J Mater Sci (2010) 45:681–687
DOI 10.1007/s10853-009-3985-7

Effect of heat treatment on the mechanical properties of North American jack pine: thermogravimetric study

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Received: 10 August 2009 / Accepted: 13 October 2009 / Published online: 27 October 2009

Abstract Heat treatment improves dimensional stability of wood, reduces its decay, and darkens its color. However, mechanical properties of wood can deteriorate during the heat treatment. The effect of heat-treatment conditions (maximum treatment temperature, heating rate, exposure time at the maximum heat-treatment temperature, and the gas humidity) on the mechanical properties of North American jack pine (*Pinus banksiana*) was studied using thermogravimetric analyzer. This type of study permits the identification of the best treatment conditions which will minimize reduction of mechanical properties of jack pine. The results showed that the degree of change in bending strength, hardness, screw withdrawal strength, and dimensional stability of jack pine during heat treatment depends strongly on the treatment conditions used. Therefore, great care should be taken to select the treatment conditions. Thermogravimetric analysis can be used as a first step for selection.

Introduction

Wood heat treatment is one of the wood preservation techniques, which is environment friendly. During heat treatment, wood is heated to temperatures above 200 °C depending on the wood species and the required wood properties. The treatment affects the wood cell wall polymers (hemicellulose, cellulose, and lignin). Hemicelluloses are very reactive, and they degrade first among the wood polymers [1] during the treatment. Cellulose and lignin [1–3] are also modified. The ratio of crystalline cellulose to amorphous cellulose increases is greater than that estimated by Sivonen et al. [2]. Cross-linking reduction in OH– (hydroxyl) groups and cleavage of the lignin-polysaccharide complex by organic acids released from hemicellulose degradation changes the chemical structure [4–9].

Dehydration, hydrolysis, oxidation, decarboxylation, and transglycosylation take place. These, in turn, change the wood properties. Wood becomes dimensionally more stable compared to untreated wood since decrease in hydroxyl groups increases the hydrophobicity of wood and reduces the absorption of water [4, 10–13].

Improvement in decay resistance of some species due to heat treatment is reported in the literature. This is attributed to reduction in hemicellulose content, moisture, and other wood components such as starch, fatty acids, and lipids which are essential for mold and fungi growth [6, 14].

However, heat treatment also decreases the mechanical properties of wood. Degradation of hemicellulose which connects cellulose and lignin in the cell wall causes deterioration in strength of wood. In literature, deterioration in toughness, hardness, bending, compression, and tension strength due to heat treatment are also reported [1, 6, 11, 15–22].

The degree of deterioration of the mechanical properties is a function of the heat-treatment conditions [23]. Therefore, the determination of the suitable heat-treatment conditions is very important for each species and different applications. Such an optimization is very costly under industrial conditions. A thermogravimetric study which uses small wood samples can determine the major trends while saving time and money for industry. There are different wood heat-treatment technologies such as ThermoWood, Bois Perdure, Retification Process, Plato, and OHT-Process.

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These technologies use different furnace designs, heating mediums, and treatment recipes. They are first developed in Europe [19, 23, 24] and brought to North America [21, 22] due to their environment friendly nature. Therefore, the adaptation of the treatment recipes to North American species is very important for obtaining good quality wood product. One advantage of the thermogravimetric analyzer is its flexibility in terms of adjustment of treatment conditions and environment. However, these tests use small wood samples and give the tendencies. The promising heat-treatment recipes should be tested under industrial environment.

In this study, the heat treatment of North American jack pine was investigated under different conditions using thermogravimetric analyzer, and the effect of the treatment parameters on the mechanical properties of jack pine is studied.

Materials and methods

Thermogravimetric heat-treatment experiments

The jack pine samples, with a dimension of 0.035 m × 0.035 m × 0.2 m, were heat treated in a thermogravimetric system under nitrogen and carbon dioxide atmosphere. This gas mixture represents the industrial conditions where the wood is treated with hot combustion gases. The gas humidity can be adjusted during the experiments by injecting vapor into the system. The uniformity of sample was verified by measuring the temperatures of gas and wood sample at different positions using copper–constantan thermocouples with a precision of ±2 °C.

