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THE ROLE OF LIGNIN PLASTICIZATION IN SOME WOODWORKING PROCESSES

ABSTRACT

Wood is a mixture of polymers, composed of cellulose microfibrils and amorphous hemicellulose and lignin molecules. The lignin phenyl propane units create chains, which are crosslinked in an amorphous, highly branched three-dimensional structure, linked to the cellulose fibrils through the hemicelluloses. The components of the wood can behave similarly to the artificial polymers: the state of it can be glassy, elastic and melted.

The moist lignin softens at about 100°C and allows the molecules to move in the cell walls. The moist hemicellulose and amorphous cellulose are in elastic state in these conditions, so during the described industrial processes, when the wood softens, the lignin plays the main role, but of course other physical and chemical reactions take place too. All of these reactions contribute to the end result of the process, but this review did not aim to map these reactions in detail, but rather to provide a summary of the industrial processes where lignin softening may play a role. These processes are pellet manufacturing, binderless panel manufacturing, wood welding, wood bonding, wood surface compacting, and veneer manufacturing by peeling.

Keywords: glass transition temperature, lignin, melting, plasticization, wood

INTRODUCTION

Wood composed of partially crystalline cellulose microfibrils, hemicellulose and amorphous lignin molecules. Lignin is built up from phenyl propane units, the chains of it are crosslinked in a three-dimensional structure. Lignin linked to the cellulose fibrils through the hemicelluloses (Rowell et al. 2005). The amount of lignin varies between species, tissues, cell types, and cell wall layers. The greatest concentrations of lignin were found in the compound middle lamella and the cell corners; lower concentrations were found in the secondary cell walls (Fergus et al. 1969; Westermark et al. 1988; Fromm et al. 2003; Gierlinger and Schwanninger 2006). Although the lignin concentration in the middle lamella and in the cell corners is high, the volume of the secondary cell wall layers is much higher, so most of the lignin was located in the secondary walls (Fergus and Goring 1970a, 1970b; Adler 1977, Saka and Goring 1985). The lignin concentration of the vessel wall is higher than that of the fiber, while ray parenchyma cells have a lower lignin concentration than that of the fibers (Saka and Goring 1985).

The state of a polymer can be glassy, elastic and melted. In the glassy state, the parts of the polymer macromolecule can only oscillate. If the temperature is increasing, the internal energy of the molecule is increasing too and the parts and segments of the molecules can move, but the relative position of the center of mass of the molecular does not change. This is the flexible state, where the polymer has a high degree of reversible deformation. The further internal energy

growth melt the polimers, where the molecules move relative to one another and the polymers flow (Miskolczi 2012). Many amorphous polymers change from a glassy state to a rubbery, elastic state above a certain temperature, called the glass transition temperature (T_g), as they plasticize (Hatakeyama and Hatakeyama 2010; Miskolczi 2012).

The temperature required for lignin to transition to glass (T_g) is influenced by a number of factors, such as the presence of rigid phenolic side groups on the main chain, the presence of crosslinking, the number of bonds between chains, hydrogen bonds, its molecular weight, species, lignin conformation, and thermal pre-history, etc. (Chow and Pickles 1971; Gellerstedt 2015; Furuta et al. 2008; 2010; Hatakeyama and Hatakeyama 2010). When dry, the differences in the glass transition temperature of cellulose, hemicellulose and lignin is not big: 200 to 250°C for the amorphous region of cellulose, 150 to 220°C for hemicelluloses, and around 205°C for lignin (Goring 1963; Back and Salmén 1982). Many researchers have found that water molecules can break hydrogen bonds inside and between large molecules and segmental motion can occur easily, so the T_g of lignin decreases with increasing moisture content until the wood or the lignin reaches its water saturated point (Goring 1963; Back and Salmén 1982, Morsing and Hoffmeyer 1998; Hatakeyama and Hatakeyama 1998, 2010).

Other wood constituents in a native wood cannot be separated from lignin. The softening temperature of native hemicellulose under dry conditions is around 180°C (Back and Salmén 1982; Olsson and Salmén 2003). Water works like plasticizer and decreases the glass transition temperature of the hemicelluloses too: at 20% water content it is around 50°C, at 30% it is around room temperature (Back and Salmén 1982; Olsson and Salmén 2003; Navi and Sandberg 2012). The amorphous regions of cellulose behave similarly.

