VTC Treatment and Phenol-formaldehyde Impregnation of Hybrid Poplar

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ABSTRACT

The viscoelastic thermal compression (VTC) method for wood densification was used on hybrid poplar (*Populus sp.*). An additional treatment with a dilute aqueous solution of low molecular weight phenol-formaldehyde was integrated with the VTC process. The resulting specimens were evaluated by water soaking and boiling to determine resistance to swelling. Anti-swelling efficiency was calculated. Dimensional stability was improved in all cases. The higher molecular weight PF resin performed the best due to a higher retention of polymer during the VTC process.

INTRODUCTION

One approach for modifying wood properties is by mechanical compression perpendicular to the grain. This approach increases the density and typically improves stiffness, strength, and hardness. Elevated temperature and steam are usually applied prior to densification to soften the wood. After compression, extended thermal treatment may be used to improve resistance to water adsorption. Viscoelastic Thermal Compression (VTC) is one method of increasing the density of wood by means of mechanical compression perpendicular to the grain under conditions of dynamic temperature and steam pressure (Kamke 2006, Kamke and Sizemore 2008). Although the steam treatment and high processing temperature do render the cell wall less hygroscopic, the increased density increases swelling potential upon exposure to water. An extended heat treatment will reduce the hygroscopic nature of wood, but the thermal degradation that occurs also significantly reduces strength. The purpose of this research was to evaluate the effectiveness of coupling phenol-formaldehyde impregnation with the VTC process to produce a highly stable material with improved mechanical properties.

MATERIALS AND METHODS

Hybrid poplar, a cross between eastern and black cottonwood (*Populus deltoides and Populus trichocarpa*) and widely cultivated in North America on intensively managed plantations, were used. Specimen dimensions were 170 mm x 30 mm x 6 mm (longitudinal x tangential x radial direction). Density ranged from 0.34-0.40 g/cm^3 at 12% moisture content. Specimens were machined so that densification occurred in the radial direction.

Phenolic resins used for this study were commercial products developed for paper impregnation and insulation products (to be referred to as PF1 and PF2). PF1 had a resin solids content of 39% and a weight average molecular weight of 780. PF2 had a resin solids content of 57% and a weight average molecular weight of 172.

Specimens were pressure-impregnated using 3 levels of an aqueous PF solution (5, 10, or 20% PF) for each of the two resins. Once submerged, specimens were subjected to 96 kPa vacuum for 15 minutes followed by 620 kPa pressure for 30 minutes. Specimens were removed from their treating solution after the vacuum-pressure impregnation and immediately processed in the VTC device. Control specimens were not treated with PF and were VTC processed with an initial moisture content of 12%.

PF-treated specimens were first subjected to a 3 minute drying step using 150 °C platens in the VTC device to drive off excess water. Specimens were then densified using the standard VTC schedule developed by O'Conner (2007). Control specimens were processed using the standard VTC processing schedule. The standard VTC schedule involved placing a specimen between heated platens and exposing it to 860 kPa saturated steam for 3 minutes prior to compression; mechanical compression at 1400 kPa was then performed for 2 minutes; steam pressure was released to atmospheric pressure; the press was then opened for 2 minutes before platen temperature was elevated to 190 °C and final compression to target thickness of 2.5 mm was performed for 5 minutes; the specimen was then cooled below 100 °C before the platens were opened. Once densification was complete, specimens were oven-dried at 103 °C for 24 hours. From the oven-dry state specimens were subjected to a room temperature 24-hour water soak, then oven-dried, and then subjected to an additional 2-hour boil in water. Mass and volumetric measurements were recorded before and after testing to determine thickness swell (TS) and anti-swelling efficiency (ASE). Specimens were then oven-dried from their swollen state for 24-hours and mass and volumetric measurements were taken to calculate irreversible swelling (IS). TS was calculated using the average of three thickness measurements and represents the amount of swelling in the wet condition as a percent of the initial oven-dried thickness. ASE and IS values were determined using the following calculations:

$$S = \frac{V_1 - V_0}{V_0} \times 100 \quad , \qquad ASE = \frac{S_0 - S_1}{S_0} \times 100 \quad , \qquad IS = \left(\frac{V_{OD1} - V_{OD0}}{V_S - V_{OD0}} \right) \times 100$$

Where S = volumetric swelling coefficient, V_1 = volume after water test, V_0 = oven-dry volume after VTC processing, ASE = anti-swelling efficiency, S_1 = volumetric swelling coefficient of chemically treated specimen, S_0 = volumetric swelling coefficient of control specimen, IS = irreversible swelling, V_S = swollen volume after water test, V_{OD0} = oven-dry volume after VTC processing, and V_{OD1} = oven-dry volume after water test.

RESULTS AND DISCUSSION

Both PF resins at all concentrations were able to significantly reduce thickness swell compared to the unmodified VTC processed control (Table 1). At 20% concentration, average thickness swell of PF1 treated specimens was more than 7 times lower in comparison to the control specimens. Thickness swell decreased as resin concentration increased for both resins tested.

