DENSIFICATION OF WOOD VENEERS COMBINED WITH OIL-HEAT TREATMENT. PART I: DIMENSIONAL STABILITY

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Although wood densification by compression improves wood mechanical strength, dimensional stability is often a problem due to compression recovery. Alternatively, oil-heat treatment (OHT) improves wood dimensional stability and enhances resistance to biological attack. This study examined combined wood densification and OHT. Large wood veneer 700 x 700 mm specimens prepared with aspen (Populus tremuloides) were densified using heat, steam, and pressure at 160°C, 180°C, and 200°C, respectively. OHT at 180°C, 200°C, and 220°C for 1, 2, and 3h was then applied to the densified veneers. Results show that OHT efficiently improved dimensional stability and reduced compression set recovery. OHT temperature and duration markedly influenced the reduction of compression set recovery: the higher the OHT temperature and duration, the lower the recovery. Less than 5% recovery was obtained under various OHT conditions, and almost 0% recovery under some OHT conditions. Radial and tangential swellings of densified veneers were reduced dramatically. Compared to OHT duration, OHT temperature had a pronounced higher impact on radial and tangential swelling. Irreversible swelling (IS) in the compression direction of densified veneers decreased after OHT, particularly with high temperature and long duration, and anti-swelling efficiency (ASE) in the compression direction improved significantly.

Keywords: Wood densification; Heat treatment; Compression; Veneer; Aspen; Dimensional stability

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INTRODUCTION

Many wood densification treatments have been attempted to increase wood density and hence improve wood mechanical properties (Boonstra and Blomberg 2007; Fang et al. 2010; Fukuta et al. 2008; Gabrielli and Kamke 2008; Higashihara et al. 2000; Inoue et al. 1993a, b, 2008; Ito et al. 1998; Kamke 2006; Kollmann 1936; Kollmann et al. 1975; Navi and Heger 2004; Seborg et al. 1945). However, wood that is densified under mechanically applied heat and compression is dimensionally unstable. Compression set recovery over time following moisture adsorption is often a problem (Inoue et al. 1993a; Navi and Girardet 2000). Various treatments have been attempted to improve the dimensional stability of compressed wood (Gabrielli and Kamke 2010; Inoue et al. 1993a, b, 2008; Navi and Heger 2004). For instance, thermal treatment is known to

improve wood dimensional stability and enhance resistance to biological attack by reducing wood hygroscopicity (Burmeste 1973). In recent years, thermal wood modification has been successfully commercialized in Europe (Wang and Cooper 2005). Several different processes are used to heat treat wood, including steam, nitrogen, or oil as an oxygen-free heat transfer medium (Wang and Cooper 2005). However, thermal treatments usually reduce the wood's mechanical properties (Korkut and Hiziroglu 2009; Poncsak et al. 2006; Rapp et al. 2006). Consequently, the aim of this study was to investigate the potential of applying a post-heat treatment to densified wood to overcome the drawbacks of densification and heat treatment. Oil is an effective heat transfer media and a potential carrier for other substances to further enhance treatment benefits (Sailer et al. 2000; Wang and Cooper 2005). Oil-heat treatment (OHT) has also been reported to improve wood's biological resistance (Lyon et al. 2007; Sailer et al. 2000). In the present study, OHT was applied to densified wood veneers described in a previous work (Fang et al. 2010). The objective was to determine the effects of OHT temperature and duration on the dimensional stability of densified veneers.

EXPERIMENTAL

Materials

Rotary-peeled aspen (*Populus tremuloides*) wood veneers were obtained from Temlam Inc., a laminated veneer lumber plant in Amos, in the northwest of the province of Quebec, Canada. The nominal thickness of the aspen veneers was 3.2 mm. Veneers were conditioned at 20°C and 60% relative humidity (*RH*) before densification. Veneers of 700 mm \times 700 mm were densified using pressure, heat, and steam, as described in a previous work (Fang et al. 2010, submitted). The theoretical compression set was 50%. Veneers densified at 160°C, 180°C, and 200°C were used for OHT. Densified veneers were conditioned at 20°C and 60% *RH* before OHT. Twelve specimens were prepared for each treatment. Forty control specimens were prepared for non-densified measurements. Mean values of the repetitions were used for analysis.