This type of wood softening – mainly the lignin – is used in several industrial processes, namely: pellet manufacturing, binderless panel manufacturing, wood welding, wood bonding, wood surface compacting, and veneer manufacturing by peeling. Of course, during these industrial processes, in addition to softening of other components, other physical and chemical reactions take place, all of which contribute to the end result of the process. We did not aim to map these reactions in detail, but rather to provide a summary of the industrial processes where lignin softening may play a role.

WOOD WELDING

Frictional wood welding is a relatively new technology for creating wood joints but this technique is widely used in the plastics industry (Ganne-Chédeville et al. 2006). The welding processes can be classified as linear, orbital and rotational friction welding (Ruponen et al. 2015). During the welding process, no need to add other material to the system, the welded pieces of wood are friction together to provide the necessary energy. After a few seconds (3-10 s) of friction, at a temperature of about 320-350°C, the wood surfaces start to decompose at the increased temperature. The wood next to the rubbed surfaces starts to soften, forming a viscous film. After reaching the maximum temperature about 420-450°C the frictional movement is terminated, but the joined parts are held together. The final cooling down leads to solidification of the interfacial film forming the connection between the wood parts (Stamm et al. 2005b).

The mechanism of welding is, in addition to the chemical reactions that take place, due to the temperature-induced softening, flowing and solidification of the intercellular material, mainly amorphous polymers: lignin and hemicelluloses (Stamm et al. 2005a). This flow of material induces high densification of the bonded interface (Ganne-Chédeville et al. 2006; Pizzi 2017). The physical entanglement of the fibers interconnected as a result of friction can improve the

connection. In the brief pressure-holding phase immediately after welding, chemical reactions occur. The main reactions are the formation and self-condensation of furfural and the cross-linking reaction of lignin with carbohydrate-derived furfural (Pizzi 2017).

The chemical changes during the friction welding similar to fast pyrolysis at lower pyrolysis temperature: bonds broke, free radicals are formed, which are then participate in re-polymerization and side-chain conversions (Kawamoto 2017). In these temperature ranges, only the amorphous components of wood are affected, lignin, hemicelluloses, and the amorphous cellulose. The amount of typical bonds of phenilpropane units decreases, while furfural and furan derivatives from hemicelluloses react with the lignin (Belleville et al. 2013; Sun et al. 2010).

The welded bondline is a mass of entangled long wood cells immersed in a matrix of amorphous, fused intercellular material, mostly lignin but also including some hemicelluloses. The bonding line can separate to melting zone, were the wood polymers are melted, decomposed and partly charred; to fully plasticized and deformed zone, where the polymers are plasticized and the cells are deformed, and a partial deformed region, where the cells are distinguished, but some of them deformed (Ganne-Chédeville et al. 2006).

The quality of a friction welding joint correlates with several welding parameters, such as welding pressure, frequency, time, holding pressure and time, amplitude or displacement, wood species, orientation of the grains, EMC, and specimen dimensions, chemical composition etc. (Kanazawa et al. 2005; Ganne-Chédeville et al. 2006, 2008; Belleville et al. 2018).

PELLET MANUFACTURING

Ligno-cellulosic materials can be densified by pelletizing, briquette or cube making. During this process, the particles are forced together by applying mechanical force and inter-particle bonding is created (Tumuluru et al. 2010; Kaliyan and Morey 2010). The name 'pellet' is usually used for product less than 15 mm in diameter, while 'briquette' is generally a larger densified material unit. Several studies examined the compression different raw materials such as wood, wood waste and bark, forest residues, straws, grasses (wheat, barley, corn etc.), olive cake (waste), palm fiber and shell. Since pellet production has been studied most frequently and in most detail, this is described in the next sections, although most statements are true for other densifying procedures (Tumuluru et al. 2010).

During the process, the size of the raw material is reduced by milling or grinding, and conditioned to an appropriate moisture content either by drying or moisturizing. In most cases the particles of raw material are forced through a channel. In a pellet mill the pressure is 100–150-200 MPa (Kaliyan and Morey 2009). The pelletizing process generates heat that maintains the temperature of the operating die at 110-130°C (Nielsen et al. 2009). During pelleting, the raw material with 8-15% moisture content the lignin softens around 110-135°C (Kuokkanen et al. 2011; Kaliyan and Morey 2009). As the pellets cool, lignin hards again and the pellet strength increases.

