavior, which may result in microfractures in the cell wall and, consequently, loss of MOE. The modulus of the cell wall is described by Kamke, et al., (2010b) which might also be influenced be the rate of compression. Laminates showed similar results although only the fastest speed level differs significantly from the others.

Table 6: Description of the data presented in Figure 34, MOE as a function of rate of compression

	MOE versus rate of compression							
Schedule	C-1-C		C-2-C		C-3-C		C-5-C	
Rate of compression (mm/min)	7		14		29		61	
Quantity	60		47		116		111	
	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)
mean	0.61	10290.70	0.72	12348.81	0.74	13801.98	0.55	9131.71
s.d.	0.07	2106.94	0.10	3235.00	0.12	4323.48	0.05	1481.41

MOE as a function of rate of compression (individual veneers)
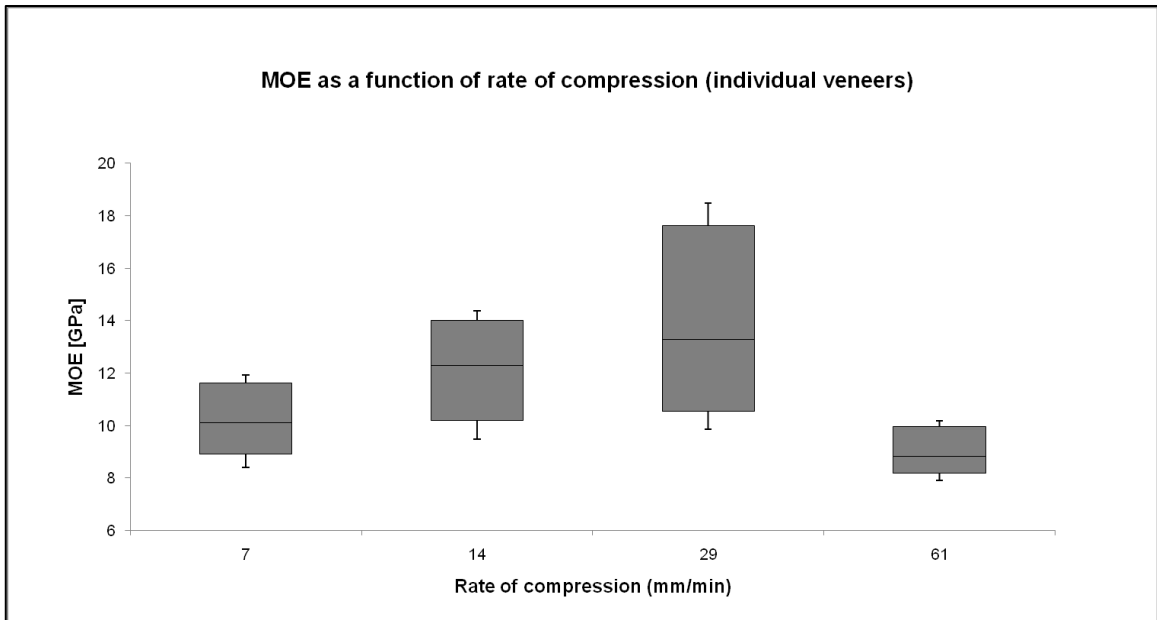
Figure 34: Effect of rate of compression to the modulus of elasticity (conditioning time:180s; compression time: 180sec)

Table 7: Description of the data presented in Figure 34, MOE as a function of compression

Schedule	C-1-C		C-2-C		C-3-C		C-5-C	
Rate of compression (mm/min)	7		14		29		61	
Quantity	10		9		10		10	
	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)
mean	0.61	12145.85	0.73	13255.03	0.75	11425.77	0.55	8305.47
s.d.	0.05	1614.26	0.09	1591.93	0.10	4222.24	0.03	919.12
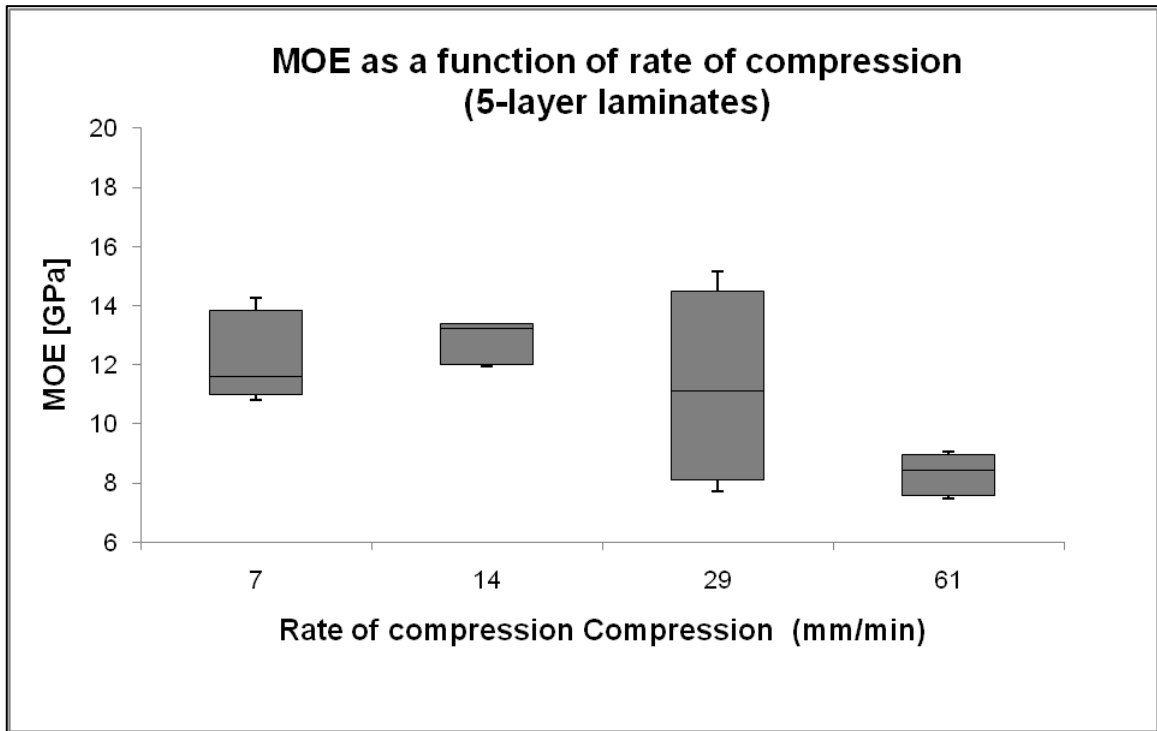
Figure 35 : Effect of rate of compression to the modulus of elasticity- 5- layer laminates (conditioning time: 180s; compression time: 180sec)

7.2.1.4 Influence of compression time

Figure 36 and Figure 38 show the influence of the compression time to the modulus of elasticity. There was no difference between compression time of 120 s and 150 s. For compression time of 180s and longer there was an increase of MOE. A compression time of 180 seconds resulted in the highest MOE as well as larger variability.

Table 8: Description of the data presented in Figure 36, MOE as a function of compression time (individual samples)

Schedule	C-3-A		C-3-B		C-3-C		C-3-D		C-3-E	
Compression time (s)	120		150		180		210		240	
Quantity	62		52		53		59		42	
	Density (g/m³) at 0% MC	MOE (MPa)	Density (g/m³) at 0% MC	MOE (MPa)	Density (g/m³) at 0% MC	MOE (MPa)	Density (g/m³) at 0% MC	MOE (MPa)	Density (g/m³) at 0% MC	MOE (MPa)
mean	0.67	10592.30	0.59	11531.21	0.74	13801.98	0.68	12029.03	0.72	13262.76
s.d.	0.11	3357.58	0.10	2781.82	0.12	4323.48	0.07	2601.85	0.08	2667.36
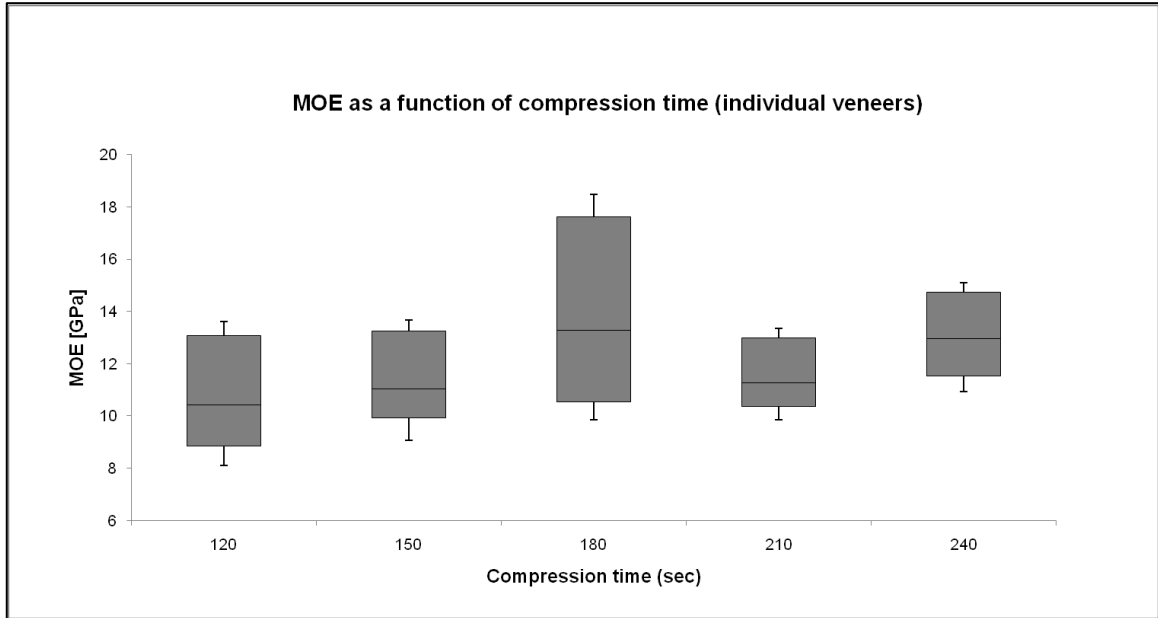
Figure 36: Effect of compression time to the modulus of elasticity (compression speed: 29mm/min; conditioning time: 180sec)

Table 9: Description of the data presented in Figure 36, MOE as a function of conditioning time (5- layer laminates)

Schedule	C-3-A		C-3-B		C-3-C		C-3-D		C-3-E	
Conditioning time (s)	120		150		180		210		240	
Quantity	10		10		10		10		8	
	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)	Density (g/m^3) at 0% MC	MOE (MPa)
mean	0.68	8025.99	0.59	9841.09	0.75	11425.77	0.67	11220.43	0.72	13948.75
s.d.	0.10	3122.26	0.08	2523.70	0.10	4222.24	0.04	1018.33	0.08	3437.73
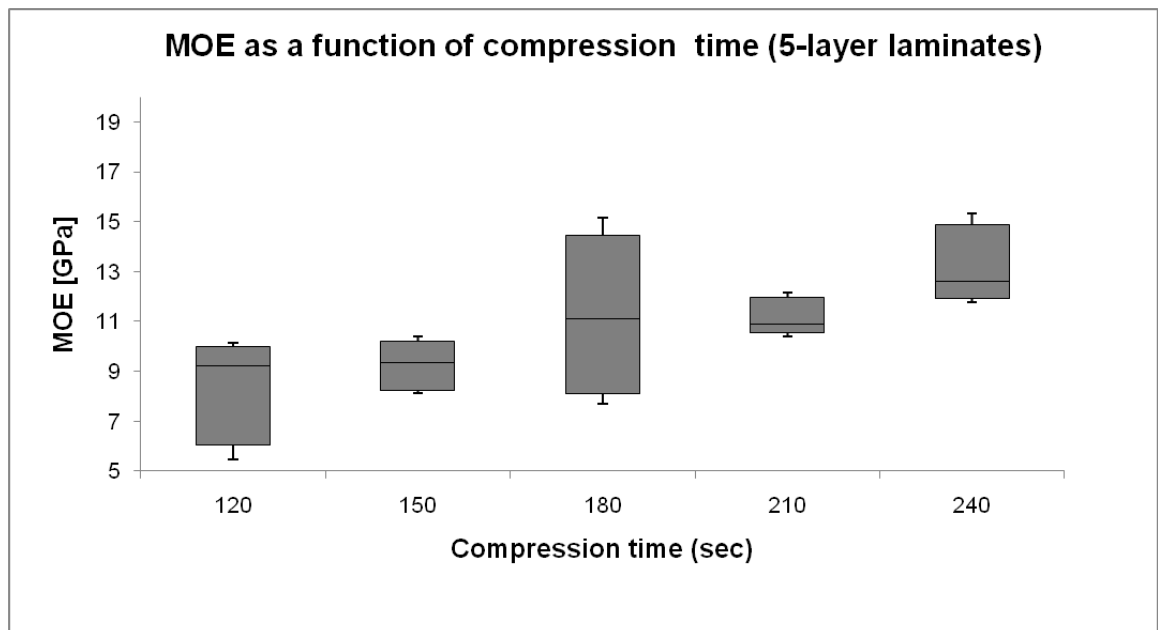
Figure 37: Effect of compression time to the modulus of elasticity – 5 layer laminates (compression speed: 29mm/min; conditioning time: 180sec)

7.2.2 Modulus of rupture

7.2.2.1 Influence of densification

A correlation between modulus of rupture and density, as it is presented in Figure 38, is well known (Kollmann, et al., 1968; Kretschmann, 2010). Modulus of rupture for untreated hybrid poplar wood was found to be 41MPa (s.d.= 5) at an average density of 0.3g/cm^3. Therefore a densification to 0.6g/cm^3 means an enhancement in MOR of two to threefold. Large variation between MOR and density did occur.
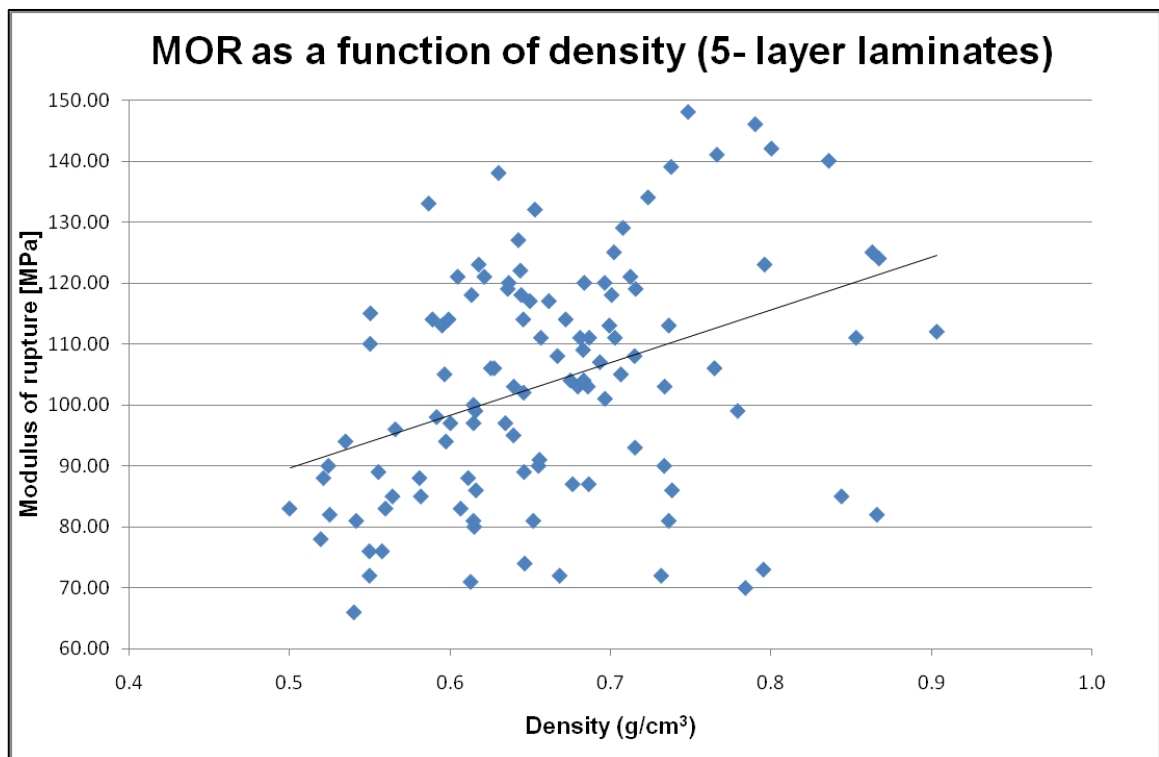
Figure 38: MOR as a function of density for all VTC treated specimens (n= 118)

7.2.2.2 Influence of conditioning time

Homogenous groups, according to a 95% confidential level, are found between 90, 120, and 240 sec and between 30, 90, and 180sec. As presented in Figure 39, a conditioning time of 180 seconds yields lower MOR values. Possible reasons for this occurrence might be a lower steam pressure (90psi) while softening the specimens due to technical issues. This might have caused drying and consequently more cell wall fracture in the densification stage.