ASE values, which represent the amount of swelling that PF treatment prevents when compared to the swelling of a control specimen, show that both high and low molecular

weight resins were able to impart a large degree of stability. An average ASE value of 86% was achieved for specimens treated with PF1 at 20% concentration. As resin concentration increased so too did dimensional stability. ASE values were normalized by their weight percent gain (WPG) to determine the amount of dimensional stability imparted per unit of resin retained in the specimen (ASE_N). This value provides insight into the effectiveness of the resins at each concentration level. As seen in Table 1, at 5% resin concentration both PF1 and PF2 had significantly higher ASE_N values than at higher concentration levels indicating that, as resin concentration increased, diminishing returns on dimensional stability were realized.

Table 1: Dimensional stability properties for specimens treated with various concentrations of high (PF1) and low (PF2) molecular weight phenol-formaldehyde resins. Values represent the average of 10 replicates with standard deviation in parentheses.

Treatment	Conc. [%]	WPG [%]	WPG_E [%]	TS [%]	ASE [%]	ASE_N [%]	IS [%]
PF1	5	3.6 (0.7)	9.1 (0.9)	21.8 (4.3)	70.6 (5.4)	20.3 (3.6)	2.1 (4.4)
	10	11.3 (2.2)	17.2 (2.8)	19.0 (4.1)	74.7 (5.0)	6.8 (1.2)	1.5 (3.0)
	20	23.0 (2.6)	32.6 (4.6)	9.2 (4.8)	86.1 (6.5)	3.8 (0.6)	2.4 (7.7)
PF2	5	4.1 (1.4)	17.1 (2.8)	39.7 (5.6)	50.3 (6.6)	13.8 (6.0)	20.6 (10.2)
	10	5.8 (1.2)	16.2 (1.8)	33.1 (5.0)	56.8 (5.4)	10.1 (2.0)	9.3 (6.0)
	20	16.5 (2.1)	32.9 (2.8)	25.8 (3.5)	66.5 (4.3)	4.1 (0.5)	7.5 (6.7)
Control	0			76.2 (11.5)			20.3 (10.8)

Irreversible swelling measures the release of internal stresses which often build up during manufacturing of highly densified wood products. PF resin has been claimed to plasticize the cell wall (Shams *et al*. 2004) and reduce internal stresses during densification. Consequently, low IS values were expected with PF impregnated VTC specimens, as was a trend of decreasing IS with increasing resin concentration. PF1 treated specimens showed no trend as concentration increased, however, IS values were extremely low with a maximum value of only 2.4%. Specimens treated with PF2 had decreasing IS values as concentration increased. At 5% concentration, IS was 20.6% and was reduced to 7.5% with specimens treated at 20% concentration. IS for the unmodified control was 20.3%.

Previous literature has shown low molecular weight PF resins to be superior for instilling dimensional stability (Deka *et al*. 2000, Wan *et al*. 2006, Shams *et al*. 2004, Ryu et al. 1993). Lower molecular weight resins more easily penetrate the wood cell wall and ultimately bulk the wood material to a greater extent, thus providing greater stability (Rowell 1999). Results for this study were dissimilar to previous research. Specimens treated with PF1, the higher molecular weight resin, exhibited greater dimensional stability over specimens treated with PF2. WPG provides insight to the degree at which the two resins were able to penetrate the wood's cellular structure. Higher WPG indicate greater penetration. Exclusive of the 5% concentration level in which WPG for PF1 and PF2 were statistically similar, all other concentration levels resulted in significantly higher WPG for PF1, implying PF1 was better able to swell the wood. However, after chemical modification, but prior to VTC processing, specimens were weighed. Using this value along with solution concentration, estimated weight percent gain (WPG_E) was calculated to determine if differences in WPG were an artefact of VTC processing. At each concentration level, except for 5%, PF1 and PF2 had similar WPG_E values. This implied that both resins were equally able to penetrate the wood cell wall, but the VTC process caused more resin loss for PF2 than PF1.

It was of interest to know if PF1 had greater stability values simply because more of it was present in the specimens (i.e. it had a higher WPG), or if some intrinsic property inherent to PF1 made it better at imparting dimensional stability. ASE values were normalized by their WPG to determine the stability imparted per unit of resin retained in each specimen. At 20% concentration ASE_N values were statistically similar between resin types and at 5% concentration PF1 was greater, while at 10% PF2 was greater. No clear trend was observed and it appears that the greater stability achieved with PF1 treated specimens was a result of the higher WPG associated with the resin.

CONCLUSIONS

Phenol-formaldehyde resin impregnation was successfully coupled with the viscoelastic thermal compression process to produce a dimensional stable product. It was found that the higher molecular weight PF resin was retained in VTC processed specimens to a greater extent, and as a result, provided greater overall stabilization due to higher resin loading levels. Dimensional stability was positively correlated with resin concentration, but was found to be most efficient at increasing stability on a unit of resin basis at lower concentration levels.

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