Effect of thermal modification temperature on the mechanical properties, dimensional stability and biological durability of black spruce (*Picea mariana*).

Lekounougou S[2]., Kocaefe D.[1]*

[1]*Department of Applied Sciences, University of Quebec at Chicoutimi,*
555 Blvd. de l'Université, Chicoutimi, QC, G7H 2B1, Canada

[2] *Experimental and Development Center in Boreal Forest (CEDFOB),*
537 Blanche, Baie-Comeau, QC, G5C 2B2, Canada
* Corresponding author: Kocaefe D**:** duygu_kocaefe@uqac.ca
Tel: (+1) 418-545 5011 ext 5215 Fax: (+1) 418- 545 5012

Abstract

This study was aimed at evaluating the effect of thermal modification temperature on the mechanical properties, dimensional stability and biological durability of *Picea mariana*. The boards were thermally modified at different temperatures, 190ºC, 200ºC and 210ºC. The results indicated that the thermal modification of wood caused a significant decrease in the modulus of rupture (MOR) after 190ºC, while the modulus of elasticity (MOE) seemed less affected with a slight increase up to 200ºC and slight decrease with further increase in temperature. The hardness of the thermally-modified wood increased in the axial direction. This increase was also observed in tangential and axial directions but at a lesser extent. The final value was slightly higher in axial direction and lower in radial and tangential directions compared to those of the untreated wood. Dimensional stability improved with thermal modification in the three directions compared to the dimensional stability of unmodified wood. The fungal degradation results showed that the decay resistance of thermally-modified wood against the wood-rotting fungi *T. versicolor* and *G. trabeum* improved compared to that of the untreated wood. By contrast, the thermal modification of *Picea mariana* had a limited effect on the degradation caused by the fungus *P. placenta*.

Keywords: *Biological resistance, dimensional stability, G. trabeum, mechanical properties, Picea mariana, P. placenta, T. versicolor, Thermally modified wood, wood rotting fungi*

Introduction

The boreal forest of the North American continent is dominated by black spruce (*Picea mariana*), an element of global biodiversity. Moreover, this species is present in North America and naturally absent elsewhere (Viereck & Johnston, 1990, Farrar, 1995). The black spruce forests are found in the east of the continent, in Québec, Ontario, Newfoundland and Labrador. However, the largest trees of this species are found in Quebec and it covers 28% of its surface area (Gagnon & Morin, 2001). The black spruce plays an ecologically and economically important role. Because of the quality of its fibers, it is highly sought after both in paper and construction industries.

Biological degradation including fungi, termites and marine borers is the main cause of deterioration of lignocellulosic materials. Among these, fungi cause the greatest financial loss (Bowyer et al., 2003, Goodell, 2003). The wood- rotting fungi can be classified as wood decay fungi, brown rot, white rot and soft rot fungi. The wood-rotting fungi secrete various enzymes including cellulase, hemicellulases, lignin peroxydase, manganese-dependent peroxydase and laccase to decompose the main constituents of the wood cell wall made of cellulose, hemicelluloses and lignin (Suziki et al., 2006, Yelle et al., 2008).

The environmental awareness in some European and American countries resulted in elimination of some preservatives containing inorganic metals in several areas (Preston, 2000). The environmental protection agency (EPA) in U.S banned the use of wood treated with copper-chrome-arsenic (CCA) for residential purposes after 2003. Alternative products such as wood-plastic composites (WPC), chemically modified wood and thermally-modified wood have grown rapidly (Baysal & Yalinkilic, 2005, Kartal et al., 2006, Chang & Chang, 2006, Clausen & Yang, 2007, Boonstra et al., 2007, Lekounougou et al., 2008). Thermal modification is an eco-friendly technology which improves the wood durability. This technology is non-toxic and does not require the use of chemicals. The utilization of thermal modification and the production of thermally-modified wood have increased considerably during the last decade (Hill, 2006, Shi et al., 2007, Bächle et al., 2010, Khalid et al., 2010, Korkut, 2012).