Table 10: Description of the data presented in Figure 39, MOR as a function of conditioning time (5- layer laminates)

Schedule	E-3-C		A-3-C		B-3-C		C-3-C		D-3-C	
Compression time (s)	30		90		120		180		240	
Quantity	10		11		12		13		14	
	Density (g/m^3) at 0% MC	MOR (MPa)	Density (g/m^3) at 0% MC	MOR (MPa)	Density (g/m^3) at 0% MC	MOR (MPa)	Density (g/m^3) at 0% MC	MOR (MPa)	Density (g/m^3) at 0% MC	MOR (MPa)
mean	0.63	105.20	0.75	111.30	0.66	114.40	0.75	104.90	0.73	116.00
s.d.	0.02	18.78	0.07	25.62	0.05	11.57	0.10	26.34	0.07	18.79
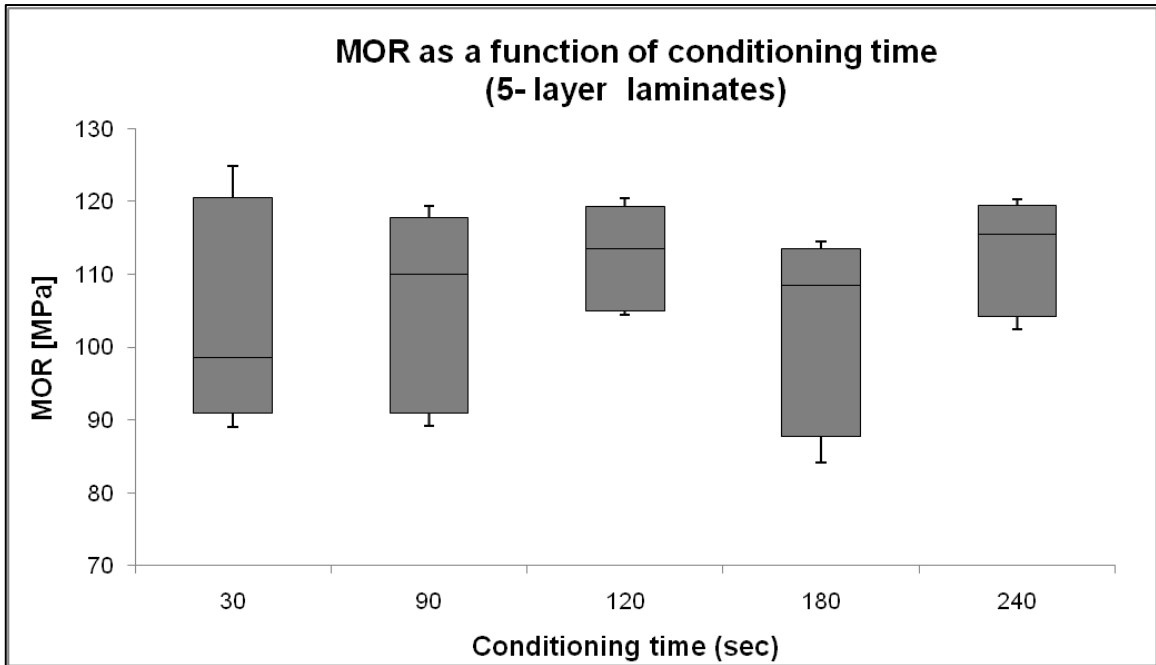
Figure 39 Effect of conditioning time to the modulus of rupture (compression speed: 29mm/min; compression time: 180sec)

7.2.2.3 Influence of rate of compression

Figure 40 represents the influence of rate of compression to the modulus of rupture. Rate 7, 14 and 29 mm/min do not show a significant difference whereas rate 61 mm/min differs significantly. Similarly to the effect of rate of compression to MOE, the MOR declines at higher compression speed levels which might be caused by cell wall fracture.

Table 11: Description of the data presented in Figure 40, MOR as a function of rate of compression (5- layer laminates)

MOR versus rate of compression								
Schedule	C-1-C		C-2-C		C-3-C		C-5-C	
Rate of compression (mm/min)	7		14		29		61	
Quantity	10		10		10		10	
	Density (g/m^3) at 0% MC	MOR (MPa)	Density (g/m^3) at 0% MC	MOR (MPa)	Density (g/m^3) at 0% MC	MOR (MPa)	Density (g/m^3) at 0% MC	MOR (MPa)
mean	0.61	110.30	0.73	118.11	0.75	104.90	0.55	86.30
s.d.	0.05	16.06	0.09	15.41	0.10	26.34	0.03	12.39
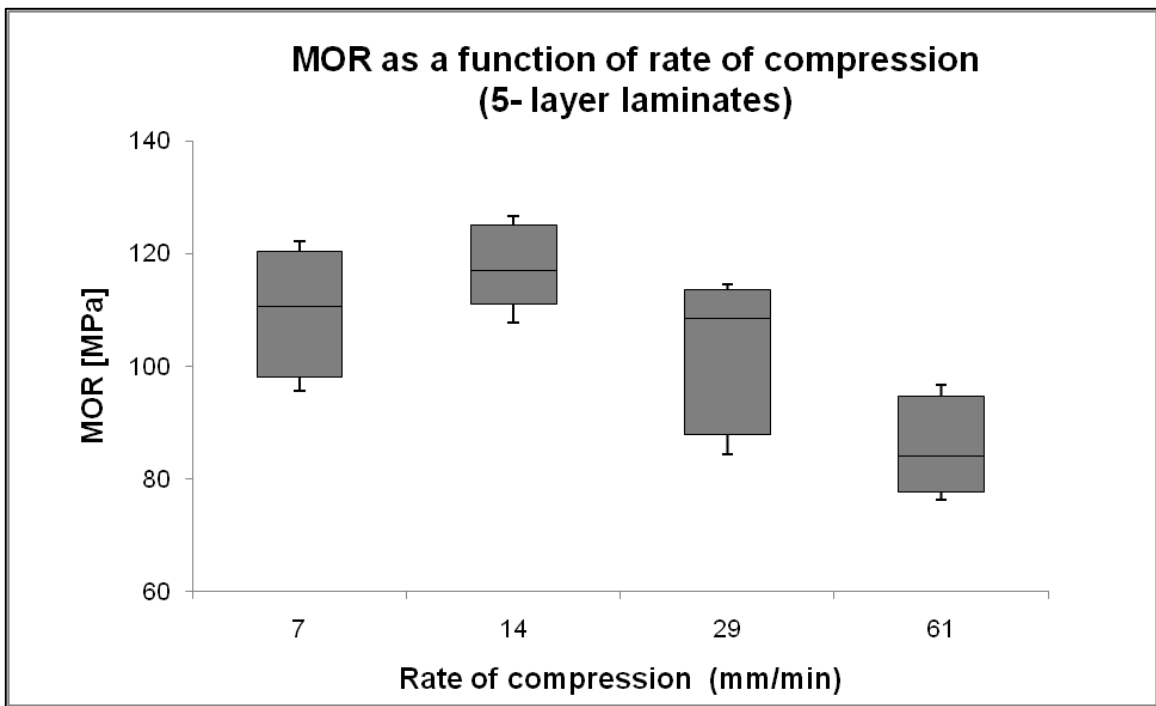
Figure 40: MOR as a function of the rate of compression (conditioning time: 180 sec.; compression time: 180 sec)

7.2.2.4 Influence of compression time

The influence of compression time to modulus of rupture is presented in Figure 41. Homogenous groups were found between time levels 120 and 150, between 150, 180, and 210 and between 180, 210, and 240. As it appears in this box plot, a gradual increase of MOR can be achieved by increasing the compression time, but cannot be confirmed with literature. Longer compression time might reduce the thickness recovery which yields higher density and consequently higher MOR values.

Table 12: Description of the data presented in Figure 41, MOR as a function of compression time (5-layer laminates)

Schedule	C-3-A		C-3-B		C-3-C		C-3-D		C-3-E	
Compression time (s)	120		150		180		210		240	
Quantity	10		11		12		13		14	
	Density (g/m^3) at 0% MC	MOR (MPa)	Density (g/m^3) at 0% MC	MOR (MPa)	Density (g/m^3) at 0% MC	MOR (MPa)	Density (g/m^3) at 0% MC	MOR (MPa)	Density (g/m^3) at 0% MC	MOR (MPa)
mean	0.67	78.82	0.59	92.30	0.75	104.90	0.67	106.40	0.72	116.00
s.d.	0.10	21.52	0.08	21.65	0.10	26.34	0.04	11.66	0.08	32.18
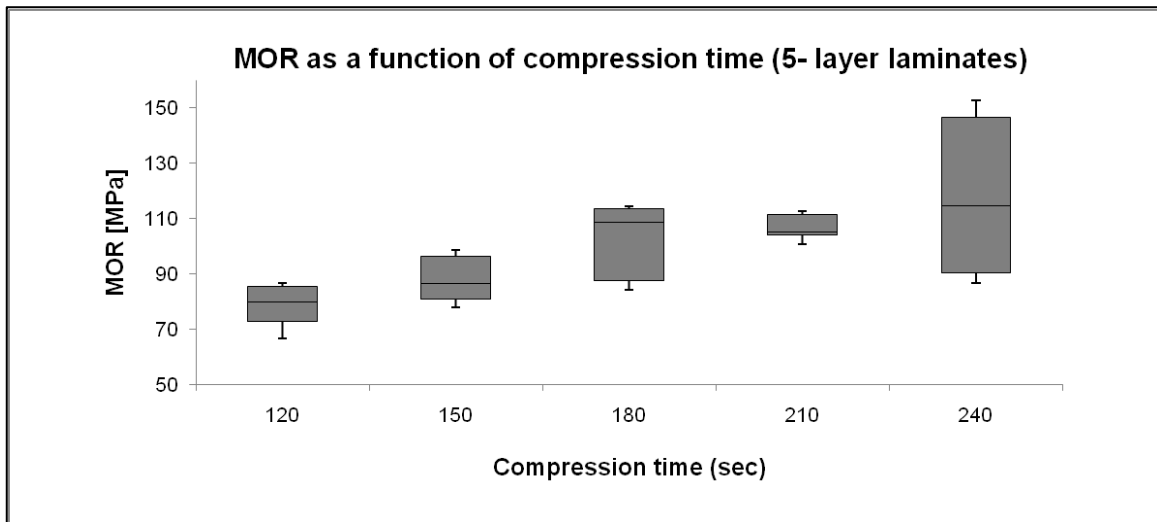
Figure 41: MOR as a function of compression time (conditioning time: 180 sec.; rate of compression: 29mm/min)

7.2.2.5 Shear test

The shear block test was carried out as described under the methods and materials section. In several cases a delamination between the VTC wood and the oak substrate did occur. Resorcinol formaldehyde resin was used. The resin was applied first to the oak substrate and then pressed together with the shear specimen. It appeared that the resin was taken up by the substrate too fast and more easily than compared to the VTC wood. However, samples with substrate glue line failure were excluded from statistical analysis. Untreated, non-densified hybrid poplar bonded with phenol formaldehyde resin and a loading rate of 80 g/cm^2 yield a shear strength of 28MPa (s.d.= 9.7) at a density of 0.3g/cm^3. In all cases the glueline shear values were significantly greater for the VTC specimens in comparison to the untreated specimens.

7.2.2.6 Influence of conditioning time

Conditioning time affects the shear strength of VTC processed hybrid poplar (Figure 42). The largest conditioning time showed a shear strength that was lower than the other treatments. Conditioning time levels 30 through 180 do not show a significant difference and are ranked as homogenous groups in contrast

to level 240. Previous literature has shown that extending exposure time of wood to high temperature will reduce surface energy, and therefore, bonding with aqueous adhesives may be less favorable.

Table 13: Description of the data presented in Figure 42, shear strength as function of conditioning time

Conditioning time (s)	30	90	120	180	240
Quantity	30	14	30	27	42
	Shear failure (MPa)	Shear failure (MPa)	Shear failure (MPa)	Shear failure (MPa)	Shear failure (MPa)
mean	60.54	60.62	64.35	65.40	55.22
s.d.	10.60	12.90	9.06	11.00	12.04
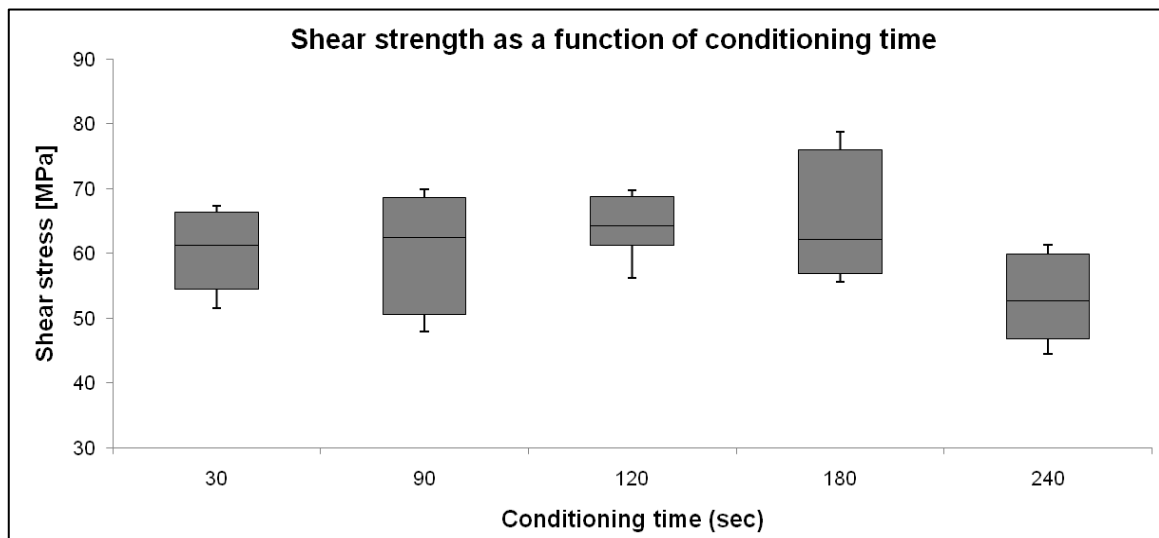
Figure 42: Shear failure stress as a function of conditioning time; rate of compression: 29mm/min; compression time: 180s

7.2.2.7 Influence of rate of compression

The plot (Figure 43) below displays the shear failure stress depending upon compression time. No significant difference between the groups could be found.

Table 14: Description of the data presented in Figure 43, shear strength as a function of rate of compression

Rate of compression (mm/min)	7	14	29	61
Quantity	28	11	5	17
	Shear failure (MPa)	Shear failure (MPa)	Shear failure (MPa)	Shear failure (MPa)
mean	28.99	38.00	41.69	47.42
s.d.	29.92	29.84	44.07	48.78

Figure 43: Shear strength as a function of the rate of compression; (conditioning time: 120s, compression time: 180s)

7.2.2.8 Influence of compression time

Homogenous groups are ranked between the compression time levels 120, 150, 210, and 240, and between the levels 120, 150, 180, and 240. However, as it is displayed in Figure 44 , no trend can be observed depending upon the compression time.

