

An Optimal Thermo-Hydro-Mechanical Densification (THM) Process for Densifying Balsam Fir Wood

Ling Li,^{a,b} Meng Gong,^{b,*} Naxin Yuan,^c and Dagang Li^a

To better utilize low-density softwood, a thermo-hydro-mechanical densification process performed in an open system was studied to enable the manufacture of densified wood with a hard surface, strong bonding, and good dimensional stability. This study was aimed at optimizing three densification parameters, *i.e.*, compression ratio (CR), temperature, and time, for balsam fir (*Abies balsamea* (L.) Mill.). The Brinell surface hardness, bond strength, and thickness recovery ratio of densified fir were examined. It was found that the optimal densification parameters were a CR of 60%, a temperature of 230 °C, and a time of 20 minutes. The surface hardness and bond strength of optimized densified fir were about 30 and 8 MPa, respectively. The thickness recovery ratio of the densified fir after a 2-hour cold water soaking and another 2-hour boiling treatment was about 10%. Because the densified fir in this study was used for indoor applications only, its thickness recovery ratio could be minimal under conditions of use.

Keywords: Bond strength; Optimal thermo-hydro-mechanical densification; Thickness recovery; Softwood; Surface hardness

Contact information: a: College of Wood Science and Technology, Nanjing Forestry University, Nanjing, Jiangsu, 210037, China; b: Faculty of Forestry and Environmental Management, University of New Brunswick, P.O. Box 4400, 28 Dineen Drive, Fredericton, NB, E3B 5A3, Canada; c: College of Forestry, South China Agricultural University, Guangzhou, Guangdong, 510642, China;

* Corresponding author: meng.gong@unb.ca

INTRODUCTION

Balsam fir (*Abies balsamea* (L.) Mill.) is a very common softwood species that, on a national level, accounts for nearly 12% of the total Canadian forest inventory. The largest proportion of total growing stock is located in southeastern Canada, especially in the Maritime Provinces, where it is considered a valuable commercial species. Because balsam fir wood is light in weight (*e.g.*, density ranging from 0.300 to 0.350 g/cm³), limber, soft, and low in decay resistance, it is not a major contributor as dimension lumber in the category of Spruce-Pine-Fir (SPF), in which it only comprises 16% of the total amount of SPF (Forintek and AFRI 2006). To expand its miscellaneous utilizations, an effective approach is to improve the mechanical and physical properties of balsam fir wood.

Most of the mechanical properties of wood are positively correlated with its density, *e.g.*, the higher the wood density is, the larger the stiffness and strength of wood (Wantanabe *et al.* 1999; FPL 2010). Due to these properties, increasing wood density can improve the mechanical properties of fast-grown, low-density softwood. Although both polymer impregnation and mechanical densification techniques can achieve this goal, the

mechanical densification technique is accepted and used because it neither causes concerns to one's physical health nor creates environmental issues.

The mechanical densification technique is briefly described as compressing wood in the radial direction with the aid of temperature and moisture until the thickness of the wood decreases to a desired value. The ratio of the decrease in thickness to the initial thickness of wood is defined as the compression ratio (CR). The density as well as the majority of mechanical properties of densified wood increase with increasing CR.

During the densification process, the mechanical behavior of cell walls undergoes three stages: 1) initial linear elastic buckling/bending, reflected in the short linear elastic region of a stress-strain curve; 2) long-term, non-linear cellular collapse at a nearly constant stress level, *i.e.*, the strain quickly increases but the stress stays almost constant; and 3) a sharp increase of stress with strain after a majority of cellular collapse (Kamke and Kutnar 2010). The linear elastic strain in stage 1) is temporarily 'stored' in densified wood and will be instantaneously released at the moment of unloading the external stress. It is thereby named 'springback' (Halligan 1970). In stages 2) and 3), viscous-elastic strain and plastic strain occur as the stress exceeds the proportional limit point of the stress-strain curve. The viscous-elastic strain can be gradually reversed with time, but the plastic strain is irreversible and permanently fixed in densified wood (Kamke and Kutnar 2010). Therefore, a big challenge for using the mechanical densification technique is to eliminate, to a maximum degree, the thickness recovery of densified wood due to the release of elastic and viscous-elastic strains in order to maintain its dimensional stability during a long-term service life.

The chemical mechanism behind the mechanical densification technique is that wood can be densified because of the rheological characteristics of wood under thermohydrous conditions (FPL 2010; Navi and Sandberg 2012). Wood can transition from a rigid state to a thermoplastic flow state at a given temperature, which is defined as the glass transition temperature (T_g). Above the T_g , wood displays a thermoplastic flow (*i.e.*, rubbery) state and can be reshaped by applying an external load. Its new shape would be fixed by reducing the temperature to a value below the T_g under pressure. Navi and Sandberg (2012) summarized the different T_g values of the three main components of wood (*i.e.*, cellulose, hemicellulose, and lignin) based on the studies done by Salmén (1982 and 1984), Back and Salmén (1982), and Irvine (1984): T_g values of anhydrous cellulose, hemicelluloses, and lignin varied from 200 to 250 °C, 150 to 220 °C, and 140 to 190 °C, respectively, depending on chemical components, moisture content (MC), measuring method, and criteria for determining T_g . Meanwhile, the T_g of each wood component can decrease to some degree with increasing MC. Salmén (1982) found that a decrease in the T_g with increasing MC for isolated hemicelluloses and semi-crystalline cellulose followed a negatively near-linear pattern. The T_g of hemicelluloses decreased much quicker than that of semi-crystalline cellulose at a given range of MC. As MC increased from 0 to 30%, the T_g values of hemicelluloses and semi-crystalline cellulose decreased from above 200 °C to 0 °C and 80 °C, respectively. Comparatively, the T_g of isolated lignin decreased from 200 to 85 °C as MC increased from 0 to 15% and then kept this value constant as the MC increased to the maximum value. Navi and Sandberg (2012) explained that this was due to the adsorbed water/moisture, placed between molecules by hydroxyl (-OH) groups, which increased the average inter-molecular distance and the mobility of the molecules and thereby decreased the inter- and intra-molecular interactions of the hydrogen bonds in amorphous polymers.