In this study, *black spruce* is thermally modified at different temperatures using the perdure technology in the prototype furnace of University of Quebec at Chicoutimi (UQAC). The aim of this study was to assess the efficacy of thermal modification temperature on the modulus of rupture (MOR), modulus of elasticity (MOE), dimensional stability and biological resistance of the black spruce used in the forest industry in Quebec and in the Saguenay-Lac-St-Jean area.

Materials and Methods

Tree species

Wood samples used in this study were collected from local sawmills of Saguenay-Lac-St-Jean, Quebec, Canada. All test samples were conditioned in a climatic chamber for 4 weeks at 65% ± 4% relative humidity and 23ºC ± 1ºC before the tests.

Thermal modification

Boards of black spruce with dimensions of 0.015 m x 0.045 m x 2.44 m were pre-dried in the air until the moisture content is reduced to 5-17%. The thermal modification of black spruce was carried out in a prototype furnace of University of Quebec at Chicoutimi

(UQAC), Quebec, Canada. The thermal modification was performed at three different temperatures, 190˚C, 200˚C and 210˚C. 15 boards were heated to maximum temperature with a heating rate of 15ºC/h in a humid atmosphere and inert gas, and were kept at this temperature for 1 hour. A detailed description of the process of thermal modification is published elsewhere (Poncsák et al., 2006).

Mechanical property and dimensional stability tests

All samples were placed in a climatic chamber for 4 weeks at 65% ± 4% relative humidity and 23ºC ± 1ºC before tests.

Three-point bending [modulus of rupture (MOR) and modulus of elasticity (MOE)], hardness, and dimensional stability tests were carried out with thermally-modified and unmodified wood under different conditions. The results were compared in order to determine the effect of thermal modification on the mechanical properties and dimensional stability of black spruce.

The bending and hardness tests are performed using the MTS ALLIANCE RT 100 Universal Mechanical Test Machine. Static three-point bending tests were carried out according to ASTMD-143-8 standard (ASTM international 2004). The size of the samples was 25.4 x 25.4 x 406.4 mm. The moving head speed and span length were 1.3 x 10^{-3} m/min and 0.1524 m, respectively. The obtained load deformation data were analyzed to determine the modulus of rupture (MOR) and modulus of elasticity (MOE). Tests were repeated 14 times for each treatment condition.

Penetration Hardness tests were performed in accordance with ASTM D-1324-83 standard (ASTM International, 2004). A maximum force of 400 N was used during the tests. Ten wood samples with dimensions of 25.4 x 76.2 x 152.4 mm were tested for each set of parameters. The diameter of the ball was 12.7 mm and the penetration rate was 6 x 10^{-3} m/min. Tests were repeated nine times on the radial, tangential and axial faces of each sample.

The tests to determine the dimensional changes were carried out with five samples with dimensions of 152.4 x 152.4 x 25.4 mm. ASTM D-1037-105 standard (ASTM International 2004) was followed for dimensional stability tests. Before the test, samples were weighed with Sartorius GW7201 analytic balance (accuracy ± 0.1 g), and their average dimensions were measured in the radial, tangential and axial directions. During the tests, the samples were kept immersed in distilled water for 24 h. The water temperature was 20 ± 1ºC. After, their weights and dimensions were again measured. Moisture content (MC) and swelling (SW) in the radial, tangential and axial directions were measured. Swelling for a given sample was calculated as:

$$SW_{t,r,a} = \left(\left(h_{treated} - h_{oven-dry}\right) / h_{oven-dry}\right) \times 100 \quad (1)$$

Where $h_{treated}$ is the dimension of the treated sample in mm; and $h_{oven-dry}$ is the dimension of the oven-dry sample in mm.

Fungi strains

The wood-rotting fungi including the white rot fungus, *Trametes versicolor* (FTK105D, *T. versicolor*) and brown rot fungi, *Poria placenta* (FTK120E, *P. placenta*) and *Gloephyllum trabeum* (FTK45D, *G. trabeum*) have been provided by FP-Innovation-Forintek, Quebec, Canada.