The heat-treatment parameters studied were the maximum heat-treatment temperature, heating rate, exposure time at the maximum treatment temperature, and the gas humidity. The conditions of the experiments are given in Table 1. The details of the thermogravimetric system are reported elsewhere [21, 22].

Measurement of the mechanical properties

In order to assess the effect of heat-treatment parameters on the mechanical properties, three point bending (MOR, MOR), hardness, screw withdrawal strength, and the dimensional stability tests were carried out for untreated samples and heat-treated samples under different conditions, and the results are compared. MTS ALLIANCE RT 100 Universal Mechanical Test Machine was used for the measurements. Samples were conditioned in a chamber with 65% RH and 22 °C for 4 weeks before the tests.

Table 1 Summary of heat-treatment parameters

Maximum treatment temperature (°C)	Heating rate (°C/h)	Exposure time (min)	Gas humidity (g water vapor/m^3 dry gas)
120	20	0	100
160	20	0	100
200	20	0	100
210	20	0	100
220	20	0	100
220	10	0	100
220	30	0	100
220	20	15	100
220	20	30	100
220	20	45	100
220	20	0	0
220	20	0	150
220	20	0	200
230	20	0	100

Three point static bending tests were carried out according to The ASTM D-143 standard [25]. The size of the samples was 0.01 m × 0.01 m × 0.02 m. The moving head speed and the span length were 1.3×10^{-3} m/min and 0.1524 m, respectively. The load deformation data obtained were analyzed to determine the modulus of elasticity (MOE) and the modulus of rupture (MOR). Tests were repeated eight times for each treatment condition.

Penetration hardness tests were performed in accordance to The ASTM D-1324-83 standard [25]. Maximum force of 400 N was used during the test. Three samples with dimensions of 0.035 m × 0.035 m × 0.2 m were tested for each set of parameter. The diameter of the ball was 0.0113 m, and the penetration rate was 6×10^{-3} m/min.

ASTM D-1761-88 standard [25] was followed for the screw withdrawal strength tests using #10 screws with 0.025-m length and the withdrawal speed was 2.54×10^{-3} m/min. The screws were inserted to the depth equal to 2/3 of the samples width. Tests were repeated 4–6 times for every treatment parameter.

ASTM D-1037-104 standard [25] was followed for the dimensional stability tests. Sample dimensions were 0.035 m × 0.035 m × 0.01 m. Before the tests, samples were weighed and their average dimensions were measured in radial, tangential and longitudinal directions. During the tests, sample surfaces were kept 0.0254 m under the distilled water surface for 24 h. Water temperature was 23 ± 1 °C. Afterward, their weights and the dimensions were again measured. Percent change in mass (%CM) and swelling due to water absorption were calculated for each sample based on their initial mass and initial dimensions, respectively.

Results and discussion

Bending

Figure 1a–d present the effect of maximum heat-treatment temperature, heating rate, exposure time, and gas humidity on MOR and MOE, respectively. The results showed that both MOR and MOE increased with maximum heat-treatment temperature (Fig. 1a) up to 160 °C, and then they both decreased as temperature increased. At the highest temperature studied, the MOR of the heat-treated jack pine was lower and MOE was higher compared to those of the untreated jack pine. Similar findings were reported in the literature for different species. The mechanical property loss was explained as the results of hemicellulose degradation, increasing crystalline cellulose content, and replacement of flexible hemicellulose–cellulose–hemicellulose bonds with more rigid cellulose–cellulose bonds. [26–28].

The MOE of the heat-treated jack pine did not seem to be affected by heating rate (Fig. 1b). MOR of the samples heat-treated at heating rates of 20 and 30 °C/h were similar to the MOR of the untreated jack pine. However, at the lowest heating rate (10 °C/h) MOR was found to be significantly lower than the MOR of untreated jack pine. This deterioration might be explained as due to higher exposure times to heat.