The T_g of lignin was used to determine the low temperature threshold for densifying wood under a saturated water state, which was considered to be at least $T_g + 25$ °C (Navi and Sandberg 2012). Some pre-treatment methods, such as soaking wood in water and steaming wood in a saturated steam state, were hence applied before and/or over the course of mechanical densification to soften wood (Rowell and Konkol 1987; Inoue *et al.* 1993; Ito *et al.* 1998a,b; Navi and Giradet 2000; Inoue *et al.* 2008; Kutnar *et al.* 2008; Fang *et al.* 2012; Li *et al.* 2012). Fukuta *et al.* (2007) found that the external stress to compress well-soaked Japanese cedar (*Cryptomeria japonica* D. Don) decreased with increasing temperature from 140 to 190 °C. Properly increasing temperature above the T_g reduces the external stress applied and thereby the residual stress ‘stored’ in densified wood. Thus, the amount of ‘springback’ would be decreased.

However, as the temperature increases to above 150 °C, the hydrolysis of hemicelluloses accompanied by the brittleness of wood fibers may occur, which can restrain a part of the thickness recovery of densified wood but decrease some mechanical properties (*e.g.*, stiffness and strength), depending on how high the temperature is and how long it is applied (Giebeler 1983; Fengel and Wegener 1989; Kocafe *et al.* 2010; Navi and Sandberg 2012; Rautkari *et al.* 2013).

The thickness recovery of densified wood can be almost eliminated by carrying out various post-treatment methods in the temperature range of 180 to 240 °C, which are mainly classified in two categories: 1) saturated steam post-treatment with/without pressure in a closed system; and 2) heat post-treatment *via* air as medium with/without pressure in an open system (Fukuta *et al.* 2007; Inoue *et al.* 2008; Gong *et al.* 2010; Fang *et al.* 2012). A study done by Fukuta *et al.* (2007) revealed that the time needed for steam treatment conducted in a closed system was much less than that for heat treatment performed in an open system. Numerous studies have confirmed that the final mechanical properties of densified wood subjected to various post-treatments are still significantly improved in comparison to undensified wood because the decrease due to the high temperature was less than the increase caused by the mechanical densification process (Navi and Girader 2000; Lamason and Gong 2007; Kutnar *et al.* 2008; Korkut and Hiziroglu 2009; Gong *et al.* 2010; Fang *et al.* 2012; Rautkari *et al.* 2013).

The above review and discussion suggests that CR, temperature, and time are three important factors for mechanically densifying wood. The CR determines the mechanical properties of densified wood. Both temperature and time govern the thickness recovery of the densified wood. This study attempted to design a thermo-hydro-mechanical (THM) densification process to efficiently form densified balsam fir with the purpose of reducing manufacturing cost and removing dimensional limitations on wood materials. Three densification parameters, *i.e.*, CR, temperature, and time, were considered in the design of the THM densification process. The surface hardness, bond strength, and thickness recovery ratio of densified fir were the three factors used for evaluation since the target application of the densified wood in this study was the surface layer of an engineered wood flooring product.

MATERIALS AND METHODS

Materials

The wood species used was balsam fir (*Abies balsamea* (L.) Mill.). The mean and standard deviation (SD) values of its oven-dried density were 0.320 and 0.010 g/cm³, respectively. Clear and flat-sawn wood specimens were prepared. The dimensions of the wood specimens were 280 mm in length (longitudinal direction) by 60 mm in width (tangential direction) by 4, 5, or 8 mm in thickness (radial direction). The final thickness was set at 3 mm. Therefore, there were three nominal CRs of 25%, 42.5%, and 60%. Before densification, all specimens were placed in a conditioning chamber at a temperature of 20 °C and relative humidity (RH) of 65% until their MCs were stabilized at about 12%.

Thermo-Hydro-Mechanical (THM) Densification Process

The THM densification process was performed at the Wood Science and Technology Centre, the University of New Brunswick, Canada. A small hot press machine was used (Fig. 1), which was equipped with two temperature-controlled ‘hot’ platens with an area of 300 × 300 mm and a capacity of 12 tons. The hot press used in this study was an open-system with an aim at reducing the manufacturing cost.

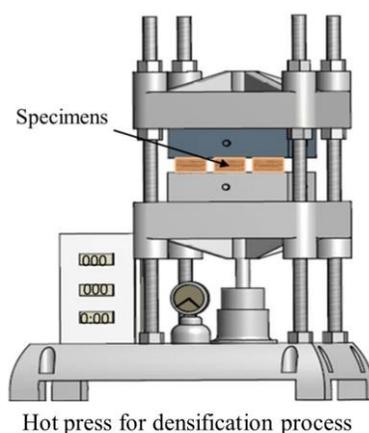


Fig. 1. Experimental set-up

The densification process included the three stages of pre-heat treatment, densification, and cooling. A batch of three pieces of wood specimens were first put into the hot press and heated by closing two ‘hot’ platens with very limited pressure until their inner temperature reached the target temperature. This temperature was determined by using a pair of thermocouples that were inserted into two pre-drilled holes in the core of specimens in the trial tests. The pre-heating time for 4-, 5-, and 8-mm-thick specimens were about 5, 8, and 12 minutes, respectively. Maintaining the same temperature, the press was subsequently closed at a rate of 1 ton/minute until the target thickness was reached. After keeping the pressure for various minutes, as given in Table 1, the whole system was then cooled down in 5 minutes via an attached cold water cooling system until the temperature in the core of each specimen was below 60 °C. At such a condition (temperature was lower than 60 °C and moisture content was lower than 12%), the T_g of wood was below the value required for glass-rubber transition, resulting in a fixed shape