Oil-heat Treatment

Samples of 50×50 mm (tangential × longitudinal) were cut from densified veneers with a laser cutter and treated in a hot oil vessel at 180°C, 200°C, and 220°C for 1, 2, and 3h. Canola oil (specific gravity 0.91, viscosity 78.2×10^{-6} m² s⁻¹, smoke point 220 to 230 °C) was used, and was pre-heated before testing. Once the samples were immersed in hot oil, the target temperature was maintained. All samples were thin and small to allow the target internal temperature to be reached rapidly and uniformly. After OHT, specimens were removed from the hot oil vessel and kept in a climate room at 20°C and 60% *RH*. Oil remaining on the specimen surface was removed with a paper cloth.

Thickness Change due to OHT

Veneer thickness was measured before (T_B) and after (T_A) OHT at air-dry condition in the climate room. Thickness change (TC) was calculated as follows:

$$TC = \frac{(T_A - T_B)}{T_B} \times 100 \,(\%) \tag{1}$$

Recovery Test

A cyclic recovery test was conducted to determine compression set recovery in the compression direction, i.e., the wood radial direction. After OHT, oven-dried specimens were saturated with water until their weight became stable and oven-dried again. This procedure was repeated five times. After the fifth soaking cycle, specimens were soaked in boiling water for 30 minutes and then oven-dried. The thickness at each saturated and oven-dry condition was recorded. Compression set recovery (R_c) was calculated as follows:

$$R_{C} = \frac{T_{R} - T_{C}}{T_{O} - T_{C}} \times 100 \,(\%) \tag{2}$$

where T_R is the veneer thickness at oven-dry condition during the cyclic recovery test, T_C is the thickness at oven-dry condition after compression followed by OHT, and T_O is the thickness before compression. Tests were conducted on veneers densified with and without OHT.

Swelling

Veneer dimensions in oven-dry and water-saturated condition were measured in the radial, tangential, and longitudinal directions. Swelling was calculated from the expansion in the wet condition as a percentage of the initial oven-dry dimension. Swelling was calculated in the radial, tangential, and longitudinal directions. Control (non-densified) specimens and densified specimens with and without OHT were measured.

Irreversible Swelling

Irreversible swelling (*IS*) due to the compression set recovery following moisture adsorption (Gabrielli and Kamke 2010; Hsu et al. 1988; Rowell et al. 1986) was calculated in the radial direction only, as veneers were compressed in this direction and high IS was expected. Test specimens were first oven-dried and then saturated in water followed by oven-drying again. Specimen thickness was measured in each state and reported as T_{OD0} , T_W , and T_{OD1} , respectively. *IS* was calculated as follows:

$$IS = \frac{T_{OD1} - T_{OD0}}{T_W - T_{OD0}} \times 100 \,(\%)$$
(3)

Anti-Swelling Efficiency

The most frequently used term for quantifying dimensional stability is antiswelling efficiency (*ASE*) (Morsing 2000; Stamm et al. 1946), which is defined as follows,

$$ASE = \frac{S_0 - S_1}{S_1} \times 100 \,(\%) \tag{4}$$

where S_0 is the swelling of control (non-densified) specimens and S_1 is the swelling of treated specimens. Swelling (S) is calculated as,

$$S = \frac{T_W - T_O}{T_O} \times 100 \,(\%) \tag{5}$$

where T_O is the thickness in oven-dry condition and T_W is the thickness in water-saturated condition. In this study, *ASE* was calculated in the radial direction only.