The MOE was slightly higher and MOR was slightly lower compared to those of the untreated jack pine at the end of the treatment without any exposure time (0 min). Then, they first decreased somewhat with exposure time, followed by an increase up to 30 min. Further increase in exposure time decreased both MOE and MOR (Fig. 3c).

Humidity of gas did not seem to affect MOR significantly. MOE increased up to 150 g/m^3 with gas humidity. Further increase in gas humidity caused a decrease in MOE.The MOE and MOR of heat-treated samples were slightly higher than those of untreated samples (Fig. 1d) at higher humidities. Gas humidity plays an important role during the heat treatment of wood. A less humid gas dries the wood surface faster, and creates higher moisture and temperature gradients between the center and the surface of the wood, which can also cause formation of cracks [29]. If the gas is humid, wood is treated uniformly. In addition, water vapor acts as a protecting screen gas, helps to reduce the rate of oxidation of the wood samples, and hence, affects the modification of chemical structure [30]. Therefore, its effect depends on the reactions taking place under given conditions and species.

Hardness

The variation of the hardness with the maximum temperature of the heat treatment is presented in Fig. 2a, both in radial and tangential directions. At 120 °C, heat-treated jack pine found to be harder than the untreated wood. At this temperature, the wood is only dried but not chemically transformed, and this can increase the rigidity. At higher temperatures, hardness started to decrease gradually. This is probably due to starting thermal degradation. Above 200 °C, the decrease in hardness was very pronounced. The variation of the hardness is found to be more significant in the tangential direction compared to the variation in radial direction. However, the radial hardness was higher than the tangential hardness at any given temperature. Increase in

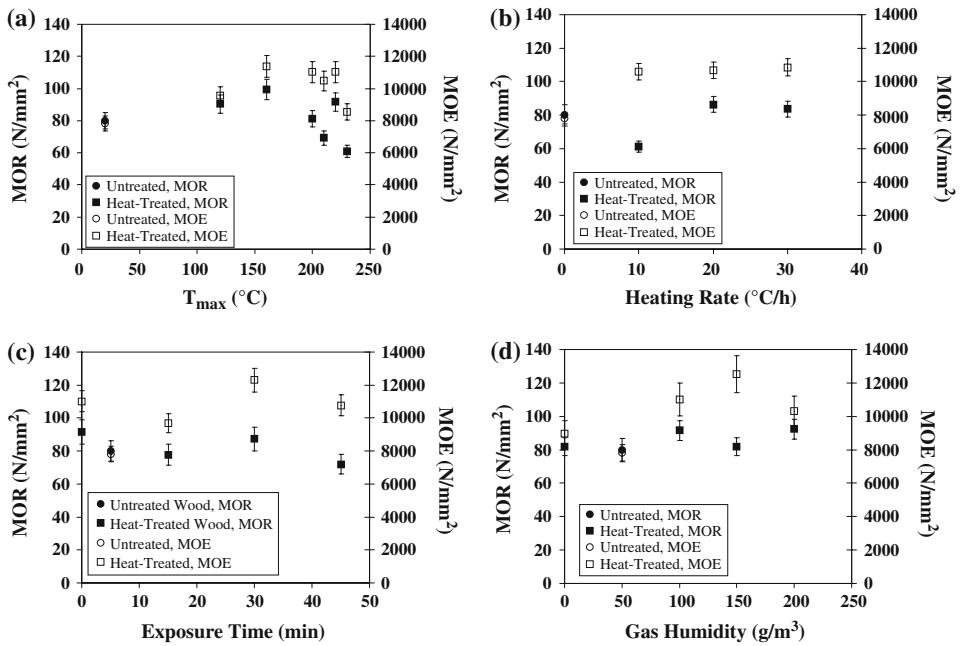

Fig. 1 Effect of **a** maximum treatment temperature (T_{max}), **b** heating rate, **c** exposure time, and **d** gas humidity on MOR and MOE