of deformed specimens. It should be pointed out that the time for cooling to the target temperature in the core of each specimen was also measured by the pair of thermocouples in the trial tests. After densification and before property testing, all specimens were stored in a conditioning chamber at 20 °C and 65% RH until they reached constant weights.

Optimization of THM Densification Process

Experimental design of THM densification process using response surface methodology (RSM)

Response surface methodology (RSM) is a collection of mathematical and statistical techniques that is one of the main functions for the design of experiments in Minitab software. It is used to model and analyze problems in which a response of interest is influenced by several variables (Castillo 2007). The levels of the three variables of compression ratio (CR), temperature, and pressing time are given in Table 1. There were 15 experimental steps with 3 replicates designed by RSM, generating a total of 45 densified wood specimens. After completing all tests, the relationships of the response with several variables were displayed in the form of a response surface plot based on the calculated results using a regression model. Meanwhile, the analysis of variance (ANOVA) was subsequently conducted by Minitab 16.0 software.

Table 1. Experimental Ranges of the Three Variables of the THM Densification Process at Three Levels

Variable	Level		
	Low	Mean	High
Compression ratio (CR, %)	25	42.5	60
Temperature (°C)	210	220	230
Pressing time (minute)	5	12.5	20

Evaluation of optimal THM densification parameters using a multi-response optimization method (MROM)

Optimal settings of the design variables for one response may be far from optimal or even physically impossible for another response (Castillo 2007). Therefore, the multi-response optimization method (MROM) is widely used, which can result in more reliable results. The responses used in this study were surface hardness, bond strength, and thickness recovery ratio. These three responses were deemed to be conflicting responses, as the surface hardness and bond strength were expected to have large values but the thickness recovery ratio was expected to be small. Thus, this method allowed for a compromise among these conflicting responses and provided an optimal solution and optimization plot for the combinations of the input variables using the response optimizer function provided in the Minitab software.

Property Evaluation

Brinell surface hardness

A Brinell hardness test was conducted using an Instron universal testing machine with a load cell of 10 kN based on EN 1534 (EN 2000). A 10-mm-diameter steel ball was used as an indenter. The maximum load of 1000 N was reached in 15 s, then kept for 25 s,

and then released over 15 s. There were six replicates for each type of specimen. The Brinell surface hardness (HB) was calculated using Eq. (1),

$$HB = \frac{F}{\pi Dh} \quad (1)$$

where, F is the load applied (N), D is the diameter of the indenter (mm), and h is the maximum depth of the indentation (mm).

Bond strength

A block shear test was used to examine the bond strength of densified wood specimens in the longitudinal and tangential plane according to ASTM D905-08e1 (ASTM 2012). The dimensions of each specimen were scaled down to 25 mm (length) by 25 mm (width) by 15 mm (thickness) due to the restriction of the densification process. The 15-mm-thick wood specimen was made of 3-mm-thick densified wood and 12-mm-thick undensified wood. There were six replicates for each type of specimen. Before bonding, the surface of each specimen was slightly sanded by using a piece of 100-grit sandpaper. A commercial one-component polyurethane (PUR) adhesive was used to glue two wood specimens together. Based on the instructions given by the supplier, the viscosity of the PUR adhesive was 8000 mPa·s. The coat weight was 160 g/m². The minimum pressure for two paired wood specimens was 0.60 N/mm², and the pressing time was about 1 h. The curing time was about 2 h. All bonded specimens were placed in a conditioning chamber at 20 °C and 65% RH for 2 weeks to release the internal stress caused during bonding.

An Instron universal testing machine with a 10-kN load cell was used to conduct the block shear test. A compressive load was increased at the speed of 1 kN/minute to ensure a specimen to fail within 90 s. After failure, the percentage of wood failure was visually examined. The bond strength (BS) was calculated using Eq. (2),

$$BS = \frac{F_{peak}}{wh} \quad (2)$$

where F_{peak} is the peak load at failure (N), w is the width (mm), and h is the length of the bond line (mm).

Thickness recovery ratio

To conduct a thickness recovery test, all densified wood specimens were first soaked in cold water until saturation for 2 h at atmospheric pressure, followed by 1 h of vacuum treatment at 85 KPa and a subsequent 1-h pressure treatment at 517 KPa. They were then placed in boiling water for another 2 h. A micrometer with 0.001-mm accuracy was used to measure the thicknesses of each specimen before and after treatment. There were six replicates for each type of specimen. To minimize the random measurement error, a cross was marked at the center of the two surfaces of each specimen before testing. The thickness recovery ratio (TR) of densified wood was calculated using Eq. (3),

$$TR(\%) = \frac{T_b - T_d}{T_i} \times 100 \quad (3)$$

where, T_b is the thickness of densified wood after boiling treatment (mm), T_d is the thickness of densified wood (mm), and T_i is the initial thickness of undensified wood (mm).