Table 15: Description of the data presented in Figure 44, shear strength as a function of compression time

Compression time (s)	120	150	180	210	240
Quantity	5	25	27	27	29
	Shear failure (MPa)	Shear failure (MPa)	Shear failure (MPa)	Shear failure (MPa)	Shear failure (MPa)
mean	60.54	60.62	64.35	65.40	55.22
s.d.	10.60	12.90	9.06	11.00	12.04
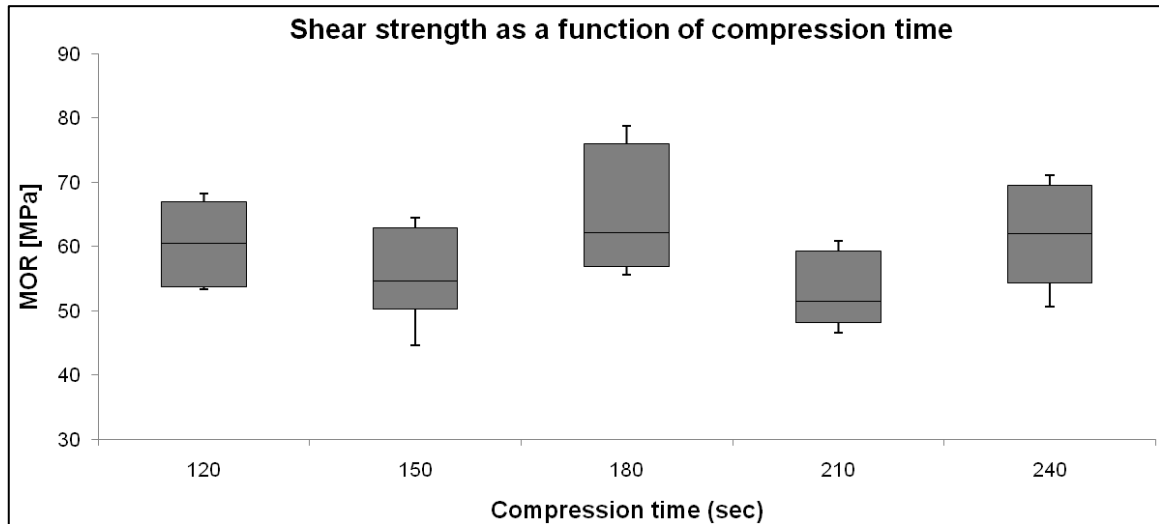
Figure 44: Shear strength as a function of compression time (conditioning time: 180s, rate of compression: 29mm/min)

7.2.2.9 Influence of adhesive coverage

Figure 45 shows glue line failure stress depending on the solid resin loading level. Loading level 25g/m^2 differs significantly from loading levels 50 and 70g/m^2. The failure stress for the untreated hybrid poplar was found to be about 50 percent compared to the densified samples. Recall that the untreated specimens had a resin loading level of 80 g/m^2. While more adhesive was used for the untreated specimens, the glueline shear strength was less than the VTC specimens. This box plot presents four VTC- treatments, namely C-2-C; C-3-A; A-3-C; C-5-C (see Table 1) and the untreated samples. Due to a small amount of glue line failure in the other treatments, a comparison between the different VTC treatments was not reasonable.

Table 16: Description of the data presented in Figure 45, shear strength as a function of the resin solid loading

Resin solid loading rate (g/cm^2)	25	50	70	80 (Untreated)
Quantity	22	25	22	9
	Shear failure (MPa)	Shear failure (MPa)	Shear failure (MPa)	Shear failure (MPa)
mean	45.61	61.67	57.61	27.30
s.d.	16.36	16.47	14.99	9.95
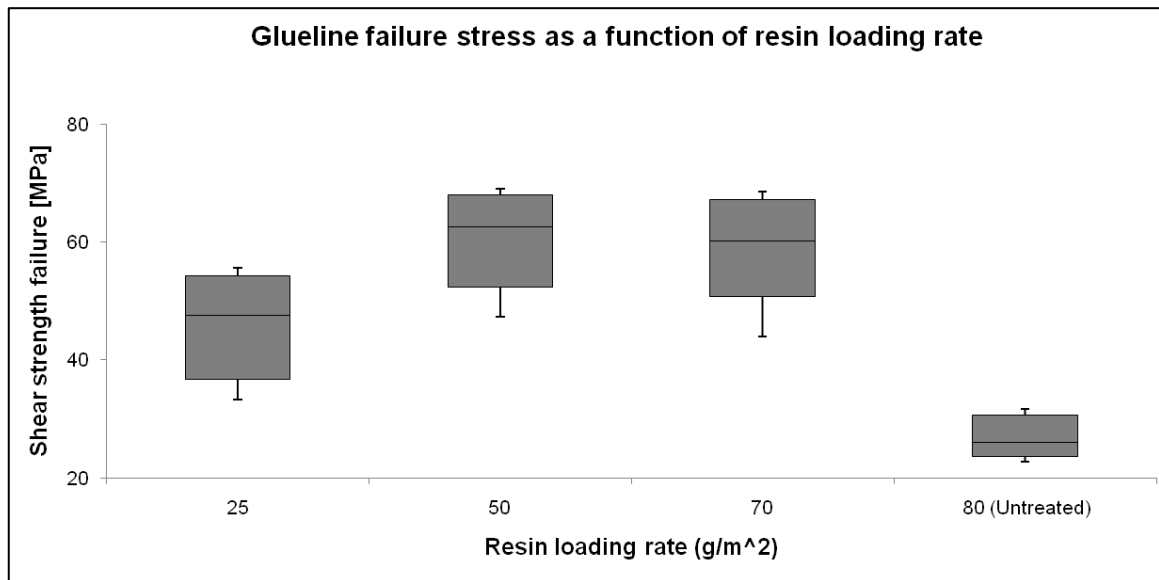
Figure 45: Glue line failure stress as a function of the resin loading rate

8 Conclusion

Densification of hybrid poplar wood with viscoelastic thermal compression enhances modulus of elasticity, modulus of rupture and shear glueline strength. In general, MOE, MOR, and the shear strength increase almost in a linear correlation with the degree of densification. Conditioning time up to 180 seconds increases the MOE, whereas no trend in the MOR could be observed. According to average shear strength values as a function of conditioning time, a peak can be seen at 120 seconds and declines significantly after a conditioning time of 180 seconds. Rate of compression improves the MOE gradually from the lowest level (7mm/min) up to 29mm/min and yields much lower values at a compression speed of 61mm/min. Higher rates of compression lowered the MOR which might be caused by cell wall fracture. No trend could be found between compression time and MOE whereas a compression time of 120 and 150 seconds results in lower MOR compared the time levels 180, 210, and 240 seconds. Annealing appears to be taking place. Neither the rate of compression nor the compression time influences the shear strength significantly. The resin coverage of $25g/m^2$ (PF resin solids) yields lower glue line stress failure strength compared to 50 and 70 g/m^2. The latter do not exhibit any significant difference which indicates that the optimum resin loading rate must be somewhere between 25 and 50 g/m^2.

9 Future prospects

This is the first time the VTC- machine was used in a research project. Several technical problems occurred during the production of the samples which probably caused some variability of the results. However, except the automatic controlled steam exit and a reduced cooling rate, all problems could be solved. At the time of experimentation, continuous data saving was not possible. This, as well as the reduced cooling rate, was solved after the current experiment was completed.

9.1 Material

Hybrid poplar was used in this project. To gain knowledge about the influence of the different species to the parameters, different wood species must be tested to verify a general behavior or to confirm differences.

9.2 Equipment

The present software does save data continuously although the file must be converted and the spreadsheet adjusted in a time consuming step. An adequate data saving tool would be helpful. Furthermore, installations of multiple thermocouples in the chamber are necessary to adjust the process parameters according to the temperature of the wood.

9.3 Tests

A swelling test would be appropriate to determine whether or not an effect of the different parameters occurs. An examination of the densified wood would explain effects of different compression speed levels to confirm observed trends.
Chemical analysis of the densified wood is necessary to determine the degree of degradation at certain process parameters.

10 Bibliography

Balatinecz, John, et al. 2010. *POPLARS AND WILLOWS IN THE WORLD, CHAPTER 10: PROPERTIES, PROCESSING AND UTILIZATION.* s.l. : Forestry Department, Food and Agriculture Organization of the United Nations, 2010.

Bergman, Richard. 2010. *Wood Handbook- Wood as an Engineering material Chapter 14: Drying and Control of Moisture Content and Dimensional Changes.* s.l. : United Stated Department of Agriculture, 2010.

Blakenhorn, P.R., et al. 1985a. *Comparison of selected fuel and chemical content values for seven Populus hybrid clones.* s.l. : Journal of Material Science, 1985a. 36, 3777–3783.

Blomberg, Jonas, Persson, Bengt und Blomberg, Anna. 2005. Effects of semi-isostatic densification of wood on the variation in strength properties with density. *Wood Science and Technology.* 2005.

Boonstra, M.J., Tjeerdsma, B.F. und Groenveld, H.A.C. 1998. *Thermal modification of non durable wood species 1. The Plato tecnology; thermal modification of wood.* 1998.

Calignum. 2010. www.calignum.com. [Online] 2010. [Zitat vom: 23. 02 2011.] http://www.calignum.com/default.asp?V_DOC_ID=858.

Chang, C.I. und Keith, C.T. 1978. *Properties of heat darkened wood, II. Mechanical properties and gluability.* Canada : Eastern Forest Product laboratory, 1978.

Chastagner, Gary A. und Hudak, Joseph M. 1999. *Crop Profile for Hybrid poplars in Washington & Oregon.* s.l. : Washington State University, 1999.

Conner, John. 2007. *Improving Wood Strength and Stiffness through Viscoelastic Thermal Compression.* Oregon State University Wood; Wood Science and Engineering : Master thesis, 2007.

Dogu, Dilek, et al. 2010. *Anatomical investigation of thermally compressed wood panels.* s.l. : BioResources, 2010. 5 (4), 2640- 2663.

Dwianto, W. 1999. *Mechanism of permanent fixation of radial compressive deformation of wood by heat or steam treatment.* Kyoto : Wood Research Institute, 1999.

Eastin, Ivan, Hendrickx, Bénédicte und Burdin, Nikolai. 2011. *WOOD-BASED PANEL MARKETS.* s.l. : CINTRAFOR / University of Washington, 2011.

FAO. 2010. *Global Forest Resources Assessment 2010 (Main report).* s.l. : Food and Agriculture Organization of the United Nations (FAO), 2010.

—. 2004. www.fao.org. [Online] 2004. [Zitat vom: 02. 03 2011.] http://www.fao.org/docrep/007/ad493e/ad493e00.htm.

Finnish Thermowood Association. 2003. *ThermoWood Handbook.* FIN-00171 Helsinki : Wood Focus Oy,, 2003.

FloorDaily. 2011. FloorDaily. [Online] 2011. [Zitat vom: 10. 02 2011.] http://www.floordaily.net/FloorFocus/Wood_Cuts__December_2010.aspx.

FPIInnovations. 2009. solutionsforwood. [Online] 2009. [Zitat vom: 13. 02 2011.] http://www.solutionsforwood.ca/_docs/reports/EWoodFlooringTrendsweb.pdf.

Gabrielli, Chris P. und Kamke, Frederick A. 2009. *Phenol–formaldehyde impregnation of densified wood.* s.l. : Springer Verlag, 2009.

Geimer, Robert L und Kwon, Jin Heon. 1999. *FLAKEBOARD THICKNESS SWELLING. PART II. FUNDAMENTAL.* s.l. : Wood and Fiber Science, 1999.

Gindl, Wolfgang, et al. 2001. *The relationship between near infrared spectra of radial wood surfaces and wood mechanical properties.* s.l. : Near infrared spectrosc., 2001. 9, 255- 261 (2001).

Goetzl, Alberto und Ekström, Håkan C. 2007. *REPORT ON THE REVIEW OF THE US MARKET FOR TROPICAL TIMBER PRODUCTS.* s.l. : The International Tropical Timber Organization, 2007.

Haygreen, JG und Daniels, DH. 1969. The simultaneous drying and densification of sapwood. *Wood fiber science.* 1969, 1: 38-52.

Hill, Callum A.S. 2006. *Wood Modification Chemical: Thermal and Other Processes.* England : John Wiley & Sons, Ltd, 2006.

Howard, James L. und McKeever, David B. 2011. *U.S. Forest Products Annual Market Review and Prospects, 2007–2011.* s.l. : Forest Products Laboratory, Madison, Wisconsin USA, 2011.

Hsu, W.E., et al. 1988. *Chemical and physical changes required for producing dimensionally stable wood-based composites. Part I: Steam pretreatment.* s.l. : Wood Science and Technology, 1988. 22, 281–289.

2009. Hybrid Poplar Wood Properties. *www.owic.oregonstate.edu.* [Online] 2009. [Zitat vom: 02. 03 2011.] http://owic.oregonstate.edu/species/hybridpoplar/euramerica.php.

Ince, Peter J. 2010. *Chapter 5 - Outlook for U.S. paper and paperboard sector and Wood fiber supply in North America.* s.l. : United States Forest Service, U.S. Forest Products Laboratory, Madison, Wisconsin, 2010.

Inoue, M, et al. 1993a. Steam or heat fixation of compressed wood. *Wood and Fiber Science.* 25(3) 1993a, S. 224-235.

Irvine, G.M. 1984. *The glass transition of lignin and hemicellulose and their measurement by differential thermal analysis.* s.l. : Tappi Journal, 1984. 67 (5), 118.

Ito, Youichi, et al. 1998. Compressive- molding of wood by high- pressure steam treatment. *Holzforschung.* 52, 1998.

Jennings, J.D. 2003. *Investigating the Surface Energy and Bond Performance of Compression.* s.l. : Virginia Tech, Blacksburg, Virginia; MasterThesis, 2003.

Jennings, J.D., A. Zink-Sharp, F.A. Kamke, C.E. Frazier. 2005. *Properties of compression densified wood. Part 1: Bond performance.* Journal of Adhesion Science and Technology 19(13-14):1249-1261.

Johanisson, TG. 1994. High pressure forming of body parts using flexible tools. *Sheet Metal Industries.* 1994.

Kamke, F. 2006. Densified radiata pine for structural composites. s.l. : Maderas. Ciencia y tecnología, 2006. 8(2): 83-92, 2006.

Kamke, F.A und Sizemore, H. 2008. Viscoelastic Thermal Compression of Wood. U.S. Patent No. 7,404,422 B2. July 29, 2008.

Kamke, Frederick A und Rathi, Vardan M. 2010a. *Apparatus for viscoelastic thermal compression of wood.* s.l. : Springer Verlag, 2010a.

Kamke, Frederick A. 2007. *Laminated Composites from Densified Wood.* 2007.

Kamke, Frederick A. und Kutnar, Andreja. 2010b. Transverse Compression Behavior of Wood in Saturated Steam at 150-170°C. *Wood and Fiber Science.* 42, 2010b, 3.

—. 2010. Transverse Compression Behavior of Wood in Saturated Steam at 150-170°C. *Wood and Fiber Science.* 42, 2010, 3.

Kamke, Frederick A. und Rautkari, Lauri. 2009. *Modified wood veneer for structural applications.* Espoo, Finland : Fourth International Symposium on Veneer Processing and Products, 2009.

Kamke, F.A. and V.M. Rathi. 2010. *Apparatus for viscoelastic thermal compression of wood.* Eur. J. Wood Prod. Published online June 24, 2010, DOI 10.1007/s00107-010-0461-7.