Wood block decay tests

In this study, the mini block test which was developed by Bravery (1979) for rapid evaluation of wood preservative fungicides was used. The methodology used for solid-state cultures of the wood has been adapted from EN-113 (1986) standard. 216 samples with dimensions of 0.015 m x 0.005 m x 0.035 m in radial, tangential and axial directions were prepared for each fungus and each treatment condition. The degradation of samples was studied based on EN 113 (1986) standard with the exception that the sample size was smaller following the works of Lekounougou et al. (2009), Bami & Mohebby (2011). In this study, sample size (0.015m x 0.005m x 0.035m) and test duration (12 weeks) were maintained in accordance with Lekounougou et al. (2009). Untreated black spruce wood was used as a reference for biological durability.

A malt agar based substrate was used as nutrient medium for cultivation of fungi in Petri dishes. It was prepared by mixing 30g of agar and 40g of malt extract dissolved in 1 L distilled water. The medium was sterilized in an autoclave at 121ºC (105 Kpa) for 35 min. 20 ml of medium was poured into sterile Petri dishes for inoculation. A disc (~5 mm diameter) of fungi was cut and placed in Petri dish and incubated in climatic chamber

(Conviron) at 22ºC ± 1ºC and relative humidity of 70% ± 4% for 2 weeks. The Petri dishes were found to be completely covered by the mycelium after 2 weeks. The modified and unmodified samples, previously conditioned at 103ºC, were placed into Petri dishes in the presence of three fungi, *T. versicolor*, *P. placenta* and *G. trabeum* and incubated for 12 weeks. Every 4 weeks, the samples were recovered and the mycelium was carefully scraped using a scalpel. Finally, the samples were placed in a furnace at 103ºC until their weight stayed constant. Three sets of each sample, both modified and unmodified, were placed in different Petri dishes. Each experiment was performed three times to ensure the reproducibility of the results. The weight loss of each individual sample was determined using the following equation:

$$\text{Weight loss (\%)} = (Wi\text{-}Wd/Wi \times 100) \tag{2}$$

Where Wi is the initial dry weight (g) of the sample before exposure fungal decay and Wd is the final dry weight (g) of the sample after exposure fungal decay.

The moisture content (MC, %) of sample was determined after fungal decay tests using following formula:

$$MC = 100 \times (W_C - W_D)/W_D \tag{3}$$

Where W_C is the wet weight after fungal decay (g) and W_D is the dry weight after fungal decay (g).

Results

Effect of thermal modification temperature on the mechanical properties and dimensional stability

Figure 1 shows the effect of thermal modification temperature on the MOR and MOE of black spruce. These data show that the effect of temperature on the MOR is more significant than its effect on the MOE. The MOR gradually decreases with increasing temperature. While the MOE increases slightly between 190ºC and 200ºC and decreases slightly at 210ºC. Similar results were found in the literature with other species (Bekhta & Niemz 2003, Pavlo et al. (2003), Poncsák et al. (2006) and Lekounougou et al. (2011)). They found that the MOR decreases when the thermal modification temperature increases, while the MOE does not appear to be significantly affected by temperature.

Figure 2 shows the impact of maximum temperature of thermal modification on the black spruce hardness. The results show that the black spruce hardness increases rapidly in the axial direction when heated to 190ºC before declining slightly with further temperature increase to 200ºC and 210ºC. The hardness of black spruce treated at these temperatures is slightly higher compared that of unmodified wood. In the radial and tangential directions, the black spruce hardness is not significantly affected at 190ºC, as shown in Figure 2. By contrast, there is a slight decrease in the black spruce hardness in radial and tangential directions at 200ºC and 210ºC. The increase in hardness in the axial direction may be due to the increase in the amount of highly ordered crystalline cellulose due to the crystallization of the amorphous cellulose and supporting structure of the wood.

It can be seen in the Figure 3 that the dimensional stability is significantly improved with thermal modification of black spruce. Indeed, the percentage of dimensional change of thermally-modified wood is considerably smaller than that of the unmodified wood in the three directions under the conditions used in this study. The effect of thermal modification on dimensional stability is more significant in the radial direction; a slight decrease is also observed in the tangential direction. By contrast, it seems that the black spruce has good dimensional stability in the axial direction before the thermal modification and it remains about the same during heat treatment.

Effect of thermal modification temperature of the black spruce on the decay fungi

Table 1 shows the effect of the thermal modification temperature on the weight loss of thermally-modified and unmodified black spruce samples incubated with three decay fungi, *T. versicolor*, *G. trabeum* and *P. placenta* for 12 weeks.