Verification of Optimized THM Densification Parameters

To verify the optimized densification parameters, six balsam fir specimens from the same batch were compressed according to the optimized results. Meanwhile, another six undensified wood specimens were prepared as a control group. The HB , BS , and TR of all densified and undensified wood specimens were measured following the aforementioned methods. In addition, these results were compared to those of undensified sugar maple (*Acer saccharum*).

RESULTS AND DISCUSSION

Effects of CR, Temperature, and Time on HB

Figure 2 displays an illustration of the response surface plots of HB versus two of three parameters (*i.e.*, CR and temperature, CR and time, and temperature and time), in which the third parameter was fixed at the medium level (*i.e.*, time at 12.5 min, temperature at 220 °C, and CR at 42.5%). As shown in Figs. 2a and 2b, HB significantly increased with increasing CR, but the effects of temperature and time on HB were not significant. However, the interaction effect of temperature and time on HB was significant, as seen in Fig. 2c. When the time was below 8 minutes, HB slightly increased with increasing temperature. However, as the time exceeded 8 min, HB decreased with increasing temperature. The maximum HB of about 22 MPa was at 210 °C and 20 min, while the minimum HB of about 14 MPa was at 230 °C and 20 min.

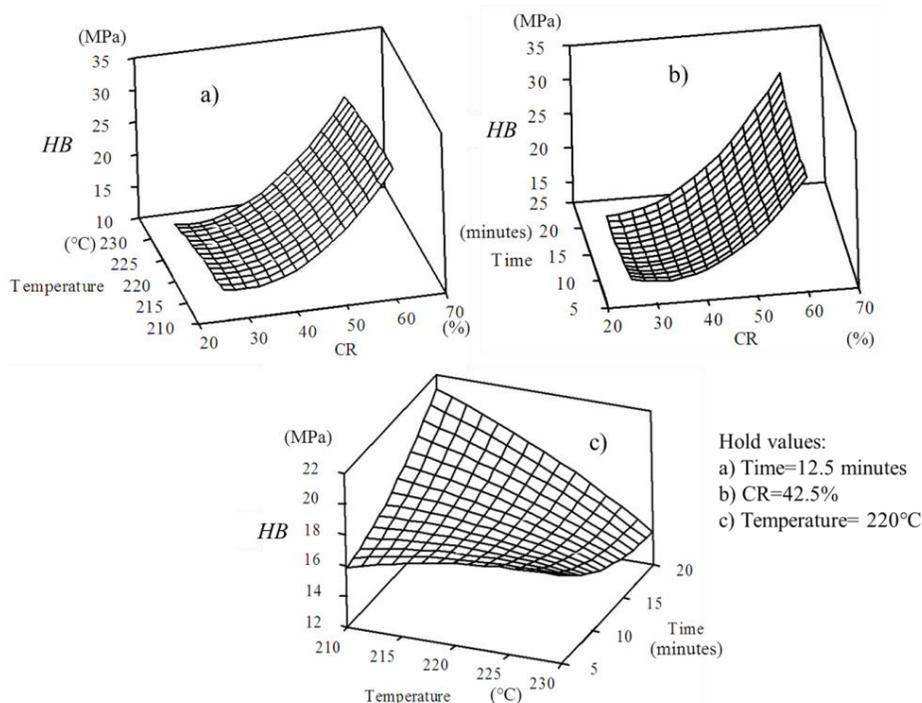


Fig. 2. Surface plots of HB versus CR, temperature, and time

The partial ANOVA results indicated that the main effect of CR and the interaction effect of temperature and time had statistical significances (p -values < 0.05) at a confidence level of 95%, as shown in Table 2. The reason that HB increased with increasing CR can be attributed to the increase of substance per unit volume due to the disappearance of cell lumens. As time increased, the phenomenon that HB decreased with increasing temperature could be explained by the material losses from thermal decomposition of lignin. Sergeeva and Miljutina (1960) found no change in lignin when temperature increased up to 155 °C, but heating at 175 °C caused lignin condensation that increased with temperature up to 240 °C. Similar findings by Fang *et al.* (2012) showed that the average HB of densified aspen (*Populus tremuloides*) veneer with 50% CR compressed at 160 °C was high, at 38 MPa, but that of densified veneer compressed at 220 °C decreased to 25 MPa.

Table 2. Partial ANOVA Results of HB vs. CR, Temperature, and Time

Source	DF	Seq. SS	Adj. SS	Adj. MS	F-value	p -value
CR	1	1536.48	1536.48	1536.48	119.17	0.000 *
Temperature	1	37.35	37.35	37.35	2.90	0.098
Time	1	4.11	4.11	4.11	0.32	0.576
CR x Temperature	1	0.53	0.53	0.53	0.04	0.841
CR x Time	1	28.14	28.14	28.14	2.18	0.149
Temperature x Time	1	56.75	56.75	56.75	4.40	0.043 *
Residual Error	35	451.26	451.26	12.89		
Total	44	2403.34				

Effects of CR, Temperature, and Time on BS

Three response surface plots of BS versus two of three parameters are shown in Figs. 3a, 3b, and 3c, in which the third parameter was fixed at the same medium level. The values of BS were in a small range of 7.0 and 10.0 MPa. In Fig. 3a and 3c, BS was the lowest when the temperature was 220 °C, regardless of CR and time. In Fig. 3b, BS differed with the changes of CR and time; however, there was not a consistent trend. Partial ANOVA results in Table 3 reveal that the main effects of the three parameters and their interaction effects on BS had no statistical significance (all p -values > 0.05) at a confidence level of 95%. Besides, the average wood failures of all densified fir specimens evaluated were in the range of 80 to 100%, indicating that the bonding ability of densified fir with the PUR adhesive was acceptable.