Kelley, SS, Rials, TG und Glasser, WG. 1987. Relaxation behaviour of the amorphous components of wood . *Wood Material Science & Engineering.* 1987, 22: 617- 624.

Kenney, W.A., Sennerbyforsse, L. und Layton, P. 1990. *A review of biomass quality research relevant to the use of poplar and willow for energy-conversion.* s.l. : Biomass, 1990. 21, 163–188.

Kollman, F.P, Kuenzi, E.W. und Stamm, A.J. 1975. *Principles of wood science and technology. Vol. II Wood based materials.* s.l. : Springer-Verlag New York Heidelberg Berlin, 1975.

Kollmann, FPP und Cote`, WA. 1968. *Principles of wood science and technology I Solid Wood.* Heidelberg, New York : Springer Verlag, 1968.

Kretschmann, David E. 2010. *Wood Handbook- Wood as an engineering material Chapter5,6&7.* s.l. : United States Department of Agriculture, 2010.

Kretschmann, David E., et al. 1999. *Structural Lumber Properties of Hybrid poplar.* s.l. : United States Department ofAgriculture, 1999.

Kunesch, RH. 1961. *The inelastic behavior of wood: A new concept for improved panel forming processes.* s.l. : Forest Product Journal, 1961.

Kunesh, R.H. 1968. *Strength and Elastic Properties of Wood in Transverse Compression.* s.l. : Forest Products Journal, 1968.

Kutnar, Andreja und Šernek, Milan. 2007. *Densification of wood.* Slovenia : Zbornik gozdarstva in lesarstva, 2007. 82 (2007), s. 53–62.

Kutnar, A.; F.A. Kamke; J.A. Nairn and M. Sernek. 2008a. *Mode II fracture behavior of bonded viscoelastic thermal compressed wood.* Wood and Fiber Sci. 40(3):362-373.

Kutnar, Andreja, Kamke, Frederick A und Sernek, M. 2009. *Density profile and morphology of viscoelastic thermal compressed wood.* s.l. : Wood Science Technology, 2009.

—. 2008b. *The mechanical properties of densified VTC wood relevant for structural composites.* s.l. : Holz Roh Werkstoff, 2008.

Kutnar A., F.A. Kamke, M. Petrič , and M. Sernek. 2008c. *The influence of viscoelastic thermal compression on the chemistry and surface energetics of wood*. Colloids Surf. A: Physicochem. Eng. Aspects, 329: 82-86.

Laborie, M.P.G., Salmen, L. und Frazier, C.E. 2004. *Cooperativity analysis of the in situ lignin glass transition.* s.l. : Holzforschung, 2004. 58, 129–133.

Lenth, Christopher A. und Kamke, Frederick A. 2007. Investigations of Flakeboard Mat Consolidation. Part I. Characterizing The Cellular Structure. *Wood Fiber and Science.* 2007.

Lenth, Christopher. 1999. *WOOD MATERIAL BEHAVIOR IN SEVERE ENVIRONMENTS.* s.l. : Dissertation; Virginia Polytechnic Institute and State University , 1999.

Loehnertz, Stephen P. 1987. *A continous press dryer for veneer.* s.l. : FOREST PRODUCTS JOURNAL , 1987. Vol 38, No. 9.

Lohmann, Ulf. 1990. *Holz Handbuch.* Rosenheim : DRW- Verlag, 1990.

Macias, Natalie. 2006. Commercialization Potential of Viscoelastic Thermal Compressed (VTC), Masterthesis. Corvallis Oregon : s.n., 2006.

Marter, Josef Franz. k. Z.a.. *Verzeichnis der österreichischen Bäume, Stauden und Buschgewächse.* Wien : Josef Gerold, k. Z.a.

Michalak, Roman. 2010. *Wood raw materials market developments: From the forest to the mill.* s.l. : UNECE Timber Committee and Society of Wood Science and Techology Market Discussions 2010, 2010.

Militz, Holger. 2002. *Thermal treatment of wood: European processes.* s.l. : International research group on wood preservation, 2002.

Mitchell, P.H. 1988. Irresversible property changes of small loblolly pine specimens heated in air, nitrogen, or oxygen. *Wood and Fiber Science.* 1988, S. 320- 355.

Modzel, Günter Georg Rolf. 2009. *Computed Tomography Analysis of Wood-Adhesive Bonds.* Oregon State University : Doctor of Philosophy in Wood Science, 2009.

Morsing, N. 2000. *Densification of wood- The influence of hygrothermal treatment on compression of beech perpendiculat to the grain.* s.l. : Technical University of Denmark, 2000.

Nairn, J.A. 2006. *Numerical Simulations of Transverse Compression and Densification.* s.l. : Wood and Fiber Science, 2006. 38(4): 576–591.

Navi, P und Giradet, F. 2000. Effects of thermo-hydro-mechanical treatment on the structure and properties of wood. *Holzforschung.* 54, 2000.

Navi, P, Huguenin, PH und Giradet, F. 1997. *Development of Synthetic- Free Plastified Wood by Thermohygromechanical Treatment. Use of Recycled Wood and Paper in Building Applications.* Madison, WI : Forest Product Society, 1997.

Noack, H. 1985. *Cornus mas- Die Kornelkirsche Deutsche Baumschule.* 1985.

Norimoto, M, et al. 1993. Permanent fixation of bending deformation in wood by heat treatment. *Wood Research.* 79, 1993.

Petrič, Marko, et al. Surface free energy of viscoelastic thermal compressed wood and its wettability by the phenol- formaldehyde adhesive

Powell, M.R. 2003. Treatment of wood with Royale stabilising oil. *Proceedings of the First Conference of Wood Modification.* 2003.

REINIATI, ISABELA. 2009. Chemical, physical, and viscoelastic properties of hotpressed hybrid poplar. s.l. Washington State University, 2009.

Reiterer, A und Stanzl- Tschegg, S.E. 2001. *Compressive behaviour of softwood under uniaxial loading at different orientations to the grain.* s.l. : Mechanics of Materials, 2001. 33: 705- 715.

Röchling, Engineering Plastics KG. 2000. *Lignostone.* Haren/ Germany : Röchling Engineering Plastics KG, 2000.

Schrepfer, V und Schweingruber, F.H. 1998. *Anatomical structures in reshaped press dried wood.* s.l. : Holzforschung, 1998. 52(6): 615–622.

Seale, Dan. 2003. *TimTek:Engineering a Wood Product Solution for Your Business.* s.l. : Mississippi State University, 2003.

Seale, R.D, et al. 2004. *Tim Tek- A new engineered product made from smal diameter trees.* s.l. : Forest Product Laboratory, Forest& Wildlife Research Center, Mississipi State University, 2004.

Seborg, R.M, Millett, M.A und Stamm, A.J. 1962a. *Heat-stabilized compressed wood (Staypak).* s.l. : Report No. 1580 (revised), 1962a.

Smith, Ian, Landis, Eric und Gong, Meng. 2003. *Fracture and Fatigue in Wood.* England : John Wiley& Sons Ltd., 2003.

Spelter, H, McKeever, D und Durbak, I. 1997. *Review of Wood-Based Panel Sector in United States and Canada.* s.l. : U.S. Department of Agriculture, Forest Service, Forest Products Laboratory, 1997. Gen. Tech. rep. FPL-GTR-99, Madison.

Stamm, A.J und Seborg, R.M. 1941. *Resin treated, laminated, compressed wood.* s.l. : Trans. Am.Inst. Chem. Eng., 1941.

Stanton, Brian J. 2007. *Hybrid Poplar Feedstock Production: Economic Opportunity for Renewable Energy in North America.* Atlanta, Georgia : TAPPI International Conference on renewable Energy, 2007.

Tabarsa, T. 1999. *Compression perpendicular- to- grain behavior of wood.* Canada : The University of New Brunswick, 1999.

TABARSA, T. und CHUI, Y.H. 2000. *Stress-Strain Response of Wood under Radial Compression. Part I. Test Method and InfluencesofCellularProperties.* s.l. : Wood and Fiber Science, 2000.

Tabarsa, T. und Chui, YH. 1997. Effects of hot- pressing on properties of white spruce. *Forest Product Journal.* 1997, 47: 71- 76.

Thant, Aye Aye, Yee, San San und Htike, Than Than. 2009. *Modeling Drying Time during Veneer Drying and Comparison with Experimental Study.* Hong Kong : Proceedings of the International MultiConference of Engineers and Computer Scientists 2009 Vol II, 2009.

Wingate- Hill, R. 1983. A review of processes which involve compressing wood perpendicular tothe grain. *Australian Forest Research.* 1983, 13:151–164.

Wolcott, M. P.; F. A. Kamke, and D. A. Dillard. 1994. *Fundamental aspects of wood deformation pertaining to manufacture of wood-based composites*, Wood and Fiber Sci. 26(4):496-511.**Wolkinger, F. 1993.** *Bäume und Sträucher Österreichs.* Graz : s.n., 1993.

Zhiyong, Cai und Robert, J. Ross. 2010. *Wood Handbook- Wood as an Engineering Material: Mechanical Properties of Wood-Based Composite Materials.* s.l. : United States Department of Agriculture, 2010.

Zhou, C, Smith, GD und Dai, C. 2009. Characterizing hydrothermal compression behavior of aspen wood strands. *Holzforschung.* 63, 2009.

11 Annex

11.1 Statistics

11.1.1   MOE vs. conditioning time

```
Independent Group Analysis
------------------------------------------------------------------------
Grouping variable is GROUP
Analysis variable is OBS
Group Means and Standard Deviations
----------------------------------
1:  mean = 11349.18            s.d. = 1833.4042         n =  50
2:  mean = 8099.628            s.d. = 2386.9248         n =  50
3:  mean = 11400.77            s.d. = 2838.8057         n =  52
4:  mean = 13801.98            s.d. = 4323.4809         n =  53
5:  mean = 13947.39            s.d. = 3293.488          n =  51


Analysis of Variance Table

Source       S.S.          DF              MS        F         Appx P
------------------------------------------------------------------------
Total        3519295000.       255
  Treatment1150052839.99    4     287513200.   30.46          <.001
  Error      2369243000.      251      9439214.

 Error term used for comparisons = 9,439,214. with 251 d.f.
Critical S  Scheffe Multiple Comp.       Difference      S     (.05)
 ------------------------------------------------------------------------
Mean(5)-Mean(2) =     5847.761           9.564        3.105 *
Mean(5)-Mean(1) =     2598.207           4.249        3.105 *
Mean(5)-Mean(3) =     2546.615          4.206        3.105 *
Mean(5)-Mean(4) =     145.4072           .241        3.105
Mean(4)-Mean(2) =     5702.354          9.414        3.105 *
Mean(4)-Mean(1) =     2452.8           4.049        3.105 *
Mean(4)-Mean(3) =     2401.208          4.004        3.105 *
Mean(3)-Mean(2) =     3301.146          5.425        3.105 *
Mean(3)-Mean(1) =     51.5918           .085        3.105
Mean(1)-Mean(2) =     3249.554          5.288        3.105 *


Homogeneous Populations, groups ranked

            Gp Gp Gp Gp Gp
             2  1  3  4  5
                     ------
              ------
             ---
```

   This is a graphical representation of the Scheffe's multiple compari-
sons
   test. At the 0.05 significance level, the means of any two groups
   underscored by the same line are not significantly different.

```
Simultaneous 95% Confidence Limits
----------------------------------
```
   Significant comparisons based on Conf. Limits indicated by ***.
   CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjust-
ment.

```
Error term used = 9,439,214. with 251 d.f.

     Group                                    Simultaneous 95%
     Comparison   Difference   p-value    Confidence Limits
  ----------------------------------------------------------------------
-----
Mean(5)-Mean(2) = 5847.761 <.001 (4160.9685, 7534.553  ***
Mean(5)-Mean(1) = 2598.207 <.001 (1010.9745, 4185.4396) ***
Mean(5)-Mean(3) = 2546.615 <.001 (1114.393, 3978.8375) ***
Mean(5)-Mean(4) =145.4072 1.000 (-1044.6282, 1335.4427)
Mean(4)-Mean(2) = 5702.354<.001 (4130.0166, 7274.6904) ***
Mean(4)-Mean(1) = 2452.8 <.001  (1020.0373, 3885.5623) ***
Mean(4)-Mean(3) = 2401.208 <.001 (1217.0183, 3585.3977) ***
Mean(3)-Mean(2) = 3301.146 <.001 (1861.7109, 4740.5801) ***
Mean(3)-Mean(1) = 51.5918 1.000 (-1150.0628, 1253.2464)
Mean(1)-Mean(2) = 3249.554 <.001 (2036.1754, 4462.932) ***
```

11.1.2   MOE vs. compression rate

```
Independent Group Analysis
-----------------------------------------------------------------------
Grouping variable is GROUP
Analysis variable is OBS

Group Means and Standard Deviations
----------------------------------
1:  mean = 10290.7            s.d. = 2106.9362        n =  60
2:  mean = 12348.81           s.d. = 3235.0038        n =  47
3:  mean = 13801.98           s.d. = 4323.4809        n =  53
4:  mean = 9131.706           s.d. = 1481.4074        n =  48

Analysis of Variance Table

Source      S.S.        DF            MS        F        Appx P
  --------------------------------------------------------------------
Total      2487303000.     207
  Treatment 668835463.39    3    222945200.  25.01          <.001
  Error    1818467000.     204      8914055.

Error term used for comparisons = 8,914,055. with 204 d.f.
Critical S Scheffe Multiple Comp.  Difference    S         (.05)
  --------------------------------------------------------------------
Mean(3)-Mean(4) =       4670.275       7.851       2.82 *
Mean(3)-Mean(1) =       3511.278       6.239       2.82 *
Mean(3)-Mean(2) =       1453.169       2.429       2.82
Mean(2)-Mean(4) =       3217.106       5.251       2.82 *
Mean(2)-Mean(1) =       2058.109       3.539       2.82 *
Mean(1)-Mean(4) =       1158.997       2.005       2.82

Homogeneous Populations, groups ranked

               Gp Gp Gp Gp
                4  1  2  3
                   ------
                ------
```

This is a graphical representation of the Scheffe's multiple comparisons
test. At the 0.05 significance level, the means of any two groups
underscored by the same line are not significantly different.

```
Simultaneous 95% Confidence Limits
```

```
---------------------------------
Significant comparisons based on Conf. Limits indicated by ***.
CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjust-
ment.
Error term used = 8,914,055. with 204 d.f.

      Group                       Simultaneous 95%
   Comparison   Difference   p-value      Confidence Limits
   ------------------------------------------------------------------

Mean(3)-Mean(4) =  4670.275 <.001 (3123.9744, 6216.5764) ***
Mean(3)-Mean(1) =  3511.278 <.001 (2178.4206, 4844.1361) ***
Mean(3)-Mean(2) =  1453.169 0.096 (270.7737, 2635.5642) ***
Mean(2)-Mean(4) = 3217.106 <.001 (1766.1633, 4668.0495) ***
Mean(2)-Mean(1) = 2058.109 0.003 (908.5885, 3207.6303) ***
Mean(1)-Mean(4) =  1158.997  0.278 (16.2104, 2301.7837) ***
```

11.1.3   MOE vs. compression time

```
Independent Group Analysis
-----------------------------------------------------------------------

Grouping variable is GROUP
Analysis variable is OBS

Group Means and Standard Deviations
---------------------------------
1:  mean = 11075.23          s.d. = 3564.4325        n =  69
2:  mean = 11531.21          s.d. = 2781.8206        n =  52
3:  mean = 13801.98          s.d. = 4323.4809        n =  53
4:  mean = 12029.03          s.d. = 2601.8457        n =  59
5:  mean = 13262.76          s.d. = 2667.3601        n =  42


Analysis of Variance Table

Source      S.S.          DF      MS        F         Appx P
-----------------------------------------------------------------------
Total        3210518000.       274
  Treatment 295547285.14     4      73886820.    6.84           <.001
  Error     2914970000.      270     10796190.

Error term used for comparisons = 10,796,190. with 270 d.f.

 Critical S  Scheffe Multiple Comp.      Difference  S        (.05)
-----------------------------------------------------------------------
Mean(3)-Mean(1) =         2726.747         4.544        3.105 *
Mean(3)-Mean(2) =         2270.773         3.541        3.105 *
Mean(3)-Mean(4) =         1772.954         2.851        3.105
Mean(3)-Mean(5) =          539.2168  (Do not test)
Mean(5)-Mean(1) =         2187.53          3.402        3.105 *
Mean(5)-Mean(2) =         1731.557          2.54        3.105
Mean(5)-Mean(4) =         1233.737  (Do not test)
Mean(4)-Mean(1) =         953.793          1.637        3.105
Mean(4)-Mean(2) =                 497.8193  (Do not test)
Mean(2)-Mean(1) =                 455.9736  (Do not test)

Homogeneous Populations, groups ranked

               Gp Gp Gp Gp Gp
                1  2  4  5  3
```

```
                        ---------
                      ---------
                    ---------
```

   This is a graphical representation of the Scheffe's multiple compari-
sons
   test. At the 0.05 significance level, the means of any two groups
   underscored by the same line are not significantly different.