Macroscopically two binding mechanisms can be distinguished: solid bridges between particles (Stelte et al. 2011a; Serrano et al. 2011) and other bonding with , hydrogen bridges, van der Waals' forces, electrostatic, and magnetic forces (Kaliyan and Morey 2010). Due to the application of high pressures and temperatures, solid bridges can develop by diffusion of molecules from one particle to another at the points of contact and are formed by a chemical reaction, hardening of the binders, and solidification of the melted components (Kaliyan and Morey 2010). Pressure, heat above glass transition temperature, and a solvent such as water are used to promote adhesion by increasing the molecular contact.

Several factors were found to influence the process and the result of the densification experiments (Kaliyan and Morey 2009). The increasing temperature and MC decreased the energy requirements for pellet manufacturing (Nielsen et al. 2009). The increasing temperature of die increases the pellet density, decreases the dimensional expansion, and increases the tensile strength of the pellets. (Kaliyan and Morey 2006). Decreasing the particle size increases the pellet density, decreases their expansion, and increases the tensile strength of the pellets (Lehtikangas 2001).

The biochemical characteristics of the raw material are also very important, higher lignin and extract content has a positive effect on the durability (Bradfield and Levi 1984; Lehtikangas 2001; Stelte et al. 2011a; Serrano et al. 2011).

Pretreatment of the raw material generally increases the pellet density and durability by activating lignin (Shaw 2008; Zandersons et al. 2004).

WOOD SURFACE DENSIFICATION

The process of densifying wood by compression requires four steps (Kutnar and Šernek 2007; Rautkari et al. 2011): 1. plasticization of the cell wall, 2. compression perpendicular to the grain in the softened state, 3. cooling and drying in the deformed state, and 4. fixation of the deformed state to eliminate the shape memory effect. The density is increasing, the color of the wood become darker and the EMC is reduced (Arruda et al. (2015).

In the first the cell wall should soften. The temperature should be at least 25°C higher than T_g of lignin, between 80-140°C, the moisture content is near the saturation point. Under these conditions, lignin, hemicelluloses and the semi-crystalline cellulose are relatively mobile and can be deformed easily. (Kutnar and Šernek 2007; Kutnar et al. 2009; Rautkari et al. 2011).

Pressing changes the wood morphology, is buckling the cell walls and reducing the volume of void spaces, but a non-uniform density profile is created as the surface becomes denser (Kutnar et al. 2009).

When compressed wood meets with water, it approximates its original dimensions. This effect occurs because internal stresses are introduced into the cell walls during compression, which can be completely eliminated by heat and/or steam treatment (Morsing and Hoffmeyer 1998; Wolcott and Shutler 2003, Kutnar et al. 2009).

BINDERLESS BOARDS MANUFACTURING

Due to its environmentally friendly properties, many researchers tried to produce fiber or particle boards without adhesive or using bio-based adhesives such as sugars or lignin. Binderless fibreboards have been produced for decades and several types of raw material were used: bark, rice and wheat straw, kenaf, banana stems, coconut husks, bagasse, oil, soybean straw and bamboo etc.

Several parameters influence the physical and mechanical properties of the panels: chemical composition (Widyorini et al. 2005; Lui et al. 2018), particle size and geometry (Widyorini et al. 2011; Kurokochi and Sato 2015a; 2015b; Lui et al. 2018; Ahmad et al. 2019), pressing temperature (Boon et al. 2013; Milawarni et al. 2019; Song et al. 2020), pressing time (Boon et al. 2013), pressure (Boon et al. 2013); water content (Widyorini et al. 2005), pretreatment (Xu et al. 2006) etc. Trichomes and wart-like protuberances on the epidermis of herbaceous straw might inhibit the bonding between particles. Wax-like substances on the epidermis of rice straw might

contribute to the water resistance of the board but inhibited the adhesion of the particles (Kurokochi and Sato 2015a; 2015b).

The chemical composition of the different biomasses is similar to wood, the main components are the same: cellulose, lignin, hemicelluloses. The bonds between the bio particles are based on chemical and physical interactions during the hot pressing between various components of the particles (Hubbe et al. 2018). In fiberboard production, during thermomechanical pulping, woodchips are converted to lignin-covered fibers by shearing wood fibers along the lignin-rich middle lamellae. Halvarsson et al. (2009), Wang et al. (2017) and Laine et al. (2019) suggested that lignin has an important role in binderless board production due to the softening of lignin at elevated temperatures and under pressure, fibers with lignin-rich surfaces fuse together as the softened lignin molecules flow from one fiber surface to another one, and possibly form covalent bonds too, the role of the softened lignin can't be separated from the chemical changes.