To evaluate the influence of the degradation of densified fir on BS , the surface inactivation of densified fir compressed at 60% CR and 230 °C for 20 min was examined in terms of initial contact angle (θ_0) and surface free energy (SFE) (Li *et al.* 2009). Their findings revealed that the θ_0 values of distilled water and formamide of the densified fir were about 85° and 70°, which were about 100% and 40% larger than those of undensified fir, respectively. The SFE of densified fir was 30.71 mJ/m², which was about 48% lower than that of the undensified fir. The BS of the densified fir compressed at the same condition in a verification test was 8.00 MPa, which was not the lowest value

shown in Fig. 3. Furthermore, the *BS* of the densified fir was about 30% larger than that of undensified fir. It could be concluded that there was no negative influence of degradation (*i.e.*, surface inactivation) of densified fir on *BS* by applying a proper surface sanding treatment before bonding.

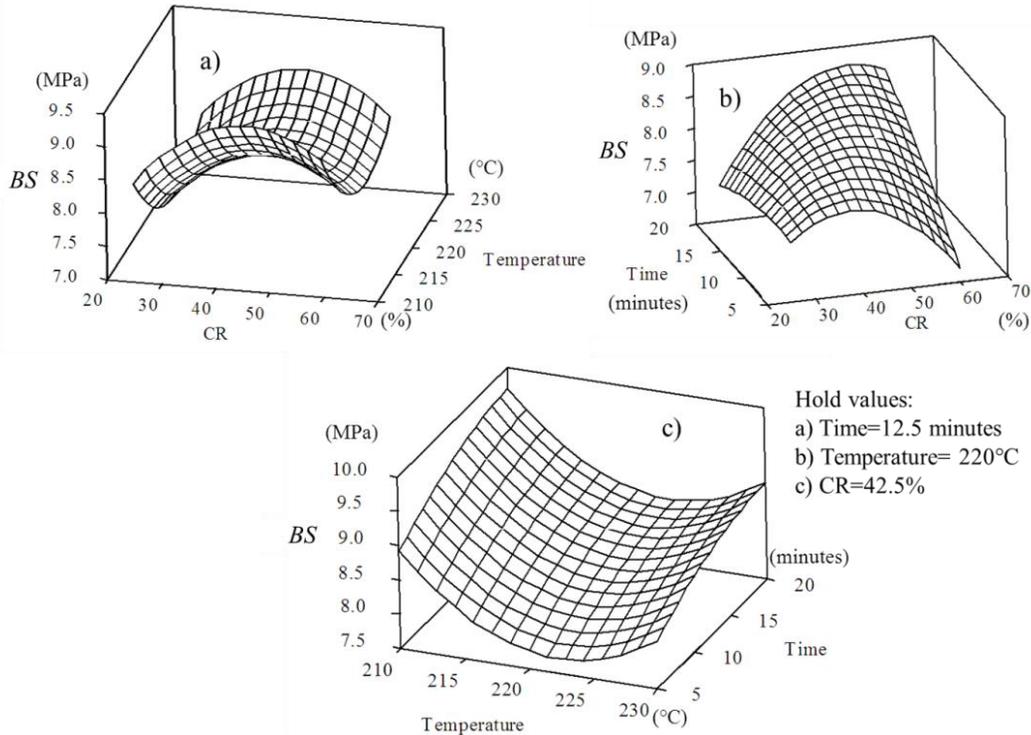


Fig. 3. Surface plots of *BS* versus CR, temperature, and time

Table 3. Partial ANOVA Results of *BS* vs. CR, Temperature, and Time

Source	DF	Seq. SS	Adj. SS	Adj. MS	F-value	<i>p</i> -value
CR	1	0.89	0.89	0.89	0.80	0.377
Temperature	1	3.38	3.38	3.38	3.06	0.089
Time	1	3.25	3.25	3.25	2.94	0.095
CR × Temperature	1	0.11	0.11	0.11	0.10	0.759
CR × Time	1	3.73	3.73	3.73	3.38	0.075
Temperature × Time	1	0.00	0.00	0.00	0.00	0.962
Residual Error	35	38.67	38.67	1.10		
Total	44	63.71				

Effects of CR, Temperature and Time on *TR*

Three response surface plots of *TR* versus two of three parameters are shown in Figs. 4a, 4b, and 4c, in which the third parameter was fixed at the medium level as well. Increasing temperature and/or time effectively decreased *TR* at any CR. The interactions of temperature and CR, CR and time, and temperature and time also strongly affected *TR*. In Figs. 4a and 4b, it can be seen that increasing temperature and time had a much

stronger influence on the reduction of TR of densified fir with a higher CR than that with a lower CR. In Fig. 4c, the largest TR (about 40%) was at 210 °C and 5 min, while the lowest TR (about 10%) occurred at 230 °C and 20 min.