After 12 weeks of incubation, the greatest weight loss was recorded for the fungus *P. placenta* (57.5%), followed by *T. versicolor* (32.4%). The weight loss of 21.4% was recorded with *G. trabeum* fungus (Table 1). These data show that *P. placenta* fungus has the highest degradation efficiency of the samples of the untreated black spruce, followed by the fungus *T. versicolor*.

The results of the thermal modification summarized in Table 1 show that the increasing modification temperature leads to a decrease in weight loss for *T. versicolor* and *G. trabeum* fungi (Table 1). In the case of *T. versicolor*, the weight loss of unmodified wood is 32.4%, while the weight loss of modified wood decreases gradually with temperature until it reaches a weight loss of 0.6% at 210ºC. Similar results were obtained with brown rot fungus *G. trabeum* (Table 1). However, the thermal modification temperature of black spruce does not seem to affect significantly the degradation by *P. placenta* fungus (Table 1). In fact, the weight loss of 33.1% recorded after 12 weeks of incubation at 210ºC is relatively high compared to those of two other fungi (Table 1). The percent reduction of 40.4% is very low compared to the percent reduction obtained with *T. versicolor* (98.1%) and *G. trabeum* (100%) fungi (Table 1).

Discussion:

<u>Effect of thermal modification temperature on the mechanical properties and dimensional stability</u>

In general, the mechanical properties of wood decrease upon heating (Haygreen & Bowyer, 1996, Kocaefe et al., 2008, Lekounougou et al., 2011). When wood is heated to high temperature, thermal degradation and formation of furfurals monomers occur, thus, resulting in weight loss (Homan et al., 2000, Waskett & Selmes, 2001). As results of this weight loss, the strength property of wood is reduced, as shown in Figure 1. However, the increases in the degree of crystallinity of the cellulose and the width of the cellulose crystallites increase the elasticity of the wood. The mannose and xylose that are not degraded by the thermal modification can increase the crystallization of the cellulose and at least minimize the influence of the weight loss on the elasticity of the wood (Bhuiyan et al., 2000).

The thermal modification of wood at high temperature causes degradation of hemicelluloses and amorphous region of cellulose, thereby contributing to the increase in the degree of crystallinity of this polymer (Figure 3). In addition, cross-linking reactions between lignin and polymers taking place due to the thermal degradation of the wood are responsible for the decrease in the hygroscopicity and an improvement of the dimensional stability of thermally-modified wood (Bhuiyan et al., 2000, Vernois, 2001, Weiland & Guyonnet, 2003, Metsä-Kortelainen et al., 2006, Calenego et al., 2010).

As shown in Table 1, the moisture content (MC) of wood is higher for the tests with *T. versicolor* and *P. placenta* fungi. These results are consistent with Mburu et al. (2007), and Lekounougou et al. (2012). The presence of high moisture content (MC) observed in some samples can be considered due to the fungal degradation explained by the oxidative degradation of carbohydrates with the release of water molecules as indicated by Zabel et Morell (1992), and Borrega et al. (2009). Boonstra et al. (2006) also suggested that the fungal attack of highly crystalline cellulose can be transformed into amorphous cellulose, which makes the wood more hygroscopic.

Effect of thermal modification temperature of the black spruce on the decay fungi

Viitanen and al. (1994) reported a significant improvement in thermally-modified spruce resistance against the brown rot fungus *Coniophora puteana*, according to EN 113 (1986) Standard and soft rot fungi as a function of thermal modification level. Weiland & Guyonnet (2003) also used the EN 113 (1986) Standard and found that the resistance to fungal degradation in maritime pine and beech was improved by 43% and 74%, respectively, compared to unmodified wood (control). Hakkou et al. (2006) found a significant correlation between biological durability and the thermal modification temperature. Decay resistance tests conducted by Metsa-Kortelainen and Viitanen (2009) on the sapwood and heartwood of Scots pine and Norway spruce, which have been thermally modified at 170-230°C using the thermowood method, against soft and brown rot fungi, also confirmed that the increasing thermal modification temperature increases the decay resistance.The improvement of biological durability in different species of thermally-modified wood was also reported by Kamdem et al. (2002), Jones et al. (2006), Calenego et al. (2010), and Lekounougou et al. (2012). According to published data, there are several reasons for the increase in the biological durability of thermally-modified wood. Indeed, during the thermal modification, the wood becomes more hydrophobic, thereby; limiting the absorption of the water into the wood can prevent fungal growth. The thermal modification changes the chemical composition of wood, which makes the attack of wood by the decay fungi more difficult. The wood polymers can also be modified or degraded during the thermal modification and for that reason; the wood cannot be used as a source of nutrition by the rotting-wood fungi (Weiland & Guyonnet 2003).