   Simultaneous 95% Confidence Limits

   Significant comparisons based on Conf. Limits indicated by ***.
   CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjust-
ment.
   Error term used = 10,796,190. with 270 d.f.

```
        Group                          Simultaneous 95%
Comparison      Difference   p-value      Confidence Limits
-----------------------------------------------------------------------
Mean(3)-Mean(1) = 2726.747  <.001    (1072.1035, 4381.3906) ***
Mean(3)-Mean(2) = 2270.773  0.005    (606.8214, 3934.7255) ***
Mean(3)-Mean(4) = 1772.954  0.047    (302.7261, 3243.1821) ***
Mean(3)-Mean(5) = 539.2168  1.000    (-800.662, 1879.0956)
Mean(5)-Mean(1) = 2187.53   0.008    (519.1383, 3855.9222) ***
Mean(5)-Mean(2) = 1731.557  0.116    (119.8825, 3343.2307) ***
Mean(5)-Mean(4) = 1233.737  0.640    (-75.6748, 2543.1494)
Mean(4)-Mean(1) = 953.793   1.000    (-423.7148, 2331.3007)
Mean(4)-Mean(2) = 497.8193  1.000    (-735.8538, 1731.4925)
Mean(2)-Mean(1) = 455.9736  1.000    (-735.0839, 1647.0312)
```

11.1.4   MOR vs. conditioning time (5- layer laminates)

```
   Independent Group Analysis
   -----------------------------------------------------------------------
   Grouping variable is GROUP
   Analysis variable is OBS

   Group Means and Standard Deviations
   -----------------------------------
   1:  mean = 105.2              s.d. = 18.7782         n =  10
   2:  mean = 111.3              s.d. = 25.6214         n =  10
   3:  mean = 114.4              s.d. = 11.5682         n =  10
   4:  mean = 104.9              s.d. = 26.3416         n =  10
   5:  mean = 116.0              s.d. = 18.7853         n =  10

   Analysis of Variance Table

   Source       S.S.      DF      MS       F         Appx P
   -----------------------------------------------------------------------
   Total         20761.52       49
     Treatment     1054.52        4            263.63         .6
0.6632
     Error         19707.       45          437.93

   Error term used for comparisons = 437.93 with 45 d.f.

   Critical S Scheffe Multiple Comp.    Difference    S         (.05)
   -----------------------------------------------------------------------
   Mean(5)-Mean(4) =            11.1      1.186       3.212
   Mean(5)-Mean(1) =                      10.8  (Do not test)
   Mean(5)-Mean(2) =                       4.7  (Do not test)
```

```
Mean(5)-Mean(3) =                        1.6  (Do not test)
Mean(3)-Mean(4) =                        9.5  (Do not test)
Mean(3)-Mean(1) =                        9.2  (Do not test)
Mean(3)-Mean(2) =                        3.1  (Do not test)
Mean(2)-Mean(4) =                        6.4  (Do not test)
Mean(2)-Mean(1) =                        6.1  (Do not test)
Mean(1)-Mean(4) =                        0.3  (Do not test)


   Homogeneous Populations, groups ranked

                  Gp Gp Gp Gp Gp
                   4  1  2  3  5
                  --------------


   This is a graphical representation of the Scheffe's multiple compari-
sons
   test. At the 0.05 significance level, the means of any two groups
   underscored by the same line are not significantly different.

   Simultaneous 95% Confidence Limits
   ----------------------------------
   Significant comparisons based on Conf. Limits indicated by ***.
   CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjust-
ment.
   Error term used = 437.93 with 45 d.f.

      Group                                         Simultaneous
95%
      Comparison           Difference    p-value       Confidence Lim-
its
   -----------------------------------------------------------------------
   Mean(5)-Mean(4) =          11.1        1.000      (-15.5065, 37.7065)
   Mean(5)-Mean(1) =          10.8        1.000      (-14.1786, 35.7786)
   Mean(5)-Mean(2) =           4.7        1.000      (-17.992, 27.392)
   Mean(5)-Mean(3) =           1.6        1.000      (-17.2565, 20.4565)
   Mean(3)-Mean(4) =           9.5        1.000      (-15.4786, 34.4786)
   Mean(3)-Mean(1) =           9.2        1.000      (-13.492, 31.892)
   Mean(3)-Mean(2) =           3.1        1.000      (-15.7565, 21.9565)
   Mean(2)-Mean(4) =           6.4        1.000      (-16.292, 29.092)
   Mean(2)-Mean(1) =           6.1        1.000      (-12.7565, 24.9565)
   Mean(1)-Mean(4) =           0.3        1.000      (-18.5565, 19.1565)
```

11.1.5   MOE vs. conditioning time (5- layer laminates)

```
   Independent Group Analysis
   -----------------------------------------------------------------------

   Grouping variable is GROUP
   Analysis variable is OBS

   Group Means and Standard Deviations
   -----------------------------------
   1:  mean = 9799.916          s.d. = 1436.7365        n =  10
   2:  mean = 11977.95          s.d. = 2583.0317        n =  10
   3:  mean = 11948.44          s.d. = 1426.2872        n =  10
   4:  mean = 11425.77          s.d. = 4222.2407        n =  10
   5:  mean = 12582.83          s.d. = 2526.2021        n =  10

   Analysis of Variance Table

   Source     S.S.    DF      MS       F        Appx P
```

```
    ---------------------------------------------------------------------
    Total          359684300.         49
      Treatment    44868136.63              4         11217030.        1.6
  0.1899
      Error        314816200.        45         6995915.
```

Error term used for comparisons = 6,995,915. with 45 d.f.

```
  Critical S  Scheffe Multiple Comp.    Difference       S        (.05)
  ---------------------------------------------------------------------
  Mean(5)-Mean(1) =        2782.913        2.353        3.212
  Mean(5)-Mean(4) =                        1157.056  (Do not test)
  Mean(5)-Mean(3) =                        634.3877  (Do not test)
  Mean(5)-Mean(2) =                        604.8779  (Do not test)
  Mean(2)-Mean(1) =                        2178.035  (Do not test)
  Mean(2)-Mean(4) =                        552.1777  (Do not test)
  Mean(2)-Mean(3) =                         29.5098  (Do not test)
  Mean(3)-Mean(1) =                        2148.525  (Do not test)
  Mean(3)-Mean(4) =                         522.668  (Do not test)
  Mean(4)-Mean(1) =                        1625.857  (Do not test)
```

Homogeneous Populations, groups ranked

```
              Gp Gp Gp Gp Gp
               1  4  3  2  5
              ---------------
```

This is a graphical representation of the Scheffe's multiple comparisons
   test. At the 0.05 significance level, the means of any two groups
   underscored by the same line are not significantly different.

   Simultaneous 95% Confidence Limits

   Significant comparisons based on Conf. Limits indicated by ***.
   CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjustment.
   Error term used = 6,995,915. with 45 d.f.

```
  Group                         Simultaneous 95%
Comparison      Difference   p-value      Confidence Limits
  ---------------------------------------------------------------------
Mean(5)-Mean(1) =  2782.913    0.231    (-579.9304, 6145.7566)
Mean(5)-Mean(4) =  1157.056    1.000    (-2000.0297, 4314.141)
Mean(5)-Mean(3) =  634.3877    1.000    (-2233.6853, 3502.4607)
Mean(5)-Mean(2) =  604.8779    1.000    (-1778.4297, 2988.1856)
Mean(2)-Mean(1) =  2178.035    0.722    (-979.0502, 5335.1205)
Mean(2)-Mean(4) =  552.1777    1.000    (-2315.8952, 3420.2507)
Mean(2)-Mean(3) =  29.5098     1.000    (-2353.7979, 2412.8174)
Mean(3)-Mean(1) =  2148.525    0.760    (-719.5476, 5016.5984)
Mean(3)-Mean(4) =  522.668     1.000    (-1860.6397, 2905.9756)
Mean(4)-Mean(1) =  1625.857    1.000    (-757.4502, 4009.165)
```

11.1.6   MOR vs. compression rate (5- layer laminates)

Independent Group Analysis

   Grouping variable is GROUP
   Analysis variable is OBS

```
Group Means and Standard Deviations
-----------------------------------
1:  mean = 110.3              s.d. = 16.0558          n =  10
2:  mean = 118.1111           s.d. = 15.4065          n =  9
3:  mean = 104.9              s.d. = 26.3416          n =  10
4:  mean = 86.3               s.d. = 12.3922          n =  10


Analysis of Variance Table

Source        S.S.          DF              MS        F         Appx P
-----------------------------------------------------------------------
Total         17163.59        38
Treatment     5317.6          3           1772.53    5.24         0.0043
  Error       11845.99        35           338.46
Error term used for comparisons = 338.46 with 35 d.f.
                                                            Critical S
 Scheffe Multiple Comp.           Difference        S          (.05)
-----------------------------------------------------------------------
Mean(2)-Mean(4) =            31.8111        3.763       2.939 *
Mean(2)-Mean(3) =             13.2111         1.563      2.939
Mean(2)-Mean(1) =               7.8111  (Do not test)
Mean(1)-Mean(4) =            24.0          2.917      2.939
Mean(1)-Mean(3) =               5.4  (Do not test)
Mean(3)-Mean(4) =              18.6  (Do not test)


Homogeneous Populations, groups ranked

              Gp Gp Gp Gp
               4  3  1  2
                 ---------
              ---------
```

   This is a graphical representation of the Scheffe's multiple compari-
sons
   test. At the 0.05 significance level, the means of any two groups
   underscored by the same line are not significantly different.

```
   Simultaneous 95% Confidence Limits
   -----------------------------------
   Significant comparisons based on Conf. Limits indicated by ***.
   CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjust-
ment.
   Error term used = 338.46 with 35 d.f.
 Group                                     Simultaneous 95%
Comparison          Difference   p-value       Confidence Limits
   -----------------------------------------------------------------------
   Mean(2)-Mean(4) =  31.8111   0.004    (8.9902, 54.632)    ***
   Mean(2)-Mean(3) =  13.2111    0.762    (-7.4954, 33.9176)
   Mean(2)-Mean(1) =  7.8111    1.000    (-9.3626, 24.9848)
   Mean(1)-Mean(4) =   24.0     0.037    (3.8457, 44.1543)    ***
   Mean(1)-Mean(3) =    5.4      1.000    (-11.3157, 22.1157)
   Mean(3)-Mean(4) = 18.6   0.181    (1.8843, 35.3157)    ***
```

11.1.7   MOE vs. compression rate (5- layer laminates)

```
Independent Group Analysis
-----------------------------------------------------------------------
Grouping variable is GROUP
Analysis variable is OBS
```

```
   Group Means and Standard Deviations
   -----------------------------------
   1:  mean = 12145.84          s.d. = 1614.2599       n =  10
   2:  mean = 13255.03          s.d. = 1591.9333       n =   9
   3:  mean = 11425.77          s.d. = 4222.2407       n =  10
   4:  mean = 8305.469          s.d. = 919.1184        n =  10


   Analysis of Variance Table

   Source      S.S.     DF        MS        F        Appx P
   ------------------------------------------------------------------
   Total        342981700.       38
     Treatment 131206339.89      3       43735450.    7.23          <.001
     Error      211775400.       35       6050725.

   Error term used for comparisons = 6,050,725. with 35 d.f.

Critical S  Scheffe Multiple Comp.  Difference       S        (.05)
   ------------------------------------------------------------------
   Mean(2)-Mean(4) =     4949.563          4.379      2.939 *
   Mean(2)-Mean(3) =     1829.258          1.619      2.939
   Mean(2)-Mean(1) =     1109.187  (Do not test)
   Mean(1)-Mean(4) =     3840.376          3.491      2.939 *
   Mean(1)-Mean(3) =     720.0713  (Do not test)
   Mean(3)-Mean(4) =     3120.305          2.836      2.939


   Homogeneous Populations, groups ranked

             Gp Gp Gp Gp
              4  3  1  2
                ---------
                ------
   This is a graphical representation of the Scheffe's multiple compari-
sons
   test. At the 0.05 significance level, the means of any two groups
   underscored by the same line are not significantly different.

   Simultaneous 95% Confidence Limits
   -----------------------------------
   Significant comparisons based on Conf. Limits indicated by ***.
   CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjust-
ment.
   Error term used = 6,050,725. with 35 d.f.

Group                         Simultaneous 95%
Comparison     Difference   p-value      Confidence Limits
   ------------------------------------------------------------------
Mean(2)-Mean(4) =   4949.563    <.001    (1898.2599, 8000.8651) ***
Mean(2)-Mean(3) =   1829.258    0.687    (-939.3376, 4597.8532)
Mean(2)-Mean(1) =   1109.187    1.000    (-1187.0518, 3405.4249)
Mean(1)-Mean(4) =   3840.376    0.008    (1145.6232, 6535.1287) ***
Mean(1)-Mean(3) =   720.0713    1.000    (-1514.9229, 2955.0655)
Mean(3)-Mean(4) =   3120.305    0.045    (885.3105, 5355.2989) ***
```

11.1.8   MOE vs. compression time (5- layer laminates)

```
   Independent Group Analysis
   ------------------------------------------------------------------
```

```
   Grouping variable is GROUP
   Analysis variable is OBS

   Group Means and Standard Deviations
   -----------------------------------
   1:  mean = 8152.882          s.d. = 2991.7865       n =  11
   2:  mean = 9841.089          s.d. = 2523.7038       n =  10
   3:  mean = 11425.77          s.d. = 4222.2407       n =  10
   4:  mean = 11220.43          s.d. = 1018.3333       n =  10
   5:  mean = 13948.75          s.d. = 3437.7309       n =  8


   Analysis of Variance Table

   Source      S.S.        DF            MS        F        Appx P
   ------------------------------------------------------------------
   Total        570419100.      48
     Treatment 171084646.54  4     42771160.   4.71       0.003
     Error      399334400.       44      9075782.

   Error term used for comparisons = 9,075,782. with 44 d.f.
    Critical S  Scheffe Multiple Comp.  Difference    S         (.05)
   ------------------------------------------------------------------
   Mean(5)-Mean(1) =     5795.867            4.14      3.219 *
   Mean(5)-Mean(2) =     4107.66            2.874      3.219
   Mean(5)-Mean(4) =     2728.321  (Do not test)
   Mean(5)-Mean(3) =     2522.976  (Do not test)
   Mean(3)-Mean(1) =     3272.892            2.486      3.219
   Mean(3)-Mean(2) =     1584.685  (Do not test)
   Mean(3)-Mean(4)       3067.546  (Do not test)
   Mean(4)-Mean(2) =     1379.339  (Do not test)
   Mean(2)-Mean(1) =     1688.207  (Do not test)

   Homogeneous Populations, groups ranked

             Gp Gp Gp Gp Gp
              1  2  4  3  5
                 ------------
              ------------
   This is a graphical representation of the Scheffe's multiple compari-
sons
   test. At the 0.05 significance level, the means of any two groups
   underscored by the same line are not significantly different.

   Simultaneous 95% Confidence Limits
   -----------------------------------
   Significant comparisons based on Conf. Limits indicated by ***.
   CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjust-
ment.
   Error term used = 9,075,782. with 44 d.f.

  Group                              Simultaneous 95%
Comparison  Difference   p-value      Confidence Limits
   ------------------------------------------------------------------
Mean(5)-Mean(1) =   5795.867   0.002   (1812.5191, 9779.2153) ***
Mean(5)-Mean(2) = 4107.66      0.062   (290.3823, 7924.938) ***
Mean(5)-Mean(4) =   2728.321    0.628    (-739.1866, 6195.8291)
Mean(5)-Mean(3) =  2522.976    0.844    (-358.0393, 5403.9904)
Mean(3)-Mean(1) =  3272.892    0.168    (-243.3265, 6789.1097)
Mean(3)-Mean(2) =  1584.685    1.000    (-1684.5129, 4853.882)
Mean(3)-Mean(4) =  205.3457    1.000    (-2510.9009, 2921.5923)
Mean(4)-Mean(1) =3067.546    0.244     (-126.4878, 6261.5796)
Mean(4)-Mean(2) =  1379.339    1.000    (-1336.9078, 4095.5855)
```

```
Mean(2)-Mean(1) =  1688.207   1.000   (-965.589, 4342.003)
```

11.1.9   MOR vs. compression time (5- layer laminates)

Independent Group Analysis

Grouping variable is GROUP

Analysis variable is OBS

Group Means and Standard Deviations

1: mean = 78.8182   s.d. = 21.5166   n = 11
2: mean = 92.3   s.d. = 21.6541   n = 10
3: mean = 104.9   s.d. = 26.3416   n = 10
4: mean = 106.4   s.d. = 11.6638   n = 10
5: mean = 116.0   s.d. = 32.1781   n = 8

Analysis of Variance Table

Source	S.S.	DF	MS	F	Appx P
Total	31695.84	48			
Treatment	8128.8	4	2032.2	3.79	0.0098
Error	23567.04	44	535.61		

Error term used for comparisons = 535.61 with 44 d.f.