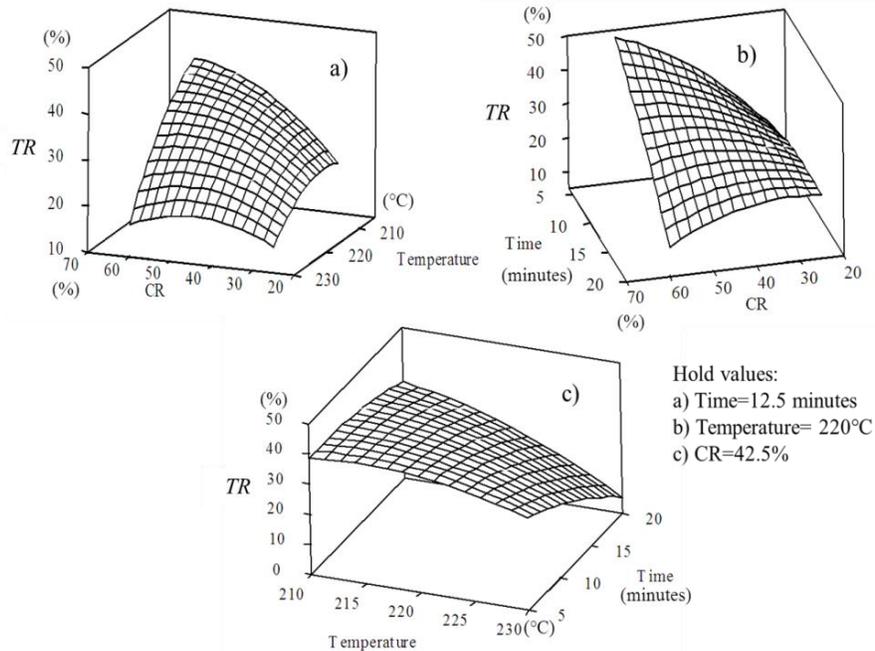


Fig. 4. Surface plots of TR versus CR, temperature, and time

The partial ANOVA results in Table 4 reveal that the three main effects of CR, temperature, and time and their interaction effects had statistical significances (all p -values < 0.05) at a confidence level of 95%. Similar results obtained by Fang *et al.* (2012) showed that the TR of densified aspen veneer with 50% CR was lowered from about 40 to 9% when the temperature was increased from 200 to 240 °C. The reason causing the decrease of TR of THM densified wood could be due to the degradation of hemi-cellulose exposed at a high temperature (>150°C) (Kocaefe *et al.* 2010; Navi and Sandberg 2012; Rautkari *et al.* 2013). With increasing temperature and/or time, the degree and amount of hemi-cellulose degradation increased, resulting in a lower TR of densified fir.

Table 4. Partial ANOVA Results of TR vs. CR, Temperature, and Time

Source	DF	Seq. SS	Adj. SS	Adj. MS	F-value	p -value
CR	1	623.56	623.56	623.56	27.12	0.000 *
Temperature	1	1745.69	1745.69	1745.69	75.93	0.000 *
Time	1	1549.84	1549.84	1549.84	67.41	0.000 *
CR × Temperature	1	236.83	236.83	236.83	10.30	0.003 *
CR × Time	1	1197.73	1197.73	1197.73	52.10	0.000 *
Temperature × Time	1	256.52	256.52	256.52	11.16	0.002 *
Residual Error	35	804.66	804.66	22.99		
Total	44	6782.36				

Optimization of THM Densification Parameters

The relationships between *HB*, *BS*, and *TR* and the three densification parameters of temperature, time, and CR were examined. MROM can provide various optimal solutions for the three design variables (*i.e.*, temperature, time, and CR) by choosing different weights for the three target responses (*i.e.*, *HB*, *BS*, and *TR*). In this study, the goal was to maximize the *HB* and *BS* of densified wood and minimize its *TR*. Therefore, the maximum values of *HB* and *BS* and the minimum value of *TR* in the RSM results were selected as the weights for the three target responses in MROM. Figure 5 shows the output window obtained when the Minitab response optimizer tool was used, in which the optimal conditions of CR, temperature, and time could be traced automatically. The optimized results of CR, temperature, and time are listed in the brackets in Fig. 5, showing that the values are 60%, 230 °C, and 20 min, respectively. Meanwhile, in Fig. 5, the predicted values of *HB*, *BS*, and *TR* based on the optimal conditions are shown in the parentheses, revealing that their predicted values are about 28.95 MPa, 8.88 MPa, and 7.98%, respectively.

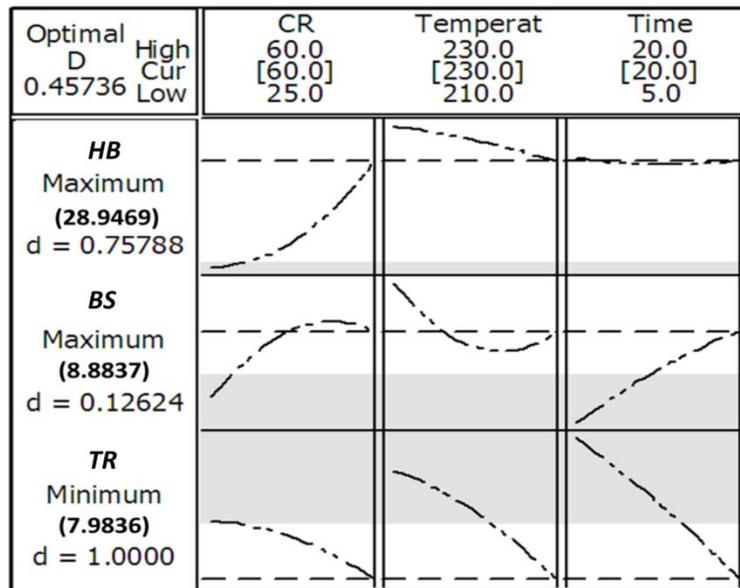


Fig. 5. Optimized THM densification parameters based on MROM

Verification of the Optimized THM Densification Parameters

Based on the optimized densification parameters, a group consisting of clear flat-sawn balsam fir of similar density was compressed. The *HB*, *BS*, and *TR* of both this group and undensified fir were measured. The experimental results of densified and undensified balsam fir, the predicted results of densified fir, and the corresponding results of sugar maple quoted in different references are all plotted in Fig. 6. The experimental results of densified fir were close to the predicted results. The experimental results of *HB* and *TR* of densified fir were about 33 MPa and 10%, which were about 8% and 20% larger than the predicted values, respectively, while that of *BS* was about 8 MPa, which was about 9% lower than the predicted value. Comparatively, the *HB*, *BS*, and *TR* values of densified fir were much higher than those of undensified fir and sugar maple. The *HB* of densified fir was about four and two times larger than that of the undensified balsam