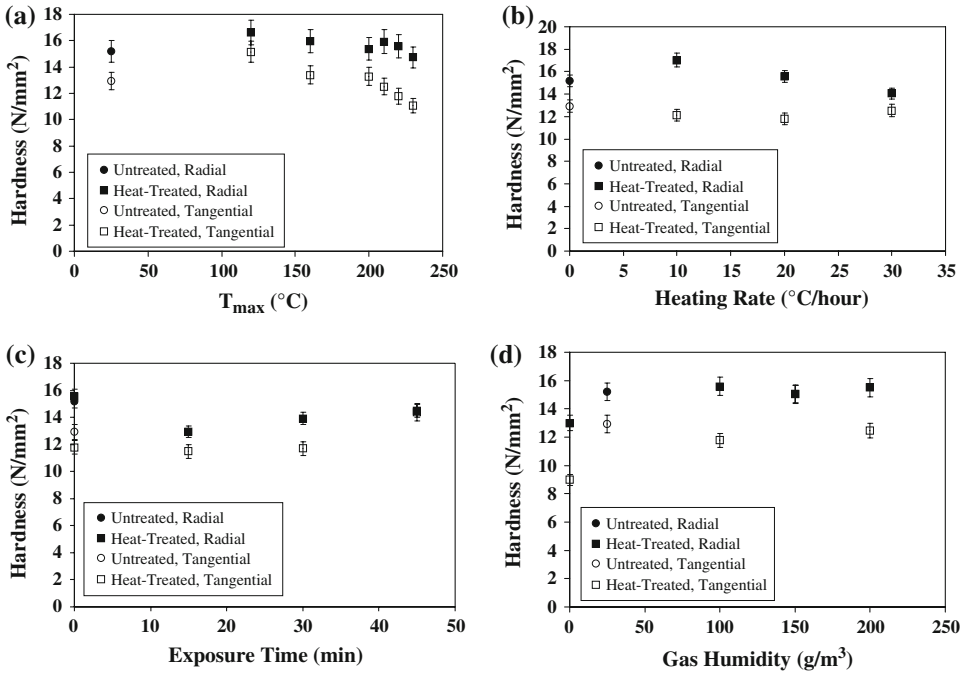

Fig. 2 Effect of **a** maximum treatment temperature (T_{max}), **b** heating rate, **c** exposure time, and **d** gas humidity on hardness

hardness during heat treatment was explained by ramification of lignin [2, 3, 31].

Tangential hardness did not seem to be affected by heating rate (Fig. 2b). Radial hardness increased when jack pine was heat treated with the lowest heating rate compared to that of untreated jack pine. Afterward, it decreased with increasing heating rate.

As shown in Fig. 2c, the tangential hardness of heat-treated jack pine did not seem to vary significantly with the exposure time. The radial hardness first decreased and then increased slightly as the exposure time increased.

If the hot gas does not contain any humidity (0 g/m^3) during the heat treatment, the hardness of the jack pine was reduced both in radial and tangential directions (Fig. 2d). The absence of humidity makes the treatment process more destructive [30]. Higher rate of oxidation and carbonation of wood manifests itself in the deterioration of the mechanical properties of the jack pine. When the gas is humid, the hardness was not deteriorated. However, increasing humidity from 100 to 200 g/m^3 did not seem to affect the hardness significantly.

Screw withdrawal strength (SWS)

Figure 3a shows the impact of the maximum heat-treatment temperature on the SWS of jack pine. Up to 160 °C, no significant effect of treatment on the SWS was observed. At higher temperatures, the thermo-degradation of the wood decreases gradually the strength of the jack pine, especially above 210 °C; thus, it becomes less resistant to screw withdrawal.