Critical S	Scheffe Multiple Comp.	Difference	S	(.05)
Mean(5)-Mean(1) =		37.1818	3.458	3.219 *
Mean(5)-Mean(2) =		23.7	2.159	3.219
Mean(5)-Mean(3) =		11.1 (Do not test)		
Mean(5)-Mean(4) =		9.6 (Do not test)		
Mean(4)-Mean(1) =		27.5818	2.728	3.219
Mean(4)-Mean(2) =		14.1 (Do not test)		
Mean(4)-Mean(3) =		1.5 (Do not test)		
Mean(3)-Mean(1) =		26.0818 (Do not test)		
Mean(3)-Mean(2) =		12.6 (Do not test)		
Mean(2)-Mean(1) =		13.4818 (Do not test)		

Homogeneous Populations, groups ranked

Gp Gp Gp Gp Gp

```
1 2 3 4 5
   ------------
   ------------
```

This is a graphical representation of the Scheffe's multiple comparisons test. At the 0.05 significance level, the means of any two groups underscored by the same line are not significantly different.

Simultaneous 95% Confidence Limits

Significant comparisons based on Conf. Limits indicated by ***.
CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjustment.
Error term used = 535.61 with 44 d.f.

Group Comparison	Difference	p-value	Simultaneous 95% Confidence Limits	
Mean(5)-Mean(1) =	37.1818	0.012	(6.581, 67.7826)	***
Mean(5)-Mean(2) =	23.7	0.364	(-5.625, 53.025)	
Mean(5)-Mean(3) =	11.1	1.000	(-15.538, 37.738)	
Mean(5)-Mean(4) =	9.6	1.000	(-12.5325, 31.7325)	
Mean(4)-Mean(1) =	27.5818	0.091	(0.5696, 54.594)	***
Mean(4)-Mean(2) =	14.1	1.000	(-11.0145, 39.2145)	
Mean(4)-Mean(3) =	1.5	1.000	(-19.3667, 22.3667)	
Mean(3)-Mean(1) =	26.0818	0.133	(1.5447, 50.6189)	***
Mean(3)-Mean(2) =	12.6	1.000	(-8.2667, 33.4667)	
Mean(2)-Mean(1) =	13.4818	1.000	(-6.9051, 33.8687)	

11.1.10 Shear failure stress vs. resin loading rate

Independent Group Analysis

Grouping variable is GROUP
Analysis variable is OBS

Group Means and Standard Deviations

1: mean = 46.5468   s.d. = 16.1033   n = 22
2: mean = 62.136   s.d. = 16.629   n = 25
3: mean = 57.0432   s.d. = 15.096   n = 22

Analysis of Variance Table

Source	S.S.	DF	MS	F	Appx P
Total	19784.56	68			
Treatment	2916.69	2	1458.35	5.71	0.0052
Error	16867.87	66	255.57		

Error term used for comparisons = 255.57 with 66 d.f.

Critical S	Scheffe Multiple Comp.	Difference	S	(.05)
Mean(2)-Mean(1) =		15.5892	3.336	2.506 *
Mean(2)-Mean(3) =		5.0928	1.09	2.506
Mean(3)-Mean(1) =		10.4964	2.178	2.506

Homogeneous Populations, groups ranked

Gp Gp Gp

1 3 2

This is a graphical representation of the Scheffe's multiple comparisons test. At the 0.05 significance level, the means of any two groups underscored by the same line are not significantly different.

Simultaneous 95% Confidence Limits

Significant comparisons based on Conf. Limits indicated by ***.

CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjustment.

Error term used = 255.57 with 66 d.f.

Group Comparison	Difference	p-value	Simultaneous 95% Confidence Limits	
Mean(2)-Mean(1) =	15.5892	0.004	(4.3722, 26.8062)	***
Mean(2)-Mean(3) =	5.0928	0.839	(-4.2459, 14.4316)	
Mean(3)-Mean(1) =	10.4964	0.099	(0.8642, 20.1286)	***

Note: Because different multiple comparisons procedures are based on different methods, they may not completely agree for marginally significant comparisons.

11.1.11   Shear failure stress vs. conditioning time

Independent Group Analysis

Grouping variable is GROUP

Analysis variable is OBS

Group Means and Standard Deviations

1: mean = 60.2148   s.d. = 10.6598   n = 54

2: mean = 60.6214   s.d. = 12.8975   n = 14

3: mean = 64.3533   s.d. = 9.0645   n = 30

4: mean = 65.4   s.d. = 11.0007   n = 27
5: mean = 55.216   s.d. = 12.0427   n = 25

Analysis of Variance Table

Source	S.S.	DF	MS	F	Appx P
Total	18921.59	149			
Treatment	1726.83	4	431.71	3.64	0.0074
Error	17194.76	145	118.58		

Error term used for comparisons = 118.58 with 145 d.f.

Critical S	Scheffe Multiple Comp.	Difference	S	(.05)
Mean(4)-Mean(5) =		10.184	3.369	3.124 *
Mean(4)-Mean(1) =		5.1852	2.02	3.124
Mean(4)-Mean(2) =		4.7786 (Do not test)		
Mean(4)-Mean(3) =		1.0467 (Do not test)		
Mean(3)-Mean(5) =		9.1373	3.099	3.124
Mean(3)-Mean(1) =		4.1385 (Do not test)		
Mean(3)-Mean(2) =		3.7319 (Do not test)		
Mean(2)-Mean(5) =		5.4054 (Do not test)		
Mean(2)-Mean(1) =		0.4066 (Do not test)		
Mean(1)-Mean(5) =		4.9988 (Do not test)		

Homogeneous Populations, groups ranked
   Gp Gp Gp Gp Gp
    5 1 2 3 4

This is a graphical representation of the Scheffe's multiple comparisons
test. At the 0.05 significance level, the means of any two groups
underscored by the same line are not significantly different.

Simultaneous 95% Confidence Limits

Significant comparisons based on Conf. Limits indicated by ***.
CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjustment.
Error term used = 118.58 with 145 d.f.

Group Simultaneous 95% Comparison	Difference	p-value	Confidence Limits	
Mean(4)-Mean(5) =	10.184	0.010	(1.819, 18.549)	***
Mean(4)-Mean(1) =	5.1852	0.452	(-1.4975, 11.8678)	
Mean(4)-Mean(2) =	4.7786	1.000	(-3.7273, 13.2845)	
Mean(4)-Mean(3) =	1.0467	1.000	(-4.67, 6.7633)	
Mean(3)-Mean(5) =	9.1373	0.023	(1.4595, 16.8151)	***
Mean(3)-Mean(1) =	4.1385	0.973	(-1.7425, 10.0196)	
Mean(3)-Mean(2) =	3.7319	1.000	(-3.2431, 10.7069)	

Mean(2)-Mean(5) =   5.4054   1.000   (-3.2159, 14.0267)
Mean(2)-Mean(1) =   0.4066   1.000   (-6.0564, 6.8697)
Mean(1)-Mean(5) =   4.9988   0.597   (-0.2142, 10.2119)

Note: Because different multiple comparisons procedures are based on different methods, they may not completely agree for marginally significant comparisons.

11.1.12   Shear failure stress vs. rate of compression

Independent Group Analysis

Grouping variable is GROUP
Analysis variable is OBS

Group Means and Standard Deviations

1: mean = 60.6857   s.d. = 12.9078   n = 28
2: mean = 57.9909   s.d. = 9.5606   n = 11
3: mean = 60.3   s.d. = 7.4993   n = 5
4: mean = 57.0782   s.d. = 16.6846   n = 17

Analysis of Variance Table

Source	S.S.	DF	MS	F	Appx P
Total	10251.93	60			
Treatment	160.38	3	53.46	.3	0.8238
Error	10091.55	57	177.04		

Error term used for comparisons = 177.04 with 57 d.f.

Critical S Scheffe Multiple Comp.	Difference	S	(.05)
Mean(1)-Mean(4) =	3.6075	.882	2.883
Mean(1)-Mean(2) =	2.6948 (Do not test)		
Mean(1)-Mean(3) =	0.3857 (Do not test)		
Mean(3)-Mean(4) =	3.2218 (Do not test)		
Mean(3)-Mean(2) =	2.3091 (Do not test)		
Mean(2)-Mean(4) =	0.9127 (Do not test)		

Homogeneous Populations, groups ranked

        Gp Gp Gp Gp
         4 2 3 1

This is a graphical representation of the Scheffe's multiple comparisons test. At the 0.05 significance level, the means of any two groups

underscored by the same line are not significantly different.

Simultaneous 95% Confidence Limits

Significant comparisons based on Conf. Limits indicated by ***.
CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjustment.
Error term used = 177.04 with 57 d.f.

Group Simultaneous 95% Comparison   Difference   p-value   Confidence Limits

  ----

Comparison	Difference	p-value	Confidence Limits
Mean(1)-Mean(4) =	3.6075	1.000	(-7.2219, 14.4369)
Mean(1)-Mean(2) =	2.6948	1.000	(-8.7012, 14.0908)
Mean(1)-Mean(3) =	0.3857	1.000	(-12.5526, 13.3241)
Mean(3)-Mean(4) =	3.2218	1.000	(-13.0711, 19.5147)
Mean(3)-Mean(2) =	2.3091	1.000	(-12.0645, 16.6826)
Mean(2)-Mean(4) =	0.9127	1.000	(-9.3993, 11.2247)

Note: Because different multiple comparisons procedures are based on different methods, they may not completely agree for marginally significant comparisons.

11.1.13   Shear failure stress vs. compression time

Independent Group Analysis

Grouping variable is GROUP
Analysis variable is OBS
Group Means and Standard Deviations

1: mean = 60.3   s.d. = 7.4993   n = 5
2: mean = 55.14   s.d. = 14.1263   n = 25
3: mean = 65.4   s.d. = 11.0007   n = 27
4: mean = 53.9407   s.d. = 9.5539   n = 27
5: mean = 55.7588   s.d. = 13.3592   n = 17
Analysis of Variance Table

Source	S.S.	DF	MS	F	Appx P
Total	15628.43	100			
Treatment	2239.13	4	559.78	4.01	0.0047
Error	13389.31	96	139.47		

Error term used for comparisons = 139.47 with 96 d.f.

Critical S Scheffe Multiple Comp.	Difference	S	(.05)

Mean(3)-Mean(4) =	11.4593	3.565	3.143 *
Mean(3)-Mean(2) =	10.26	3.13	3.143
Mean(3)-Mean(5) =	9.6412 (Do not test)		

Mean(3)-Mean(1) =   5.1 (Do not test)
Mean(1)-Mean(4) =   6.3593   1.106   3.143
Mean(1)-Mean(2) =   5.16 (Do not test)
Mean(1)-Mean(5) =   4.5412 (Do not test)
Mean(5)-Mean(4) =   1.8181 (Do not test)
Mean(5)-Mean(2) =   0.6188 (Do not test)
Mean(2)-Mean(4) =   1.1993 (Do not test)

Homogeneous Populations, groups ranked

        Gp Gp Gp Gp Gp
         4 2 5 1 3

This is a graphical representation of the Scheffe's multiple comparisons
test. At the 0.05 significance level, the means of any two groups
underscored by the same line are not significantly different.

Simultaneous 95% Confidence Limits

Significant comparisons based on Conf. Limits indicated by ***.
CI uses Tukey-Kramer procedure. P-values reflect a Bonferroni adjustment.
Error term used = 139.47 with 96 d.f.

Group Comparison	Difference	p-value	Simultaneous 95% Confidence Limits	
Mean(3)-Mean(4) =	11.4593	0.006	(2.5019, 20.4167)	***
Mean(3)-Mean(2) =	10.26	0.023	(1.6711, 18.8489)	***
Mean(3)-Mean(5) =	9.6412	0.098	(0.9198, 18.3626)	***
Mean(3)-Mean(1) =	5.1	1.000	(-6.3311, 16.5312)	
Mean(1)-Mean(4) =	6.3593	1.000	(-8.7066, 21.4251)	
Mean(1)-Mean(2) =	5.16	1.000	(-8.6398, 18.9598)	
Mean(1)-Mean(5) =	4.5412	1.000	(-7.4038, 16.4861)	
Mean(5)-Mean(4) =	1.8181	1.000	(-6.9033, 10.5395)	
Mean(5)-Mean(2) =	0.6188	1.000	(-6.7621, 7.9998)	
Mean(2)-Mean(4) =	1.1993	1.000	(-5.3175, 7.716)	

Note: Because different multiple comparisons procedures are based on different
methods, they may not completely agree for marginally significant comparisons.

