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Effect of heat treatment on hardness, density and color of *Populus* × *canadensis* 'I-214' wood

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Abstract

Aim of study: To evaluate the effect of heat treatment (HT) on hardness, density and color of *Populus* × *canadensis* ′I-214′ (poplar) wood.

Area of study: 15-years-old poplar wood from Pomona, Río Negro, Argentina.

Materials and methods: 352 samples were exposed to different HT schedules: 120ºC, 160ºC, 180ºC and 200°C for 45 min, 90 min, 135 min and 180 min. Hardness, density and color were determined before and after each HT combination. Hardness and density tests were performed following the specifications of IRAM standards and wood color was determined according to CIE*Lab* system.

Main results: Hardness improved significantly at 160°C for 45 min and 90 min in comparison with control (14.34% and 9.08%, respectively) whereas this property was improved at 120°C in all cases without significant differences. The 200°C: 45 min schedule showed the worst performance with a 20.26% hardness loss. Density was lower than the control in all schedules with losses ranging from 2.50% to 10.00%. Color became darker (decrease in L value, increase in a and b values) as HT intensity increased (mainly temperature), with changes becoming evident at 180°C and 200°C.

Research highlights: HT on *P.* × *canadensis* 'I-214' improved its hardness under two HT schedules, although was not enough to extend the feasible applications, since it still belongs to a category of "soft" wood. Hardness and density did not show a clear correlation and color of poplar wood became darker as HT intensity increased..

Additional key words: physico-mechanical properties; CIE*Lab* system; wood quality.

Abbreviations used: EMC (equilibrium moisture content); HT (heat treatment); MC (moisture content).

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Introduction

Heat treatment (HT) is one of the most environmentally friendly alternative processes to modify and improve some properties of wood. Mainly, the industrial process involves wood modification using high temperatures at different levels and with various time intervals –HT schedule– (Boonstra, 2008; Korkut *et al.,* 2008; Esteves & Pereira, 2009; Baysal *et al.,* 2014; Elaieb *et al.,* 2015). Several studies have reported on enhanced properties of thermally modified wood such as resistance to fungal attack, equilibrium moisture content, shrinking and swelling. In addition, the color of heat-treated wood adds aesthetic value (Korkut & Hiziroglu, 2013; Salca &

Table 1. Number of *Populus* × *canadensis* 'I-214' wood samples according to HT schedules.

Time (min)	Control	Temperature (ºC)				Replications
		120	160	180	200	
Control	88	0	0	0	0	88
45	0	22	22	0	22	66
90	0	22	22	22	0	66
135	0	22	22	22	0	66
180	0	22	22	22	0	66
Total	88	88	88	66	22	352

Hiziroglu, 2014; Van Nguyen *et al.,* 2018; Pratiwi *et al.,* 2019). These properties allow for its use in indoor products (parquet, tile, kitchen furniture, sauna walls or floors) and outdoor products (entrance doors, windows, exterior cladding and garden furniture). Therefore, thermally modified wood from fast-growing plantation forests could be used as a substitute for natural forest resources, which are scarcemore valuable and less available on the market (Salca & Hiziroglu, 2014; Borůvka *et al.,* 2018).

Despite the improved qualities mentioned above, thermally modified wood generally has an undesirable side-effect: mechanical property reduction, which might limit the range of feasible applications (Boonstra *et al.,* 2007; Esteves & Pereira, 2009). Although mechanical properties decrease as temperature increases, the scale varies depending on the chemical and anatomical nature of the wood and the HT method used (Esteves *et al.,* 2008; Korkut *et al.,* 2008; Gündüz *et al.,* 2009; Lekounougou & Kocaefe, 2014; Kesik *et al.,* 2014). Consequently, there are studies which show different results, such as decreases or increases in different mechanical properties of heat-treated wood (Boonstra *et al.,* 2007; Gündüz *et al.,* 2008, 2009; Kocaefe *et al.,* 2008; Korkut *et al.,* 2008; Moliński *et al.,* 2016).