The SWS of untreated jack pine was slightly higher than that of heat-treated jack pine using heating rates of 10–20 °C/h (maximum temperature 220 °C). However, when 30 °C /h heating rate was used, wood became more resistant to screw withdrawal compared to wood heat treated at lower heating rates as well as the untreated wood (Fig. 3b). The fast increase of the temperature during heat treatment seems to improve the screw withdrawal strength. This is probably due to short contact time between hot gas and wood. The higher heating rate reduces the extent of the thermo-degradation which is directly related to the deterioration of the wood quality.

The duration of the exposure time at the maximum heat-treatment temperature did not seem to have any significant effect on the SWS of the jack pine (Fig. 3c). The SWS of heat-treated wood were found to be lower than that of the untreated wood for any exposure time.

The increase in the gas humidity during heat treatment improved the SWS of jack pine (Fig. 3d). Above certain gas humidity, the SWS is comparable (even higher at the highest gas humidity used) to that of the untreated jack pine. On the contrary, treatment with non-humid gas deteriorated the SWS of jack pine.

Dimensional stability

Figure 4a presents the percent mass change (%CM) of the heat-treated and untreated jack pine samples with maximum treatment temperature, due to water absorption during the dimensional stability tests. The values given at 20 °C represent the untreated wood. The heat-treatment temperature

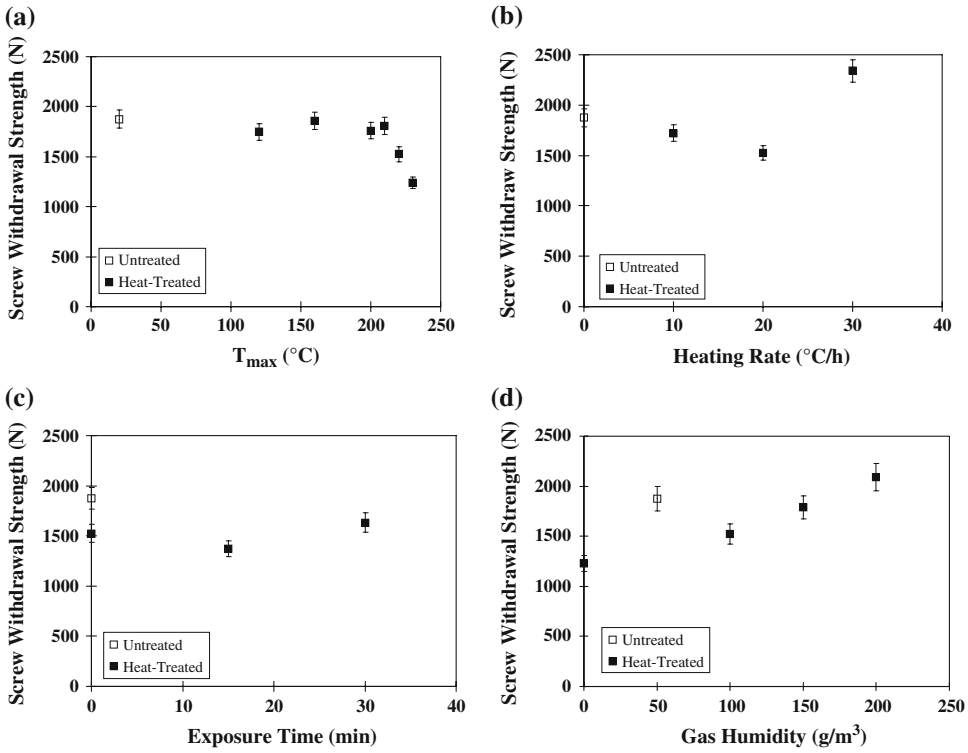

Fig. 3 Effect of **a** maximum treatment temperature (T_{max}), **b** heating rate, **c** exposure time, and **d** gas humidity on screw withdrawal strength