RESULTS AND DISCUSSION

Density and Compression Set

After densification using pressure, heat, and steam, the density of aspen veneer increased significantly. Average oven-dry density and standard deviation for non-densified aspen veneer was $388 \pm 25 \text{ kg/m}^3$. After densification, average oven-dry density and standard deviation was $830 \pm 64 \text{ kg/m}^3$. A slight density increase trend with increasing temperature was found in densified veneers. This could be explained by the variation in compression set, as a similar trend was found for compression set, as presented in detail in a previous work (Fang et al. 2010, submitted).

Thickness Change Due to OHT

Figure 1 shows the thickness change (TC) in densified veneer caused by OHT. Negative values indicate a reduction in veneer thickness. A strong negative correlation between TC and densification temperature was found, except for specimens that were oilheat treated for 1 hour. A similar result was reported by Welzbacher et al. (2008) in densified Norway spruce boards. However, they considered "vessel spring-back" instead of TC. They found some spring-back due to OHT, which might have been caused by the extension and evaporation of cell-wall bonded water and the relaxation of internal compression tension. The negative values measured in the present study could be caused by two mechanisms: 1) thermal degradation of cell wall components, such as hemicelluloses, as it is well known that heat treatment can cause weight loss (Bhuiyan et al. 2000; Johansson 2008; Korkut and Hiziroglu 2009; Obataya et al. 2006); or 2) the thicknesses used for calculation were measured in an air-dry condition. It was found in this study that in the same air-dry condition the equilibrium moisture content was reduced after OHT due to reduced wood hygroscopicity. Therefore, the wood veneers shrank after OHT due to moisture content loss.

Except for specimens that were oil-heat treated for 1 hour, OHT temperature also markedly impacted TC (Fig. 1). With the same densification temperature, the higher the applied OHT temperature, the lower the TC. This indicates that higher OHT temperature caused increased thermal degradation of cell wall components and greater reduction in wood hygroscopicity.



Fig. 1 Thickness change in densified veneers due to oil-heat treatment at different OHT temperatures: 180°C (diamonds), 200°C (squares), and 220°C (circles)

Compression Set Recovery

Figure 2 shows the variation in veneer thickness during the cyclic recovery test. Thickness increased greatly (from D0 to W1) in control densified veneers (without OHT) after initial water soaking, particularly at low temperatures. This was caused by reversible and irreversible swelling, indicated by the difference between W1 and D1 and between D0 and D1, respectively. After OHT, these differences were reduced. Furthermore, these differences decreased with increasing OHT temperature and duration, indicating improved veneer dimensional stability.

Compression recovery over time following moisture adsorption, also known as the "shape memory effect" (Navi and Heger 2004), is often a problem with densified wood. Therefore, compression set recovery was calculated (Eq. 2, Fig. 3) from the thickness variation in oven-dry condition (D in Fig. 2) during the cyclic recovery test and the initial thickness in oven-dry condition before densification. High compression set recovery was found in control densified veneers (without OHT), particularly for densification at low temperatures. Compression set recovery was strongly reduced in OHT densified veneers. Both OHT temperature and duration markedly influenced compression set recovery: higher OHT temperature and longer duration resulted in lower recovery. Compression set recovery was reduced to less than 5% under various OHT conditions (Fig. 3). Welzbacher et al. (2008) reported that compression set recovery in thermo-mechanically densified Norway spruce was reduced to less than 5% using an oilheat treatment at 220°C for 2h or 4h, and Tang et al. (2004) obtained less than 5% recovery in compressed Chinese fir using heat treatment at above 200°C for more than 5h. In the present study, almost 0% recovery was achieved under some OHT conditions: OHT at 180°C for 3h on specimens densified at 200°C; OHT at 200°C for 3h, and OHT at 220°C. Incidentally, some compression set recovery values were less than 0%. A similar result was found by Ito et al. (1998). They speculated that part of the hemicelluloses became water-soluble and were extracted from the specimen, causing the wood to shrink.