Mechanical properties depend not only on HT parameters, but also on wood species and the property being examined (Moliński *et al.,* 2018). Considering that the hardness shows different performances, several authors have reported a decrease in values on softwoods species depending on HT intensity (Elaieb *et al.,* 2015; Ulker *et al.,* 2018). On the other hand, Shi *et al.* (2007) reported an increase in hardness of *Picea* spp., *Abies* spp., *Pinus* spp. and *Betula* spp. In the same line, Kocaefe *et al.,* (2008) reported an increase in hardness of birch heat-treated wood. Reports on *Populus* spp. show conflicting views as regards hardness. For example, Spavento (2015) studied the Brinell hardness of *P.* × *canadensis* 'I-214' heat-treated wood and reported an increase in hardness, whereas Shi *et al.* (2007) studied the hardness of *Populus* spp. heat-treated wood and reported a decrease on its property. Both species came from different places and had different growth, what influences the intrinsic characteristics of wood.

Generally, the hardness is strongly related with wood density, therefore an increase in this physical property brings an increase of hardness. Mostly, the wood used from fast-growing forests has lower density than the wood from natural forest resources, due to a high proportion of juvenile wood (Tuong & Jian, 2010) and, consequently, lower hardness. Therefore, HT could be an alternative technology to increase the density, thus the hardness. Several authors have found an increase in this property on conifers and hardwood species, as *Robinia pseudoacacia*, *Alnus glutinosa*, *Pinus radiata*, among others (Kesik *et al.,* 2014; Herrera-Díaz *et al.,* 2017). Nevertheless, Ockajova *et al.* (2019), Priadi *et al.* (2019) and Sözbir *et al.* (2019) found that the density decreased with increasing temperature and time of HT schedule for softwood and hardwoods, like *Picea Abies*, *Tectona grandis* and *Populus usbekistanica* among others.

Color change with HT is the most studied surface property as it turns wood darker (Esteves *et al.,* 2008; Baysal *et al.,* 2014; Griebeler *et al.,* 2018; Ulker *et al.,* 2018). Like in the case of the above-mentioned properties, difference in color also depends on HT schedule, becoming darker as temperature and time increase (Cademartori *et al.,* 2013). Gündüz & Aydemir (2009) reported that temperature has a much more significant effect on color than exposure duration. Griebeler *et al.* (2018) and Priadi *et al.* (2019) concluded that HT causes darkening in *Eucalyptus grandis* and *Tectona grandis* species compared with controls. Korkut *et al.* (2013) reported that wood color changed significantly in heat-treated *Prunus avium*, showing lower redness and yellowness. Moreover, Srinivas & Pandey (2012) and Nasir *et al.* (2018) concluded that wood color varies due to the chemical changes that occur during HT.

The *P.* × *canadensis* 'I-214' wood is a fast-growing species with low hardness and density, and it has a light white to greyish yellow color. The HT might increase the hardness and density of wood and could change the color to a more attractive one, thus expanding its range of feasible applications and ends uses. According to this, the aim of this study was to evaluate the effect of HT on hardness, density and color of *P.* × *canadensis* ′I-214′ wood.

Material and methods

Wood material

Fifteen-years old *P.* × *canadensis* `I-214´ (poplar) wood from Pomona, Río Negro (39º 29′ 40.74″S, 65º 35′ 28.01″W), Argentina, was used. The harvest was carried out by avoiding differences in plantation density, growth rate and border effect. The sawn wood was obtained in 50 mm × 150 mm × 3000 mm boards (thickness, width and length, respectively), which were then conditioned at the Laboratorio de Investigaciones en Maderas (LIMAD-UNLP) to achieve, by air dry conditions, an equilibrium moisture content of 12% ± 3%, determined by a Hydromette HT 85-GANN digital hygrometer. Subsequently, boards were cut into defect-free radial 20 mm × 50 mm × 50 mm samples (thickness, width and length, respectively); 352 samples were selected.

Heat treatment

Heat treatment was performed using a temperature-controlled laboratory oven. The samples were exposed to different temperature: time combinations (HT schedules): 120°C, 160°C, 180°C and 200°C for 45 min, 90 min, 135 min, and 180 min (Table 1) under atmospheric pressure and in the presence of air. Beyond the 200°C: 90 min schedule, the specimens ignited, that is the reason why only 22 samples were subjected to 200°C: 45 min; later, 66 samples for three 180°C combinations were added. Table 1 shows the HT schedules and the control (C) without treatment. The thermo-treated samples were conditioned at LIMAD-UNLP to achieve, by air dry conditions, an equilibrium moisture content (EMC). The moisture content (MC) was calculated following the Eq. (1):

$$MC = \left(\frac{W_h - W_o}{W_o}\right) * 100 \quad (1)$$

where W_h was the weight at EMC and W_o was the oven-dry weight of the same sample, both after HT. In both cases, W_h and W_o, the weight was evaluated through successive weighing (weight differences of ± 0.01 g).