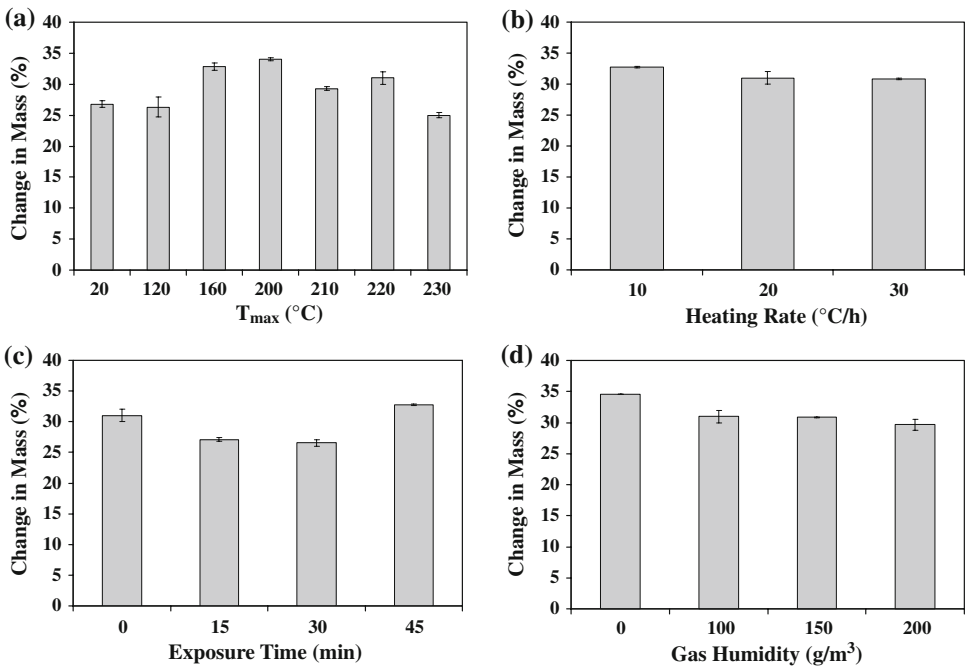

Fig. 4 Percent change in mass of heat-treated and untreated wood as a function of **a** maximum treatment temperature, **b** heating rate, **c** exposure time, and **d** gas humidity after immersion water for 24 h (%CM at 20 °C in (**a**) corresponds to untreated wood)

affected the %CM. It was observed that the change was the highest between 160 and 200 °C. At higher temperatures, it decreased slightly, but it was not significantly different than that of untreated jack pine. %CM decreased slightly with increasing heating rate (Fig. 4b) and gas humidity (Fig. 4d). As reported in the literature, heat treatment promotes the ramification of the lignin and the formation of ether linkages in the cellulose, and eliminates the hydrophilic hydroxyl groups 2, [3, 4, 10–13, 31]. These changes contribute to the reduction of the water absorption capacity of the wood. On the contrary, heat treatment at high temperature can also increase wood porosity [32] which may cause a higher water penetration during immersion test. These two phenomena determine mass change due to the water