fir and sugar maple, respectively. The *BS* of densified fir was about 30% and 60% higher than undensified balsam fir and sugar maple, respectively. It should be pointed out that although the *TR* of densified fir was about three and two times larger than the *TR*s of undensified balsam fir and sugar maple, respectively, and was caused by free swelling of the cell walls, this would not happen often during indoor applications due to the moderate moisture changes.

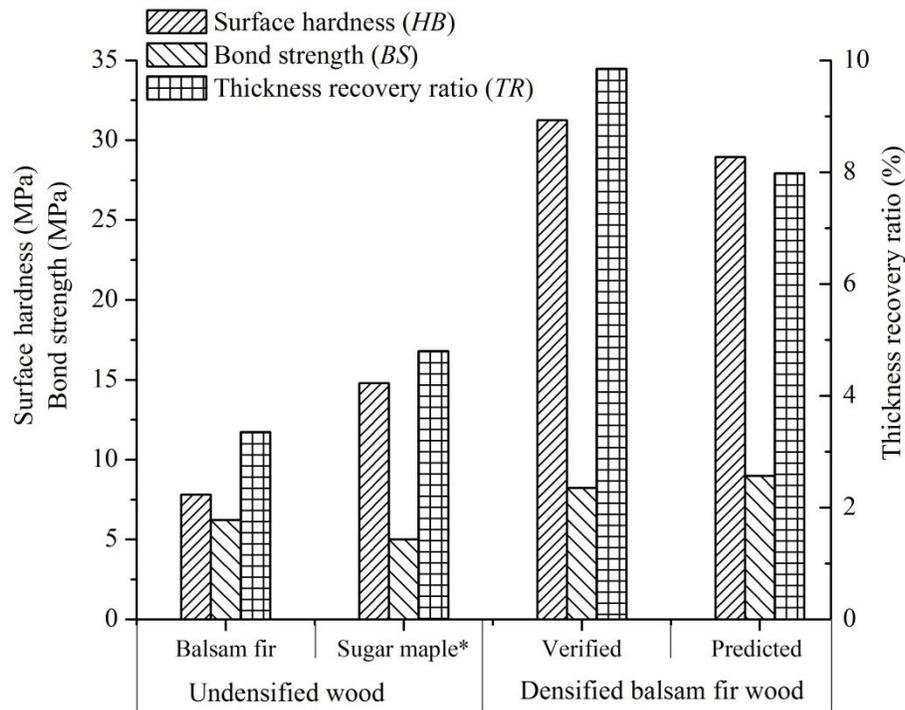


Fig. 6. Comparisons of *HB*, *BS*, and *TR* among undensified balsam fir, undensified sugar maple, and densified balsam fir (average values)(*Zhang *et al.* 2006; Blanchet *et al.* 2003; FPL 2010)

CONCLUSIONS

1. The optimal parameters were a CR of 60%, a temperature of 230 °C, and a pressing time of 20 min.
2. The experimental results for the *HB*, *BS*, and *TR* of densified balsam fir formed with the optimal parameters were about 30 MPa, 8 MPa, and 10%, respectively, very similar to the values predicted by the response surface method.
3. The *HB* and *BS* values of densified balsam fir were about four and two times, and 30% and 60%, larger, respectively, than those of undensified balsam fir and sugar maple.
4. The *TR* of densified fir was about three and two times larger than the corresponding values for undensified balsam fir and sugar maple, respectively.

ACKNOWLEDGEMENTS

This study was supported by the Doctorate Fellowship Foundation of Nanjing Forestry University, China, the Natural Sciences and Engineering Research Council (NSERC) of Canada (Grant #: RGPIN 311808-05), and the New Brunswick Innovation Foundation (NBIF) under its Research Assistantships Initiative Program and Emerging Project Program.