Hardness

The Janka hardness test was conducted on a Shimadzu UH-300 kN universal testing machine according to IRAM 9570 (1971) standard. Hardness was determined by the force (in kg) applied on the wood surface by a steel ball that leaves a 1-cm^2 indentation. This process was repeated on the same sample before and after HT, twice on diagonally opposite sides of the control samples, and twice on diagonally opposite sides after HT. Before carrying out the hardness test, moisture content of each control sample was evaluated through Eq. (1), but in this case W_h was the weight at EMC and W_o was the oven-dry weight of the same sample, both before HT. After HT, the MC was determined as indicated previously and then, the hardness test was performed in the same way as above.

Density

Air-dry wood density was measured on each test sample before and after HT in accordance with IRAM 9544 (1985) standard. Density was calculated as the ratio of air-dry wood mass (kg) to air-dry wood volume (m^3).

Color

Colorimetric analysis was carried out at the same two locations on each sample before and after HT. Color was measured with a BYK No 115772 spectrophotometer; the measurements were made by a standard illuminant D65 at a 10-degree observation angle according to CIE*Lab* color system. CIE*Lab* is a three-dimensional color space which measures the lightness (*L*) and the color coordinates (*a* and *b*) of each sample. The *L* axis represents reflectance, which ranges from 0 to 100, from black to white, respectively. The *a* chromaticity coordinate represents red (+*a*) and green (-*a*), whereas *b* represents yellow (+*b*) and blue (-*b*), with values ranging between 0 and 60. Total color difference (ΔE) was calculated according to Eq. (2) (Cademartori *et al.,* 2013):

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (2)$$

where ΔL, Δa and Δb represent the reflectance and colorimetric differences between two different states of the samples.

In order to define the levels of total perceived color change (with the naked eye), ΔE was classified according to the Hidayat *et al.* (2016) scale as follows: ΔE from 0 to 0.5 is defined as negligible; ΔE from 0.5 to 1.5 is slightly perceivable; ΔE from 1.5 to 3.0 is noticeable; ΔE from 3.0 to 6.0 is appreciable; ΔE from 6.0 to 12.0 is very appreciable; and ΔE over 12.0 is totally changed.

Statistical analysis

All statistical analyses were performed using R© software (v. 4.1.1; R Core Team, 2021). Data corresponding to different treatments (Table 1) were analysed on 352 samples. The assumptions of independence, normality and homoscedasticity were checked for all groups on Janka hardness, density and color. Normality and homoscedasticity requirements were checked using the Kolmogorov Smirnov Lilliefors and Levene's test, respectively, and then, different

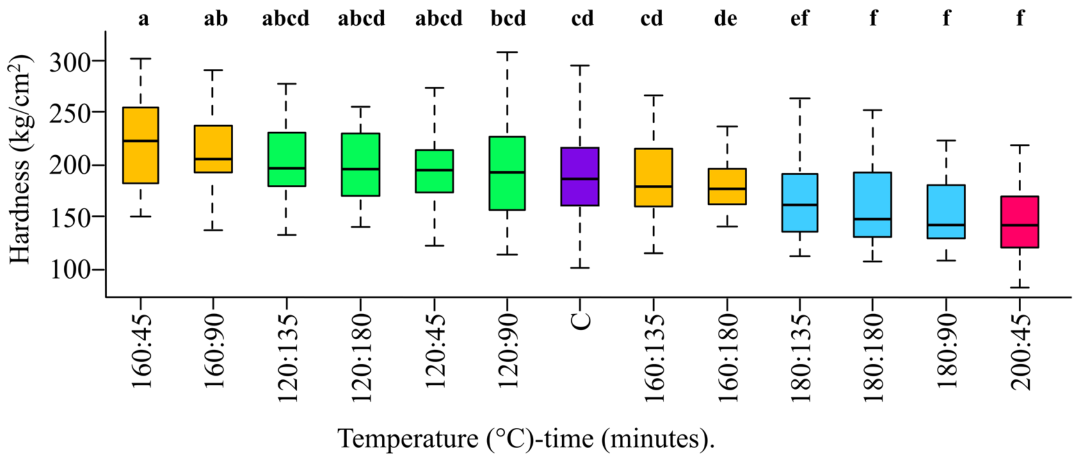

Figure 1. Janka hardness values according to HT schedules (temperature, ºC: time, min). C: control. Different letters mean a significant difference ($p < 0.05$).

comparison techniques were used. Since hardness and density were non-normal and homoscedastic, a Kruskal-Wallis test was run to detect differences. A Spearman correlation was carried out to assess the correlation between hardness and density. Color data were non-normal and heteroscedastic, hence, a Welch´s generalized comparison robust test and Lincon's post hoc analysis were used. For all analyses, a significance level of α = 0.05 was used.