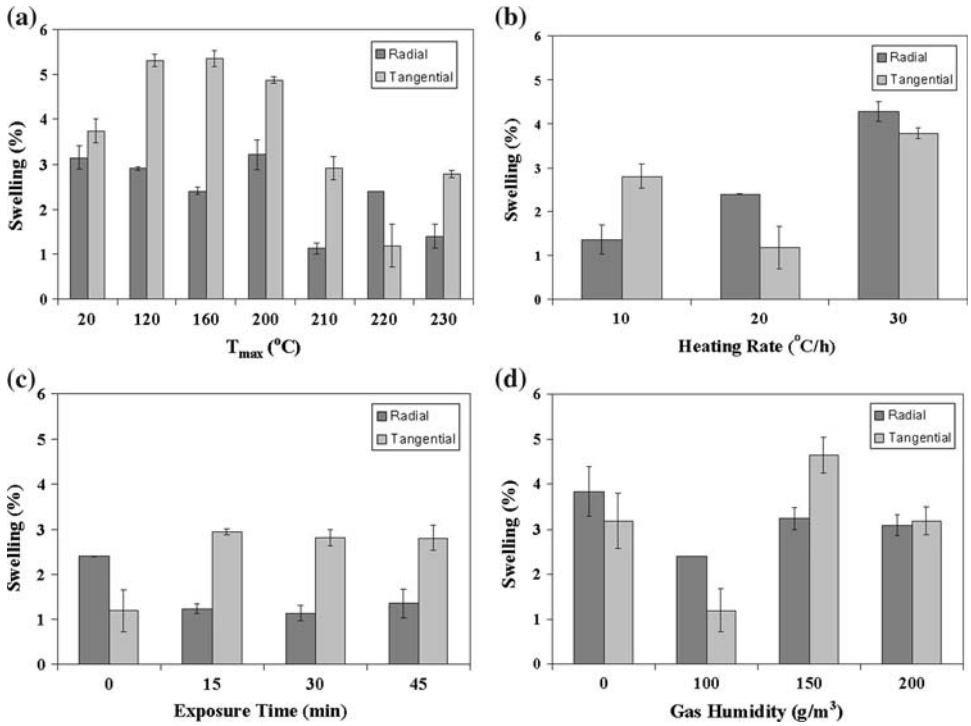

Fig. 5 Percent change in dimension of heat-treated and untreated wood as a function of **a** maximum treatment temperature, **b** heating rate, **c** exposure time, and **d** gas humidity after immersion water for 24 h (%CD at 20 °C in (**a**) corresponds to untreated wood)

absorption. The %CM is caused not only by absorption of water to the cell wall but also due to the water simply filling the pores. Therefore, swelling gives a better idea of the change in dimensional stability.

The swelling of the sample during the dimensional stability tests is shown in Fig. 5. Figure 5a shows that the dimensional stability is significantly affected by the maximum treatment temperatures. Between 200 and 210 °C, both the radial and tangential swelling decreased significantly compared to those of untreated wood. Improvement of dimensional stability depends on the extent of modification in wood's chemical composition due to heat treatment. Figure 5b shows that radial swelling increased slightly, and tangential swelling decreased between 10 and 20 C/h heating rates. When the heating rate is further increased swelling increased significantly in both directions. This could be due to the effect of increasing exposure time of wood to high temperature on the chemical composition. Exposure time seemed to have no significant impact on the dimensional stability within the range of 15–45 min of exposure time. However, decrease in radial swelling and increase in tangential swelling was observed when the exposure time was increased from 0 to 15 min. Swelling first decreased, and then increased with gas humidity in both directions. Another decrease was observed if the gas humidity was continued to increase (Fig. 5d). The gas humidity affects directly the thermotransformation reactions as explained above.

Conclusions

During this study, jack pine samples were heat treated at high temperature under an environment similar to that of industrial wood treatment process using thermogravimetric analyzer. Afterward, the mechanical properties and the chemical properties of the treated wood samples were measured and compared with those of the untreated ones. The results showed that thermogravimetric analyzer can be used as the first step in heat-treatment recipe development.

The results showed that there is a significant change in mechanical properties (MOE, MOR, hardness, etc.) due to heat treatment. Mechanical properties of heat-treated jack pine generally were deteriorated at temperatures higher than 200 °C compared to those of untreated jack pine; however, its dimensional stability was improved as a result of this treatment. The degree of change in these properties varied with different treatment conditions such as maximum treatment temperature, heating rate, exposure time, and gas humidity. Therefore, it is important to choose the conditions such that the treatment will yield to minimum flexibility loss and maximum improvement in dimensional stability.

Since different applications and species require different recipes, recipe development is very costly in industrial scale. The thermogravimetric tests results give the tendencies and reduce the cost for industry. However, the promising recipes should be tested under industrial conditions.

Acknowledgments The authors would like to thank the University of Quebec at Chicoutimi (UQAC) and the Foundation of the UQAC (FUQAC) for the financial support.

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