Conclusion :

The effect of thermal modification temperature on the mechanical properties, dimensional stability and biological durability of black spruce is investigated. The results showed that increasing temperature decreases in the modulus of rupture (MOR) significantly, while the modulus of elasticity is affected to a lesser extent. The thermal modification of black spruce improves also the dimensional stability as well as the biological durability against wood-rotting fungi, *T. versicolor* and *G. trabeum*. However, this treatment has not significant effect on the black spruce durability against *P. placenta* fungus.

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Table 1 Effect of thermal modification temperature on the decay resistance of black spruce *(Picea mariana)* wood to the three decay fungi after 12 weeks of colonization and reduction in weight loss as compared to the untreated wood.

Fungal species	Heat treatment temperature (°C)	MC* (%)	Weight loss (%)	Reduction in weight loss due to thermal modification (%)
T. versicolor	Untreated	80.7 ±1	32.4	-
	190°C	38.9 ±1	13.2	59.2
	200°C	49.2 ±1	2.2	93.2
	210°C	77.3 ±1	0.6	98.1
G. trabeum	Untreated	61.1 ±1	21.4	-
	190°C	90.2 ±1	10.4	51.4
	200°C	69.1 ±1	4.2	80.4
	210°C	11.5 ±1	0.0	100.0
P. placenta	Untreated	135.8 ±1	57.5	-
	190°C	114.6 ±1	30.9	44.3
	200°C	95.6 ±1	34.2	38.4
	210°C	79.9 ±1	33.1	40.4

*MC: moisture content

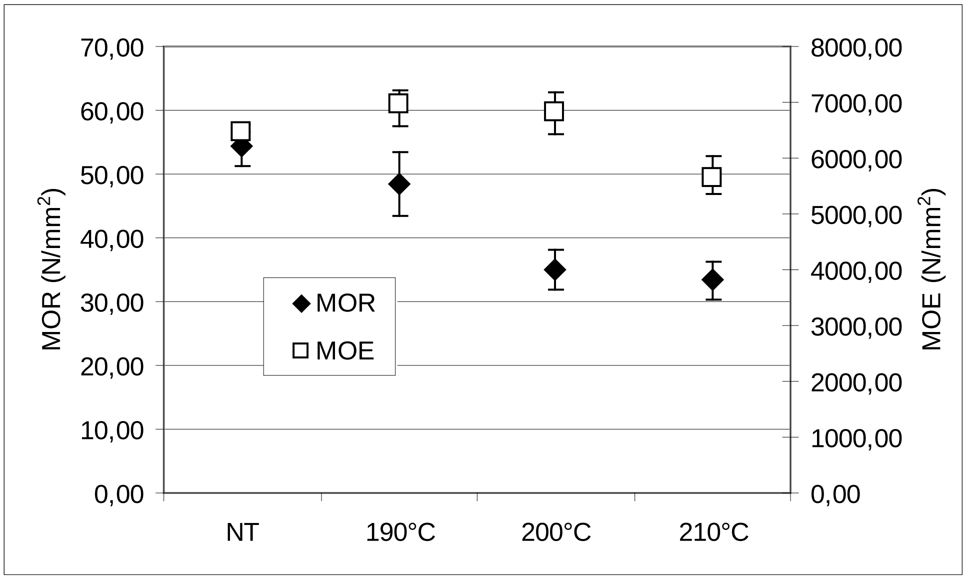

Fig. 1 Impact of maximum treatment temperature on MOR and MOE of black spruce (*Picea mariana*). Each point is the mean ± standard deviation of fourteen different experiments (NT: untreated sample, MOR: modulus of rupture, MOE: modulus of elasticity)