11.2 Rawdata

Nr.	MOE versus conditioning time										
	E-3-C		A-3-C		B-3-C		C-3-C		D-3-C	
	Density (g/m³)	Modulus of elasitci-ty (MPa)	Density (g/m³)	Modulus of elasit-city (MPa)	Density (g/m³)	Modulus of elasit-city (MPa)	Density (g/m³)	Modulus of elasit-city (MPa)	Density (g/m³)	Modulus of elasit-city (MPa)
1	0.60	10173.00	0.69	7396.20	0.67	9224.20	0.84	13449.70	0.71	9839.20
2	0.59	14017.10	0.78	8494.20	0.74	12143.70	0.70	8873.80	0.82	16106.10
3	0.64	11324.30	0.74	6177.50	0.73	14281.50	0.68	10531.50	0.83	16912.00
4	0.68	8181.40	0.68	5493.20	0.65	12067.10	0.71	13427.70	0.74	14381.20
5	0.64	12866.70	0.69	5039.20	0.62	12365.10	0.74	15359.70	0.79	19244.90
6	0.64	10748.40	0.86	11540.80	0.56	9073.60	0.68	9168.30	0.74	12731.60
7	0.58	10672.30	0.90	12438.30	0.70	10406.00	0.75	7707.80	0.95	18632.70
8	0.52	10376.00	0.86	12780.50	0.69	9524.50	0.90	18800.40	0.84	14213.40
9	0.69	13077.60	0.73	5951.90	0.78	13132.00	0.89	17909.80	0.89	18100.90
10	0.66	12303.50	0.88	12424.20	0.70	12113.90	0.70	7269.80	0.93	21388.80
11	0.59	11419.20	0.76	6548.40	0.73	14286.90	0.89	18699.90	0.87	20109.50
12	0.61	12043.10	0.75	8152.20	0.77	17698.30	0.81	18373.50	0.69	12351.60
13	0.67	13510.70	0.76	11093.30	0.72	13377.90	0.81	24236.70	0.65	8414.00
14	0.66	11687.20	0.72	8176.20	0.84	18755.10	0.72	11866.00	0.68	7955.60
15	0.58	11781.60	0.83	9644.30	0.77	13040.30	0.75	14474.50	0.61	12143.20
16	0.58	9949.40	0.83	8663.90	0.66	9385.60	0.70	14357.00	0.62	11009.80
17	0.72	13012.90	0.82	7633.40	0.61	11059.40	0.88	18564.10	0.60	10012.50
18	0.70	13321.50	0.89	10862.50	0.71	10312.50	0.91	18457.10	0.82	12964.60
19	0.59	9046.60	0.88	10189.40	0.68	9662.60	0.89	20342.10	0.70	11261.40
20	0.59	11564.10	0.91	9753.60	0.54	8034.50	0.89	22909.40	0.76	16377.10
21	0.65	12004.00	0.76	8049.70	0.58	10177.70	0.81	18532.20	0.69	11556.60
22	0.62	10479.70	0.71	9757.40	0.53	8843.30	0.95	17618.00	0.80	15265.20

23	0.57	9507.20	0.69	8922.80	0.69	12876.30	0.93	15751.60	0.81	18436.60
24			0.68	8119.90	0.70	14223.20	0.89	21158.70	0.78	16831.40
25	0.56	9720.20	0.56	6615.20	0.74	16520.50	0.93	16539.40	0.80	13328.00
26	0.71	15154.50	0.58	7249.20	0.62	10959.50	0.79	16893.90	0.79	17174.70
27	0.70	12571.40	0.74	5903.70	0.60	9753.90	0.75	12753.10	0.76	14776.60
28	0.70	8641.10	0.83	8213.90	0.63	11290.60	0.90	14599.10	0.72	12461.60
29	0.67	14507.60	0.77	6843.90	0.73	10824.70	0.94	20651.00	0.71	17269.50
30	0.66	10085.80	0.70	5750.30	0.65	11634.00	0.90	18985.20	0.68	14876.80
31	0.67	12603.70	0.72	7339.20	0.81	19075.50	0.74	11713.80	0.63	9734.30
32	0.69	14330.70	0.74	7413.40	0.72	14549.60	0.68	10778.00	0.61	10071.60
33	0.60	13676.90	0.84	11642.20	0.62	9187.30	0.74	13289.20	0.74	17454.20
34	0.69	12080.80	0.68	6586.10	0.65	13133.50	0.70	13946.50	0.63	13565.10
35	0.61	7451.40	0.71	7154.90	0.62	9274.50	0.68	15991.90	0.61	12563.20
36	0.63	11422.20	0.83	8595.50	0.60	7502.60	0.66	11860.40	0.60	10466.40
37	0.67	15287.60	0.60	2921.00	0.66	10725.40	0.77	12985.90	0.66	13274.20
38	0.65	11814.30	0.93	12925.00	0.59	8289.00	0.64	13435.90	0.90	18315.10
39	0.67	11703.30	0.95	11675.40	0.57	11615.70	0.68	8961.40	0.77	14069.40
40	0.61	11948.10	0.91	11270.90	0.56	10618.10	0.64	10004.30	0.76	16282.40
41	0.59	10317.10	0.68	6044.70	0.72	15137.00	0.62	12902.80	0.72	14726.40
42	0.66	11356.20	0.76	8666.80	0.60	9583.70	0.59	9508.70	0.83	19039.00
43	0.59	10870.90	0.69	6422.50	0.60	12065.80	0.61	9201.70	0.68	13852.50
44	0.64	10715.60	0.62	5541.30	0.67	9067.10	0.66	10791.70	0.68	12828.30
45	0.60	10944.60	0.67	6787.70	0.64	7960.70	0.52	8220.20	0.67	9022.40
46	0.55	7569.20	0.68	7778.10	0.66	7392.80	0.53	8214.70	0.62	14109.70
47	0.49	8843.30	0.73	5089.00	0.61	8492.10	0.71	10722.00	0.62	10645.20
48	0.67	10338.90	0.73	7404.80	0.56	7737.60	0.67	10277.00	0.66	12309.80
49	0.56	9422.20	0.63	4999.00	0.60	7168.80	0.62	11211.30	0.67	10909.60
50	0.56	10868.50	0.59	4844.70	0.76	12749.60	0.61	11291.40	0.62	11623.70
51	0.53	10145.50			0.70	13852.90	0.60	10874.30	0.59	10327.20
52					0.65	10612.50	0.54	7206.50		
53							0.59	9854.40		
mean	0.63	11349.18	0.75	8099.63	0.66	11400.77	0.74	13801.98	0.73	13947.39
s.d.	0.05	1833.40	0.10	2386.92	0.07	2838.81	0.12	4323.48	0.09	3293.49

| Nr. | MOE versus rate of compression | | | | | | | |
| | C-1-C | | C-2-C | | C-3-C | | C-5-C | |
	Density (g/m³)	Modulus of elasitci-ty (MPa)	Density (g/m³)	Modulus of elasit-city (MPa)	Density (g/m³)	Modulus of elasit-city (MPa)	Density (g/m³)	Modulus of elasit-city (MPa)
1	0.64	10092.60	0.96	14041.50	0.84	13449.70	0.62	10737.50
2	0.62	9294.60	0.87	14654.60	0.70	8873.80	0.66	7893.40
3	0.62	10262.70	0.86	16179.20	0.68	10531.50	0.60	6745.90
4	0.57	10618.30	0.83	17999.80	0.71	13427.70	0.59	10041.20
5	0.57	10081.50	0.80	12338.50	0.74	15359.70	0.60	13925.50
6	0.51	7314.20	0.82	13419.50	0.68	9168.30	0.58	8398.00
7	0.73	14443.50	0.88	21465.20	0.75	7707.80	0.57	9008.80
8	0.65	12660.20	0.90	21605.20	0.90	18800.40	0.65	13755.80
9	0.59	9103.30	0.77	8592.00	0.89	17909.80	0.52	9690.00
10	0.60	11634.70	0.81	13931.70	0.70	7269.80	0.51	8603.60
11	0.72	15417.70	0.76	10226.90	0.89	18699.90	0.61	10343.40
12	0.68	10907.60	0.81	14000.60	0.81	18373.50	0.58	8624.40
13	0.70	12953.70	0.83	16299.30	0.81	24236.70	0.58	10187.50
14	0.69	12512.50	0.81	13577.20	0.72	11866.00	0.53	8166.40
15	0.69	15219.20	0.77	14953.90	0.75	14474.50	0.52	6869.20
16	0.61	9246.30	0.75	10554.60	0.70	14357.00	0.54	8099.10
17	0.62	13410.70	0.75	13919.30	0.88	18564.10	0.58	9293.70
18	0.60	12829.70	0.75	14753.60	0.91	18457.10	0.51	6938.70
19	0.66	10135.20	0.70	13114.00	0.89	20342.10	0.49	8529.70
20	0.58	9771.40	0.75	11736.90	0.89	22909.40	0.50	7923.40
21	0.61	10275.60	0.72	11216.80	0.81	18532.20	0.60	8004.70
22	0.49	8609.10	0.80	12666.30	0.95	17618.00	0.58	10734.20
23	0.66	9298.60	0.69	12904.90	0.93	15751.60	0.59	9289.00
24	0.69	9178.60	0.66	12305.30	0.89	21158.70	0.59	9488.90
25	0.72	11533.30	0.64	11401.20	0.93	16539.40	0.60	11774.20
26	0.65	12475.80	0.74	9374.30	0.79	16893.90	0.54	8124.00
27	0.65	12722.90	0.69	9314.90	0.75	12753.10	0.54	8415.90
28	0.61	10214.40	0.61	9725.20	0.90	14599.10	0.54	9946.10
29	0.60	6814.60	0.63	9914.10	0.94	20651.00	0.58	8706.60
30	0.62	9020.20	0.61	7167.70	0.90	18985.20	0.55	10534.90

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31	0.62	9616.40	0.55	8389.50	0.74	11713.80	0.49	8308.90
32	0.50	8089.40	0.57	8090.50	0.68	10778.00	0.48	9035.60
33	0.54	8551.30	0.55	10450.70	0.74	13289.20	0.47	9100.00
34	0.53	8850.70	0.69	11111.70	0.70	13946.50	0.55	9250.80
35	0.75	15264.40	0.63	6616.90	0.68	15991.90	0.50	8751.00
36	0.68	11672.40	0.61	8313.30	0.66	11860.40	0.50	7927.90
37	0.72	11896.10	0.58	11721.10	0.77	12985.90	0.54	8755.40
38	0.64	10524.30	0.71	11802.30	0.64	13435.90	0.49	8800.20
39	0.60	10385.90	0.73	13469.50	0.68	8961.40	0.57	10491.00
40	0.62	11125.20	0.68	14452.50	0.64	10004.30	0.52	9917.00
41	0.55	7376.10	0.69	14510.90	0.62	12902.80	0.62	10155.60
42	0.49	8004.30	0.69	15564.10	0.59	9508.70	0.57	9959.90
43	0.58	7488.40	0.72	12466.10	0.61	9201.70	0.51	7467.70
44	0.50	6503.20	0.68	8248.30	0.66	10791.70	0.47	8191.30
45	0.64	10035.80	0.64	11355.80	0.52	8220.20	0.57	9127.40
46	0.56	10632.40	0.64	10300.90	0.53	8214.70	0.56	8857.70
47	0.60	9785.80	0.61	10175.90	0.71	10722.00	0.52	7171.50
48	0.49	6398.40			0.67	10277.00	0.49	8259.30
49	0.52	10972.30			0.62	11211.30		
50	0.51	8937.40			0.61	11291.40		
51	0.64	12217.60			0.60	10874.30		
52	0.63	11606.60			0.54	7206.50		
53	0.54	8475.20			0.59	9854.40		
54	0.54	10100.20						
55	0.66	8085.70						
56	0.68	8834.60						
57	0.64	8914.00						
58	0.61	10329.80						
59	0.62	8778.20						
60	0.56	9937.40						
mean	0.61	10290.70	0.72	12348.81	0.74	13801.98	0.55	9131.71
s.d.	0.07	2106.94	0.10	3235.00	0.12	4323.48	0.05	1481.41

	MOE versus compression time									
	C-3-A		C-3-B		C-3-C		C-3-D		C-3-E	
Nr.	Density (g/m³)	Modulus of elasitcity (MPa)	Density (g/m³)	Modulus of elasitcity (MPa)	Density (g/m³)	Modulus of elasitcity (MPa)	Density (g/m³)	Modulus of elasitcity (MPa)	Density (g/m³)	Modulus of elasitcity (MPa)
1	0.70	7594.00	0.59	9638.40	0.84	13449.70	0.78	11096.10	0.89	11598.00
2	0.87	13252.60	0.62	7949.80	0.70	8873.80	0.66	12814.90	0.87	15545.40
3	0.84	17790.80	0.58	10665.00	0.68	10531.50	0.81	10256.00	0.86	17801.20
4	0.83	17477.90	0.60	9965.70	0.71	13427.70	0.66	8642.50	0.88	20348.50
5	0.89	19219.90	0.68	11491.90	0.74	15359.70	0.59	11749.50	0.80	13294.60
6	0.75	11445.90	0.64	13031.60	0.68	9168.30	0.64	10016.30	0.83	18135.50
7	0.76	9972.00	0.64	14017.00	0.75	7707.80	0.67	11885.00	0.78	16503.10
8	0.71	10420.90	0.61	13231.80	0.90	18800.40	0.65	8596.80	0.81	12951.40
9	0.73	13489.00	0.53	13351.30	0.89	17909.80	0.55	10505.70	0.80	14133.50
10	0.72	12525.10	0.52	10617.90	0.70	7269.80	0.55	7814.60	0.74	11512.20
11	0.68	8806.40	0.52	11299.20	0.89	18699.90	0.68	12251.00	0.79	16180.00
12	0.87	10808.40	0.62	11857.50	0.81	18373.50	0.67	11636.20	0.75	14912.20
13	0.89	13082.30	0.60	12188.50	0.81	24236.70	0.68	11645.00	0.70	13416.20
14	0.90	15351.40	0.60	12171.10	0.72	11866.00	0.58	9492.00	0.75	12119.20
15	0.78	11352.10	0.57	10665.30	0.75	14474.50	0.60	11176.20	0.74	14004.30
16	0.81	11098.50	0.51	10772.90	0.70	14357.00	0.59	11146.50	0.74	13100.10
17	0.72	8803.30	0.55	13073.30	0.88	18564.10	0.71	10923.90	0.75	17233.50
18	0.74	10575.80	0.55	10239.90	0.91	18457.10	0.73	11432.10	0.69	15260.80
19	0.72	10104.80	0.55	11909.20	0.89	20342.10	0.74	9799.80	0.69	13778.20
20	0.69	11044.00	0.55	7066.00	0.89	22909.40	0.73	19762.90	0.69	17398.90
21	0.68	8891.70	0.49	10487.20	0.81	18532.20	0.71	15246.20	0.69	12888.60
22	0.65	10139.10	0.48	10968.70	0.95	17618.00	0.69	12857.20	0.68	14886.60
23	0.62	10389.90	0.48	7469.30	0.93	15751.60	0.68	10437.40	0.79	12096.50
24	0.59	9737.30	0.58	12008.60	0.89	21158.70	0.66	11269.00	0.70	14009.40
25	0.69	11567.60	0.56	11255.40	0.93	16539.40	0.64	11245.90	0.72	11070.40

26	0.62	8878.50	0.51	9942.90	0.79	16893.90	0.64	11925.10	0.64	8623.60
27	0.67	11398.70	0.47	7669.00	0.75	12753.10	0.66	13322.70	0.65	9648.80
28	0.66	13141.00	0.52	8696.60	0.90	14599.10	0.66	15708.40	0.61	10712.60
29	0.61	9460.70	0.55	10999.50	0.94	20651.00	0.75	14602.50	0.58	9998.90
30	0.72	11099.10	0.56	9923.40	0.90	18985.20	0.71	12246.10	0.56	8479.20
31	0.62	10439.90	0.49	8921.00	0.74	11713.80	0.80	11523.20	0.69	12867.50
32	0.68	10568.40	0.49	9812.20	0.68	10778.00	0.71	15185.70	0.66	12942.20
33	0.62	10736.30	0.48	8953.30	0.74	13289.20	0.73	10769.20	0.61	11455.10
34	0.67	13443.30	0.65	11889.80	0.70	13946.50	0.67	15462.10	0.62	12950.90
35	0.62	11349.60	0.59	9524.50	0.68	15991.90	0.61	13140.30	0.75	11083.30
36	0.65	8331.30	0.48	5551.30	0.66	11860.40	0.64	11055.70	0.70	13276.90
37	0.63	9077.10	0.52	11050.20	0.77	12985.90	0.77	11675.90	0.69	12635.40
38	0.60	5172.60	0.64	13464.40	0.64	13435.90	0.69	13832.20	0.62	9137.70
39	0.60	8994.80	0.59	13249.40	0.68	8961.40	0.72	10105.00	0.67	12666.60
40	0.60	8655.80	0.55	10016.60	0.64	10004.30	0.66	12560.50	0.66	9746.80
41	0.56	9545.70	0.54	10309.10	0.62	12902.80	0.61	11037.20	0.70	12352.70
42	0.59	7345.20	0.51	10895.30	0.59	9508.70	0.62	10869.50	0.66	14279.60
43	0.58	7010.00	0.47	7983.30	0.61	9201.70	0.61	8167.50		
44	0.53	7667.60	0.83	15642.80	0.66	10791.70	0.59	15951.40		
45	0.53	6294.40	0.73	14959.90	0.52	8220.20	0.56	10146.80		
46	0.51	5292.30	0.70	15025.70	0.53	8214.70	0.62	14068.50		
47	0.59	8841.70	0.84	19232.40	0.71	10722.00	0.68	9576.90		
48	0.58	8131.70	0.87	15903.70	0.67	10277.00	0.72	16076.20		
49	0.61	10410.10	0.81	18222.00	0.62	11211.30	0.64	9834.30		
50	0.52	8580.90	0.78	13665.50	0.61	11291.40	0.62	9672.60		
51	0.52	8922.90	0.72	13294.50	0.60	10874.30	0.86	19854.40		
52	0.52	7514.50	0.78	17432.00	0.54	7206.50	0.78	12670.20		
53	0.64	10432.40			0.59	9854.40	0.81	15345.00		
54	0.61	9349.30					0.79	17866.00		
55	0.61	7216.40					0.70	10640.40		
56	0.58	5948.20					0.63	9136.50		
57	0.56	9141.80					0.65	10589.70		
58	0.53	5730.80					0.66	10267.70		
59	0.82	15048.10					0.65	11128.50		
60	0.86	20744.60								