During hot pressing several chemical reactions have been observed: hydrolysis of the hemicelluloses occurs (Xu et al. 2006; Zhang et al. 2015), mainly furfural is formed. Linakges between the lignin and furfural monomers are formed or self-polymerization occurs during the pressing, which provided the main self-bonding strength of binderless fiberboards (Zhang et al. 2015).

The surfaces of the particles and fibers can be activated by different pretreatments, the amount of the chemical bonds can be increased. Fenton's reagent contains ferrous chloride and hydrogen peroxide, hydroxyl radicals are generated by decomposition of hydrogen peroxide with the assistance of ferrous ions. In the end reactive components formed in lignin, the properties of the boards is improved (Halvarsson et al. 2009; Zang et al. 2015). Enzymatic systems also can activate lignin on fiber surfaces by generating free radicals (Pereira et al. 2005; Nyanhongo et al. 2010). Steam explosion also can improve the panel properties (Anglès et al. 1999; 2001; Gao et al. 2011).

SOLID WOOD BENDING

Wood bending is one of the oldest wood processing techniques. Long experience has evolved from the practice of bending techniques and skilled craftsmen can apply them. Plasticizing treatments can soften wood sufficiently to enable it to make a curve (Peck 1957). The plasticity of wood can be increased by increasing the moisture and/or temperature of the wood. Hot water and steam are commonly used treatments to prepare wood for bending. Some chemicals can also soften wood. The glass transition temperature (T_g) of the lignin in moist wood is 80-100°C. Above T_g the lignin undergoes thermoplastic flow and resets in the modified configuration when cooling. This is the principle behind bending of wood (Nakajima et al. 2009; Ibach 2010). Heat and moisture make certain species of wood sufficiently plastic for bending operations. In general, hardwoods are more readily softened than softwoods, and certain hardwoods more so than others (Peck 1957).

The temperature of saturated steam at atmospheric pressure, about 100°C or boiling or nearly boiling water, is generally sufficient to plastify wood for bending. When chemicals are used for plasticization, the connections between the matrix (lignin) and cellulose and the ties between the cell walls loosen. The chemical plasticization commonly uses water solutions of ammonia, urea, dicyandiamide, ethylenediamine, and ammonia (Angelski 2014). The high-frequency microwave heating of wood is also used for plasticization (Ibach 2010; Gašparík and Gaff 2013).

VENEER MANUFACTURING

Several factors have effect on veneer manufacturing and veneer quality (Olufemi 2012). Successful veneer production requires wood softening. The heating of green wood prior to peeling has traditionally been accomplished by immersing the whole logs in hot water basins or by steaming them in vats.

During soaking or steaming the wood softens, and the deformability of wood is increasing (Baldwin 1975, Bardet et al. 2003, Yamauchi et al. 2005). Under optimal conditions, shallower lathe checks formed (Rohumaa et al. 2016a, 2016b, 2016c). For this purpose, the temperature should exceed the glass transition temperature T_g, of lignin at the MC of green wood across the log. This softening reduces the required energy (Dupleix et al. 2012; 2013; Xu et al. 2017), because the shear strength of the wood decreases and less pressure needed on the knife during peeling. This reduces the power consumption and reduces wear of the tools and the quality of the veneer is improved as influencing the formation of lathe checks and other surface quality (Dupleix et al. 2012; Olufemi 2012; Rohumaa et al. 2016a, 2016b, 2016c).

SUMMARY

Lignin softening plays a role in several woodworking processes, which are basically divided into two groups. The first group includes processes where lignin softens or possibly liquefies, and the soft lignin penetrates into the adjacent particles and cell walls, and acts as an adhesive. This includes wood welding, the manufacture of binderless boards or manufacturing pellets. The other group consists of processes where the wood has to undergo some deformation and therefore the cell walls and the materials that compose the cell wall must move relative to one another. This group includes veneer manufacturing, solid wood bending and surface densification. We hope that a better understanding of these processes can help improve the manufacturing processes.

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