REFERENCES CITED

- American Society for Testing and Materials (ASTM). (2012). "ASTM D905-08e1: Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading," *Annual Book of Standards*, 15.06.
- Back, E. L., and Salmén, N. L. (1982). "Glass transitions of wood components hold implications for molding and pulping processes," *Tappi. J.* 65(7), 107-110.
- Blanchet, P., Beauregard, R., Erb, A., and Lefebvre, M. (2003). "Comparative study of four adhesives used as binder in engineered wood parquet flooring," *Forest Prod. J.* 53(1), 89-93.
- Canada's Wood products research institute (Forintek Canada Corp.) and Alberta Forestry Research Institute (AFRI). (2006). http://albertacanada.com/files/albertacanada/AIS-BP_BalsamFur.pdf
- Castillo, D. E. (2007). *Process Optimization: A Statistical Approach*, Springer, Berlin.
- European Standard (EN). (2000). "EN 1534:2000 E. Wood and parquet flooring - determination of resistance to indentation (Brinell) - test method," *European Standard*, CEN.
- Fang, C. H., Mariotti, N., Cloutier, A., Koubaa, A., and Blanchet, P. (2012). "Densification of wood veneers by compression combined with heat and steam," *Eur. J. Wood Prod.* 70(1-3), 155-163.
- Fengel, D., and Wegener, G. (1989). *Wood: Chemistry, Ultrastructure, Reactions*, Walter de Gruyter, Berlin.
- For. Prod. Lab. (FPL) Gen. Tech. Rep. FPL-GTR-113. (2010). "Wood Handbook- Wood as an Engineering Material," U.S. Dept. Agri., For. Prod. Lab., Madison, Wis., 4-27.
- Fukuta, S., Takasu, Y., Sasaki, Y., and Hirashima, Y. (2007). "Compressive deformation process of Japanese cedar (*Cryptomeria japonica*)," *Wood Fiber Sci.* 39(4), 548-555.
- Giebel, E. (1983). "Dimensional stabilization of wood by moisture-heat-pressure," *Holz Roh-Werkst.* 41(1), 87-94.
- Gong, M., Lamason, C., and Li, L. (2010). "Interactive effect of surface densification and post-heat-treatment on aspen wood," *J. Mater. Process. Technol.* 210(2), 293-296.
- Halligan, A. F. (1970). "A review of thickness swelling in particleboard," *Wood Sci. Technol.* 4(4), 301-312.
- Inoue, M., Norimoto, M., Tanahashi, M., and Rowell, R. M. (1993). "Steam or heat fixation of compressed wood," *Wood Fiber. Sci.* 25(3), 224-235.

- Inoue, M., Sekino, N., Morooka, T., Rowell, R. M., and Norimoto, M. (2008). "Fixation of compressive deformation in wood by pre-steaming," *J. Trop. Forest. Sci.* 20(4), 273-281.
- Irvine, G. M. (1984). "The glass transitions of lignin and hemicelluloses and their measurements by differential thermal analysis," *Tappi. J.* 67(5), 118-121.
- Ito, Y., Tanahashi, M., Shigematsu, M., Shinoda, Y., and Ohta, C. (1998a). "Compressive-molding of wood by high-pressure steam treatment: Part 1. Development of compressively molded squares from thinnings," *Holzforschung* 52(2), 211-216.
- Ito, Y., Tanahashi, M., Shigematsu, M., and Shinoda, Y. (1998b). "Compressive-molding of wood by high-pressure steam-treatment," *Holzforschung* 52(2), 217-221.
- Kamke, F. A., and Kutnar, A. (2010). "Transverse compression behavior of wood in saturated steam at 150-170 °C," *Wood Fiber Sci.* 42(3), 377-387.
- Kocaeffe, D., Poncsak, S., Tang, J., and Bouazara, M. (2010). "Effect of heat treatment on the mechanical properties of North American jack pine: thermogravimetric study," *J. Mater. Sci.* 45(3), 681-687.
- Korkut, S., and Hiziroglu, S. (2009). "Effect of heat treatment on mechanical properties of hazelnut wood (*Corylus columa* L.)," *Mater. Design.* 30(5), 1853-1858.
- Kutnar, A., Kamke, F. A., and Sernek, M. (2008). "Density profile and morphology of viscoelastic thermal compressed wood," *Wood Sci. Technol.* 43(1-2), 57-68.
- Lamason, C., and Gong, M. (2007). "Optimization of pressing parameters for mechanically surface-densified aspen," *Forest Prod. J.* 57(10), 64-68.
- Li, L., Gong, M., Huang, X., and Li, D. G. (2009). "Effect of surface free energy on Mode I fracture critical energy release rate of a laminated wood product," *International Symposium on Biomass Materials Science & Technology*, Huangshan, China.
- Li, L., Gong, M., and Li, D. G. (2012). "Recovery of mechanically induced residual stresses in densified softwoods," *Wood Fiber Sci.* 44(4), 365-373.
- Navi, P., and Girardet, F. (2000). "Effects of thermo-hydro-mechanical treatment on the structure and properties of wood," *Holzforschung* 54(3), 287-293.
- Navi, P., and Sandberg, D. (2012). *Thermo-Hydro-Mechanical Wood Processing*, EPFL Press, Lausanne.
- Rautkari, L., Laine, K., Kutnar, A., Medved, S., and Hughes, M. (2013). "Hardness and density profile of surface densified and thermally modified Scots pine in relation to degree of densification," *J. Mater. Sci.* 48(6), 2370-2375.
- Rowell, R. M., and Konkol, P. (1987). "Treatments that enhance physical properties of wood," *For. Prod. Lab. Gen. Tech. Rep. FPL-GTR-5512*.
- Salmén, N. L. (1982). "Temperature and water induced softening behavior of wood fiber based materials," *PhD thesis*, Royal Institute of Technology, KTH, Stockholm.
- Salmén, N. L. (1984). "Viscoelastic properties of in situ lignin under water-saturated conditions," *J. Mater. Sci.* 19(9), 3090-3096.
- Sergeeva, V. N., and Miljutina, S. V. (1960). "Variability of the properties of spruce wood fibres under the influence of heating," *Trudy Inst. Lesohoz. Probl., Riga*, 21, 101-106.

- Wantanabe, U., Norimoto, M., Ohgama, T., and Fujita, M. (1999). "Tangential Young's modulus of coniferous earlywood investigated using cell models," *Holzforschung* 53(2), 209-214.
- Zhang, Y. L., Zhang, S. Y., Chui, Y. H., and Wan, H. (2006). "Effect of impregnation and in-situ polymerization of methacrylates on hardness of sugar maple wood," *J. Appl. Polym. Sci.* 99(4) 1675-1683.

Article submitted: April 23, 2013; Peer review completed: May 22, 2013; Revised version received and accepted: June 2, 2013; Published: June 5, 2013.