Nr.	Density	Modulus of rupture	Density	Modulus of rupture	Density	Modulus of rupture	Density	Modulus of rupture	Density	Modulus of rupture
61	0.81	20089.50								
62	0.76	11776.40								
mean	0.67	10592.30	0.59	11531.21	0.74	13801.98	0.68	12029.03	0.72	13262.76
s.d.	0.11	3357.58	0.10	2781.82	0.12	4323.48	0.07	2601.85	0.08	2667.36

MOR versus conditioning time										
	E-3-C		A-3-C		B-3-C		C-3-C		D-3-C	
Nr.	Density (g/m³)	Modulus of rupture (MPa)	Density (g/m³)	Modulus of rupture (MPa)	Density (g/m³)	Modulus of rupture (MPa)	Density (g/m³)	Modulus of rupture (MPa)	Density (g/m³)	Modulus of rupture (MPa)
1	0.63	105.7	0.72	112.2	0.68	117.1	0.73	104.1	0.78	120.9
2	0.62	109.4	0.85	109.1	0.69	118.2	0.78	106.1	0.87	129.5
3	0.62	108.3	0.76	111.0	0.77	131.1	0.80	106.5	0.70	112.8
4	0.64	104.0	0.87	108.6	0.64	110.7	0.85	108.7	0.70	113.2
5	0.60	114.6	0.68	113.0	0.65	112.3	0.90	110.7	0.78	120.7
6	0.69	89.1	0.72	112.0	0.65	111.6	0.86	108.8	0.73	116.5
7	0.65	99.3	0.74	111.7	0.68	117.7	0.71	103.1	0.64	107.8
8	0.65	101.0	0.84	109.1	0.60	103.5	0.68	101.9	0.74	116.7
9	0.61	110.3	0.68	113.0	0.65	111.7	0.60	98.9	0.72	114.6
10	0.61	110.3	0.67	113.3	0.64	110.2	0.63	100.0	0.64	107.3
mean	0.63	105.20	0.75	111.30	0.66	114.40	0.75	104.90	0.73	116.00
s.d.	0.02	7.32	0.07	1.77	0.05	7.32	0.10	3.94	0.07	6.60

MOR versus compression rate								
	C-1-C		C-2-C		C-3-C		C-5-C	
Nr.	Density (g/m³)	Modulus of rupture (MPa)	Density (g/m³)	Modulus of rupture (MPa)	Density (g/m³)	Modulus of rupture (MPa)	Density (g/m³)	Modulus of rupture (MPa)
1	0.60	121.0	0.86	136.1	0.73	104.1	0.61	71.0
2	0.62	86.0	0.84	132.6	0.78	106.1	0.57	96.0
3	0.70	101.0	0.80	127.3	0.80	106.5	0.56	85.0

Nr.	Density	Modulus of rupture	Density	Modulus of rupture	Density	Modulus of rupture	Density	Modulus of rupture
4	0.61	118.0	0.74	119.6	0.85	108.7	0.52	90.0
5	0.63	97.0	0.70	115.0	0.90	110.7	0.59	98.0
6	0.63	106.0	0.66	108.9	0.86	108.8	0.55	110.0
7	0.59	133.0	0.60	101.1	0.71	103.1	0.50	83.0
8	0.65	132.0	0.66	109.6	0.68	101.9	0.53	82.0
9	0.55	115.0	0.69	112.8	0.60	98.9	0.55	76.0
10	0.54	94.0			0.63	100.0	0.55	72.0
mean	0.61	110.30	0.73	118.11	0.75	104.90	0.55	86.30
s.d.	0.05	16.06	0.09	11.74	0.10	3.94	0.03	12.39

MOR versus compression time										
Nr.	C-3-A			C-3-B		C-3-C		C-3-D		C-3-E
Nr.	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)
1	0.83	58.5	0.61	98.2	0.73	104.1	0.70	110.9	0.86	164.7
2	0.73	70.8	0.59	92.6	0.78	106.1	0.61	99.4	0.79	139.9
3	0.82	58.6	0.58	90.9	0.80	106.5	0.64	103.5	0.75	125.4
4	0.74	70.2	0.54	82.0	0.85	108.7	0.70	110.5	0.71	112.9
5	0.65	82.0	0.52	77.0	0.90	110.7	0.68	107.4	0.72	113.9
6	0.66	80.8	0.52	77.4	0.86	108.8	0.68	108.5	0.61	76.3
7	0.64	82.5	0.54	81.6	0.71	103.1	0.71	111.4	0.67	97.2
8	0.62	86.1	0.56	85.0	0.68	101.9	0.69	109.7	0.67	97.7
9	0.56	93.6	0.62	98.5	0.60	98.9	0.60	97.6		
10	0.56	93.3	0.80	139.7	0.63	100.0	0.66	105.0		
11	0.58	90.5								
mean	0.67	78.82	0.59	92.30	0.75	104.90	0.67	106.40	0.72	116.00
s.d.	0.10	12.66	0.08	18.40	0.10	3.94	0.04	4.88	0.08	27.56

	MOE versus conditioning time (5-layer laminate)									
	E-3-C		A-3-C		B-3-C		C-3-C		D-3-C	
Nr.	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)
1	0.63	11665.10	0.72	12397.22	0.68	10860.54	0.73	7325.27	0.78	11524.49
2	0.62	10584.92	0.85	16607.90	0.69	12341.84	0.78	6274.36	0.87	16976.44
3	0.62	11075.91	0.76	11015.21	0.77	15160.92	0.80	6113.85	0.70	13923.94
4	0.64	12007.11	0.87	10770.30	0.64	11462.72	0.85	11171.18	0.70	13611.35
5	0.60	9007.55	0.68	14709.35	0.65	11629.57	0.90	11014.16	0.78	14991.46
6	0.69	8210.36	0.72	14026.23	0.65	11385.49	0.86	19128.54	0.73	9486.88
7	0.65	8864.27	0.74	8730.79	0.68	12840.25	0.71	15108.50	0.64	9371.22
8	0.65	8335.70	0.84	8292.58	0.60	11207.41	0.68	15091.13	0.74	14089.12
9	0.61	9793.30	0.68	11562.78	0.65	9918.56	0.60	10443.48	0.72	11518.15
10	0.61	8454.94	0.67	11667.15	0.64	12677.11	0.63	12587.27	0.64	10335.24
mean	0.63	9799.92	0.75	11977.95	0.66	11948.44	0.75	11425.77	0.73	12582.83
s.d.	0.02	1436.74	0.07	2583.03	0.05	1426.29	0.10	4222.24	0.07	2526.20

	MOE versus compression rate (5-layer laminates)							
	C-1-C		C-2-C		C-3-C		C-5-C	
Nr.	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)
1	0.60	9949.84	0.86	14703.53	0.73	7325.27	0.61	9707.04
2	0.62	11578.45	0.84	16622.38	0.78	6274.36	0.57	9294.06
3	0.70	10406.80	0.80	11680.62	0.80	6113.85	0.56	7836.58
4	0.61	11400.96	0.74	13383.56	0.85	11171.18	0.52	9073.02
5	0.63	14145.37	0.70	13393.06	0.90	11014.16	0.59	8521.86

Nr.	Density	Modulus	Density	Modulus	Density	Modulus	Density	Modulus
6	0.63	11600.62	0.66	13234.00	0.86	19128.54	0.55	8366.27
7	0.59	13447.84	0.60	12012.00	0.71	15108.50	0.50	7512.44
8	0.65	14121.36	0.66	12493.13	0.68	15091.13	0.53	8596.51
9	0.55	13937.05	0.69	11773.00	0.60	10443.48	0.55	6911.49
10	0.54	10870.16			0.63	12587.27	0.55	7235.42
mean	0.61	12145.85	0.73	13255.03	0.75	11425.77	0.55	8305.47
s.d.	0.05	1614.26	0.09	1591.93	0.10	4222.24	0.03	919.12

MOE versus compression time (5-layer laminates)										
Nr.	C-3-A		C-3-B		C-3-C		C-3-D		C-3-E	
Nr.	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)	Density (g/m^3)	Modulus of rupture (MPa)
1	0.83	3182.03	0.61	9439.32	0.73	7325.27	0.70	13062.47	0.86	21583.89
2	0.73	5861.91	0.59	10411.00	0.78	6274.36	0.61	10515.06	0.79	14680.85
3	0.82	3710.75	0.58	9267.00	0.80	6113.85	0.64	12468.90	0.75	15470.92
4	0.74	6211.86	0.54	7971.88	0.85	11171.18	0.70	11110.45	0.71	12309.64
5	0.65	11113.08	0.52	7851.67	0.90	11014.16	0.68	10678.65	0.72	12900.50
6	0.66	9402.52	0.52	7686.11	0.86	19128.54	0.68	9971.78	0.61	11202.62
7	0.64	10570.35	0.54	8929.18	0.71	15108.50	0.71	12185.70	0.67	11318.78
8	0.62	9212.01	0.56	9596.00	0.68	15091.13	0.69	10398.97	0.67	12122.79
9	0.56	12251.70	0.62	10907.55	0.60	10443.48	0.60	11233.91		
10	0.56	8743.65	0.80	16351.18	0.63	12587.27	0.66	10578.39		
mean	0.68	8025.99	0.59	9841.09	0.75	11425.77	0.67	11220.43	0.72	13948.75
s.d.	0.10	3122.26	0.08	2523.70	0.10	4222.24	0.04	1018.33	0.08	3437.73

Nr.	Compressive shear strength (MPa) from the different resin solid loading rates (g*cm^{-2})			
	25	50	70	Untreated
1	33.3	60.3	70.4	30.7
2	33.4	62.2	60.6	19.3
3	52.5	63	65	23.1
4	69.3	74.5	59.8	26
5	20.7	47.4	17.05	23.7
6	46.5	27.4	40.1	
7	48.5	42.9	67.9	30
8	20	50.1	51.2	24.5
9	22.03	69.8	63.2	35.2
10	88.8	62.6	43	33.2
11	50.3	56	76.1	
12	60.2	68	51.5	
13	51.4	61.7	40.5	
14	46.2	64.5	51.1	
15	60.9	88.7	62.5	
16	49.1	101.4	74.7	
17	43.1	56	63	
18	38.7	42.6	37.2	
19	54.8	39.8	50.7	
20	55.4	88.7	68.8	
21	42.9	63	81.4	
22	36	64.5	59.2	
23		52.4		
24		63		
25		82.9		
mean	45.61	61.67	57.61	27.30
s.d.	16.36	16.47	14.99	9.95

s	Shear failure stress versus conditioning time (MPa)				
	30	90	120	180	240
	E-3-C	A-3-C	B-3-C	C-3-C	D-3-C
1	39.9	37.3	55	67.8	46.8
2	66.9	49.9	70.6	59.1	47.4
3	55.7	60.9	79.8	52	52.1
4	88	65.6	68	57.3	75.1
5	59	52.6	64.1	62.2	72.7
6	71.4	62.3	69	55	55.1
7	62.3	83.4	56	52.2	50
8	64.6	49.9	67	66.5	47.3
9	60.3	42	71.5	50.4	46.4
10	42.8	62.5	52.8	53.1	66.2
11	62.7	69.3	63.1	58.1	54.9
12	64.2	73.3	64.3	51.8	44.6
13	57.9	66.6	72.5	82.5	52.7
14	67.6	73.1	68.2	76.9	35
15	65.1		55.4	56.5	37.5
16	73.2		75.4	75.6	46.4
17	49.7		71.6	76.3	69.2
18	53.3		61.2	66.6	80.1
19	49.6		63.4	61.1	59.9
20	66.7		61.2	59.7	57.9
21	54.3		36.3	62	56
22	64.9		84.9	66.4	59.3

23	54.8		67.1	80.2	74.8
24	40.6		64.1	78.6	50.6
25	52.7		64.7	78.8	42.4
26	76.9		56.2	89.1	
27	65.4		56	70	
28	70.6		63.6		
29	54.8		61.7		
30	60.2		65.9		
mean	60.54	60.62	64.35	65.40	55.22
s.d.	10.60	12.90	9.06	11.00	12.04

Shear failure stress versus rate of compression (MPa)

speed	7	14	29	61
	C-1-C	C-2-C	C-3-A	C-5-C
1	50.80	47.1	68.5	42.7
2	104.1	52.3	66.9	24.03
3	61.40	66.1	53.7	64.7
4	69.20	69	60.5	45
5	64.40	64.7	51.9	56.1
6	72.00	56.8		50.2
7	56.10	42		53
8	54.70	68.9		53
9	65.90	53		57.4
10	57.2	67		56.2
11	57.9	51		65.5
12	52.9			50.1

13	40.5			76.2
14	52.9			56.6
15	69.5			104.2
16	50.8			48.7
17	49.3			66.7
18	48.5			
19	49.2			
20	82.3			
21	61.9			
22	65.4			
23	54.7			
24	55			
25	60.6			
26	80.1			
27	63.1			
28	48.8			
mean	58.83	54.33	55.08	57.30
s.d.	19.14	21.24	24.64	20.52

	Shear failure versus compression time				
CT	120	150	180	210	240
	C-3-A	**C-3-B**	**C-3-C**	**C-3-D**	**C-3-E**

1	68.5	32.8	67.8	48.4	61.7
2	66.9	21.7	59.1	78.3	69.4
3	53.7	51.5	52	50.4	54.4
4	60.5	54.7	57.3	48.8	43.3
5	51.9	62.1	62.2	74.4	36.7
6		85	55	44.3	40.5
7		63.2	52.2	52	55.2
8		62.3	66.5	51.5	36.3
9		36.7	50.4	48	35.5
10		36.7	53.1	40.6	81.4
11		55.8	58.1	60.3	60.1
12		50.3	51.8	42.9	58.5
13		50.9	82.5	59.9	66.2
14		59.7	76.9	64.8	62
15		51.2	56.5	40.5	65.6
16		56.1	75.6	42.1	52.9
17		65.9	76.3	51.3	68.2
18		75.1	66.6	54.6	79.6
19		42.2	61.1	49.7	58.5
20		62.9	59.7	47.1	54.3
21		53.4	62	57.1	60.8
22		50.1	66.4	62.6	68.9
23		73.3	80.2	58.7	76.8
24		70.7	78.6	64.9	90
25		54.2	78.8	57.6	69.6
26			89.1	56.2	91.3
27			70	49.4	86
28					88.8

29					62
mean	60.30	55.14	65.40	53.94	63.26
s.d.	25.51	17.56	16.41	13.85	19.46