Results

Hardness and density

Increase or decrease in hardness depends on the temperature:time combination. Fig. 1 shows a tendency to a decrease in hardness with higher temperatures (180°C: light blue, 200°C: pink) in comparison with control (purple C). At 160°C (yellow) for 180 min, the specimens show a decrease in hardness, whereas at 160°C for 45 min and 90 min the results were significantly higher than control, being at 45 minutes better than 90 minutes. At 120°C (green), performance tended to improve as compared with control in all time intervals, but without significant differences.

Furthermore, when the intensity of HT increased the MC decreased (Table 2), and when the MC was equal or lower than 8.32% (Table 2) there was a lost in hardness. Moreover, there were critical points with a slightly change of MC meant gain or loss of hardness. The first was from 120°C: 180 min (MC=10.27%) to 160°C: 45 min (MC= 10.07%), when MC loss of 0.2% meant 9.87% gain in hardness, going from 201.21 g/cm² to 220.21 g/cm² (Table 2). The other critical point was between 160°C: 45 min (MC 10.07%) and 160°C: 90 min (MC= 8.97%), where the MC descended 1.1% and lost 5.26% in hardness, going from 220.21 g/cm² to 210.09 g/cm², even though the schedule 160°C: 90 min, was better than control (Table 2).

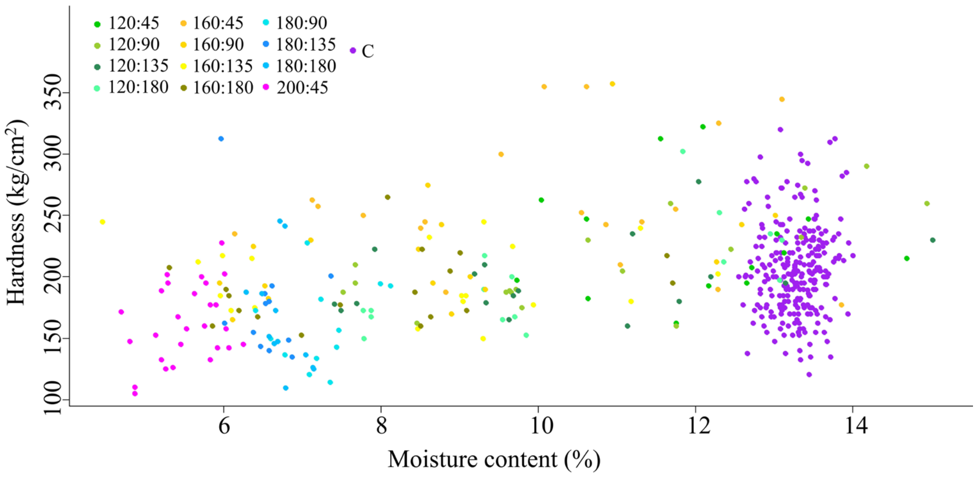

Figure 2. Dispersion of moisture content (5) and hardness (kg/m²) values according to HT schedule (in color legend, temperature, ºC: time, min).

Table 2. Hardness and density of heat-treated poplar wood.