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Fig. 2. Thickness variation due to water absorption and oven-drying for veneers densified at 160°C (diamonds), 180°C (circles), and 200°C (squares), respectively, with OHT at 180°C, 200°C, and 220°C for 1h, 2h, and 3h, respectively, and for controls (without OHT). D, W, and B stand for oven-drying, wetting, and boiling, respectively

378



Fig. 3. Compression set recovery for veneers densified at 160°C (diamonds), 180°C (circles), and 200°C (squares), respectively, with OHT at 180°C, 200°C, and 220°C for 1h, 2h, and 3h, respectively, and for controls (without OHT). D stands for oven-dry

In order to prevent compression set recovery of densified wood after water soaking, Norimoto et al. (1993) considered three mechanisms: 1) formation of cross-linkages between molecules of the matrix constituents to prevent the relative displacement of microfibrils; 2) relaxation of the stresses stored in the microfibrils and matrix; and 3) treatment to isolate the hydrophilic cell wall constituents, especially the hemicelluloses, from moisture to prevent their re-softening. In OHT, the second and third mechanisms might dominate. The heat treatment process is generally accompanied by breakdown of the lignin–polysaccharide complex by organic acids released from hemicelluloses (Korkut and Hiziroglu 2009; Zaman et al. 2000). Therefore, the stress stored in the microfibrils and matrix was relaxed to some extent. The argument for the degradation of hydrophilic cell wall constituents (especially the hemicelluloses) by OHT is supported by the reduced wood hygroscopicity and wood swelling (Fig. 4, 5, and 6). These results indicate that OHT was efficient in reducing the compression set recovery of densified veneers.

Swelling, Irreversible Swelling, and Anti-Swelling Efficiency

Figure 4 shows the swelling from oven-dry to moisture-saturated condition in the compression direction (radial, R) in control (non-densified), control densified (without OHT), and densified veneers with OHT at different temperatures and durations. Very high swelling, from 30% to 75%, was found in densified veneers without OHT. After OHT, swelling decreased dramatically. Furthermore, OHT temperature had a marked impact on swelling: the higher the OHT temperature, the lower the swelling. Unlike OHT temperature, OHT duration had no clear impact on swelling. The swelling of densified veneer was composed of reversible and irreversible swelling, where reversible swelling was due to wood hydroscopicity and irreversible swelling was due to compression set recovery. However, although approximately 0% compression set recovery was obtained after OHT, the radial swelling of OHT veneers remained higher than that of the non-densified controls (Fig. 4). This can be largely explained by swelling in the compression (radial) direction, calculated as follows:

Swelling =
$$\frac{(T_{CWW} + D_{LRW}) - (T_{CWD} + D_{LRD})}{T_{CWD} + D_{LRD}} \times 100\%$$
 (6)

where T_{CWW} and T_{CWD} are the thickness of the cell wall in moisture-saturated and ovendry condition, respectively; and D_{LRW} and D_{LRD} are the diameter of the lumen in the radial direction in moisture-saturated and oven-dry condition, respectively. It has been reported that lumen size varies only slightly in normal wood cells during drying, compared to macroscopic shrinkage (Fang et al. 2007; Kelsey 1963; Keylwerth 1951). Stable lumen size would also be assumed during moisture absorption. This can be expressed as:

$$D_{LRW} \approx D_{LRD}$$
 (7)

Therefore, radial swelling can be simplified as:

Swelling
$$\approx \frac{T_{CWW} - T_{CWD}}{T_{CWD} + D_{LRD}} \times 100\%$$
 (8)

The lumen diameter in the radial direction of densified wood was much less than that of non-densified wood. Therefore, despite 0% compression set recovery, the swelling of OHT densified wood in the compression direction was greater than that of non-densified wood. Unlike R swelling, tangential (T) swelling decreased after densification (Fig. 5): the higher the densification temperature, the less the T swelling. Furthermore, OHT temperature had a significant impact on T swelling. T swelling values near 4% were obtained on densified veneers after OHT at 220°C. The reduced T swelling might be caused by the degradation of hydrophilic cell wall constituents, especially the hemicelluloses, during densification and OHT. Similar results were found for longitudinal (L) swelling (Fig. 6). The L swelling of densified veneer was almost 0%. However, because the L swelling of wood is very low, the impact of OHT temperature on L swelling was not clear. The effect of OHT duration on both T and L swelling was not significant.