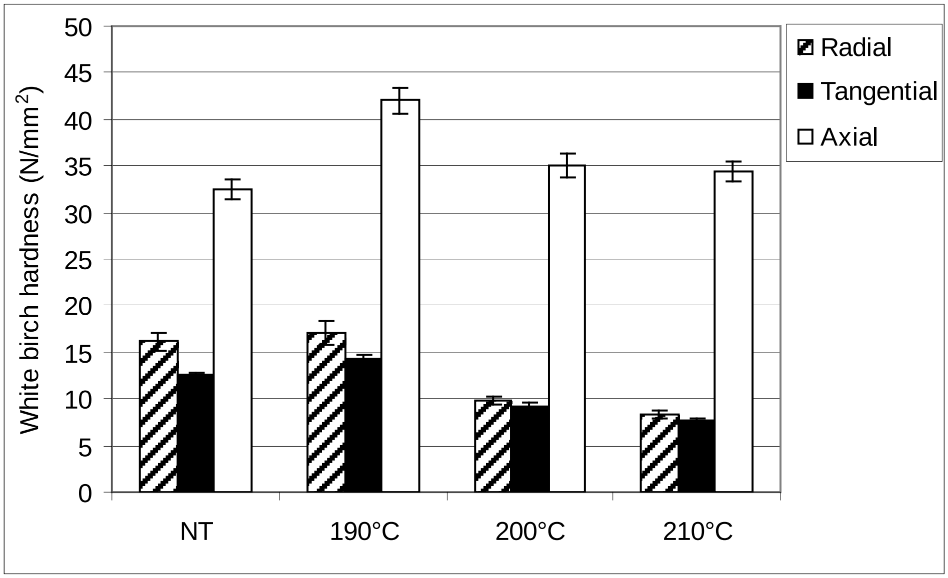

Fig. 2 Impact of maximum treatment temperature on black spruce hardness (*Picea mariana*) treated in the prototype furnace of UQAC. Each point is the mean ± standard deviation of fourteen different experiments (NT: untreated sample)

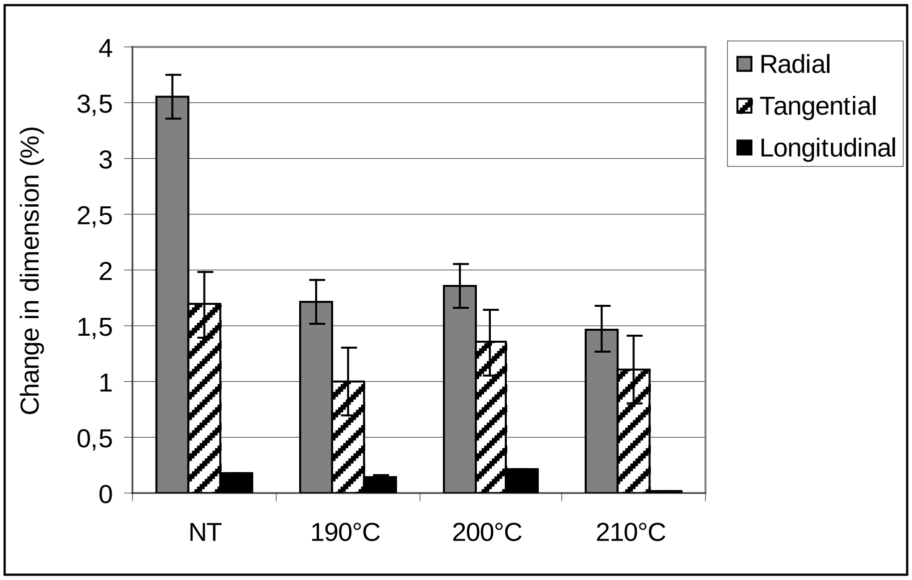

Fig. 3 Percent change in the dimension of thermally modified and untreated black spruce as a function of maximum treatment temperature after immersion in water for 24 hours (% CM at 20^{o}C). Each point is the mean ± standard deviation of fourteen different experiments. (NT: untreated sample, CM: moisture content)