Fig. 4. Radial (R) swelling of densified veneers with and without OHT and non-densified veneers. Circles: without OHT; diamonds: OHT for 1h; squares: OHT for 2h; and triangles: OHT for 3h

Irreversible swelling (*IS*) often occurs in densified wood in the compression direction after the first water sorption cycle due to compression recovery (Gabrielli and Kamke 2010). Irreversible swelling in the compression direction of densified veneers with and without OHT is presented in Fig. 7. Very high *IS* was found in densified veneers without OHT, especially for veneers densified at low temperature. *IS* decreased after OHT, especially with high-temperature and long-duration OHT. High OHT temperature resulted in a marked reduction in *IS*. Increased OHT duration also caused low *IS*, particularly for veneers that were oil-heat treated for 3h. A comparable reduction in *IS* by impregnation of phenol formaldehyde was reported by Gabrielli et al. (2010) in viscoelastic thermal compression processed poplar.



Fig. 5. Tangential (T) swelling of densified veneers with and without OHT and non-densified veneers. Circles: without OHT; diamonds: OHT for 1h; squares: OHT for 2h; and triangles: OHT for 3h



Fig. 6. Longitudinal (L) swelling of densified veneers with and without OHT and non-densified veneers. Circles: without OHT; diamonds: OHT for 1h; squares: OHT for 2h; and triangles: OHT for 3h



Fig. 7. Irreversible swelling (*IS*) of densified veneers with and without OHT. Circles: without OHT; diamonds: OHT for 1h; squares: OHT for 2h; and triangles: OHT for 3h



Fig. 8. Anti-swelling efficiency (*ASE*) of densified veneers with and without OHT. Circles: without OHT; diamonds: OHT for 1h; squares: OHT for 2h; and triangles: OHT for 3h

Figure 8 presents the *ASE* values calculated based on thickness swelling in the compression direction of non-densified veneers. All values are negative, because the R swelling values for densified veneers were all higher than those for non-densified veneers. *ASE* improved significantly after OHT. OHT temperature showed a pronounced positive effect on *ASE*. The effect of OHT duration on *ASE* did not show a clear trend.

CONCLUSIONS

Oil-hot treatment (OHT) was shown to be efficient for improving the dimensional stability of thermo-hygro-mechanically densified veneers. After OHT, a marked reduction in compression set recovery was found in densified veneers. Both OHT temperature and duration had a marked influence on the reduction of compression set recovery. The higher the OHT temperature and duration, the lower the recovery. Recovery was reduced to less than 5% under various OHT conditions. Recovery values of about 0% were achieved under some OHT conditions: OHT at 180°C for 3h for specimens densified at 200°C; OHT at 200°C for 3h; and OHT at 220°C. Both R and T swelling in densified veneers were reduced dramatically. Compared to OHT duration, OHT temperature had a more pronounced impact on R and T swelling. Irreversible shrinkage in the compression direction in densified veneers decreased after OHT, particularly with high-temperature and long-duration OHT. Anti-swelling efficiency in the compression direction improved significantly after OHT, and oil-heat treatment temperature showed a pronounced positive effect on ASE.

Due to their dimensional stability and high density, densified wood veneers treated by the oil-heat treatment process show good potential for appearance products. They could be used as the face ply of laminated composites. Further work is required to assess their mechanical and gluing properties.

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