Temperature (°C)	Time (min)	MC average (%) [1]	Statistical values [2]	Density (kg/m^3)	Density (%)	Hardness (kg/cm^2)	Hardness (%)
Control		13.28	Avg.	400.00	-	192.60	-
			±sd	2.00		39.64	
			CV	6.09		20.58	
120	45	12.01	Avg.	380.00	-5.00	199.20	+3.43
			±sd	2.00		33.87	
			CV	4.94		17.01	
	90	10.65	Avg.	370.00	-7.00	200.73	+4.22
			±sd	3.00		47.93	
			CV	8.72		23.88	
	135	10.52	Avg.	380.00	-5.00	201.82	+4.79
			±sd	1.00		36.08	
			CV	3.51		17.88	
	180	10.27	Avg.	380.00	-5.00	201.21	+4.47
			±sd	2.00		33.56	
			CV	4.70		16.68	
160	45	10.07	Avg.	380.00	-5.00	220.21	+14.34
			±sd	2.00		43.32	
			CV	5.75		19.67	
	90	8.97	Avg.	380.00	-5.00	210.09	+9.08
			±sd	2.00		32.79	
			CV	6.02		15.61	
	135	8.32	Avg.	380.00	-5.00	190.29	-1.20
			±sd	2.00		36.75	
			CV	4.60		19.31	
	180	7.93	Avg.	390.00	-2.50	182.56	-5.21
			±sd	1.00		24.70	
			CV	1.76		13.53	
180	90	7.22	Avg.	370.00	-7.50	157.46	-18.25
			±sd	2.00		34.18	
			CV	6.47		21.71	
	135	6.55	Avg.	390.00	-2.50	169.13	-12.19
			±sd	2.00		37.94	
			CV	5.86		22.43	
	180	6.74	Avg.	370.00	-7.50	164.94	-14.36
			±sd	3.00		39.51	
			CV	7.45		23.95	
200	45	5.51	Avg.	360.00	-10.00	153.58	-20.26
			±sd	2.00		34.52	
			CV	6.09		22.48	

[1] MC: moisture content, in %. [2] Avg: average. ±sd: standard deviation, in kg/m^3 (density) or kg/cm^2 (hardness). CV: coefficient of variation, in %.

As shown in Fig. 2 there was a generally positive trend between the hardness and MC for all schedules analysed together, with no significant and positive correlation (r= 0.24). Also, the control points (Fig. 2, purple) were concentrated nearly to 13.28% of MC (Fig. 2, Table 2). On the contrary, the lowest results of hardness obtained at 200ºC: 45 min (pink) were concentrated closely to 5.51% of MC (Fig. 2). Although, analysing the intermediate programs

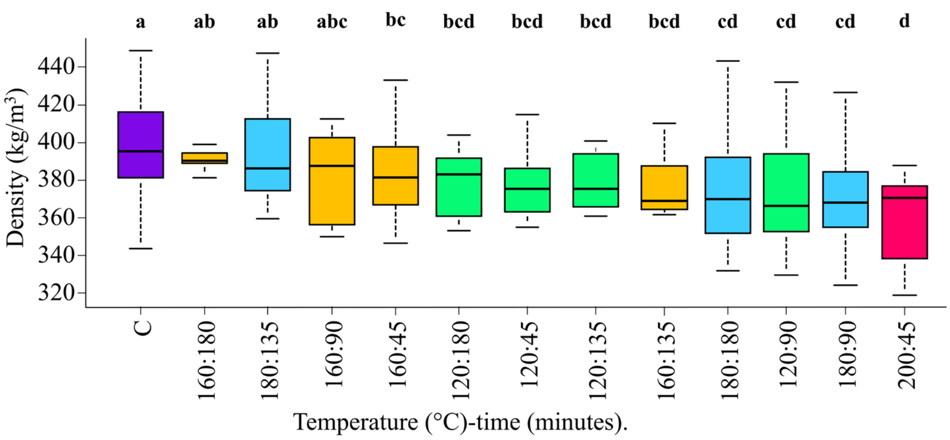

Figure 3. Density values according to HT schedules (temperature, ºC: time, min). C: control. Different letters mean a significant difference ($p < 0.05$).

separately, the tendency was not clearly at all. At 160ºC: 45 min the tendency was the opposite and showed a negative correlation between MC-hardness (r= -0.39), that confirms the general rule that the lower the MC, the higher the resistance mechanics properties, although without differences as regards to control. Nonetheless, the 120ºC: 180 min and 160ºC: 90 min schedules, showed a positive MC-hardness correlation (r = 0.11 and r = 0.32, respectively), and a positive trend, with the gain of hardness values, which, probably, could be related to the chemical changes occurred during HT. Fig. 3 shows changes in density of *P.* × *canadensis* 'I-214' against HT schedules. As regards wood density, it showed a trend to decrease with all temperature and time combinations compared with control. Only 160ºC: 180 min; 160ºC: 90 min and 180ºC135 min schedules showed values without significant difference as regards control.

As shown in Fig. 1 and Table 2, the best hardness value was obtained at 160°C: 45 min (220.21 kg/cm^2), showing a significant increase of 14.34% as regards control. Moreover, at 160°C: 90 min hardness increased significantly (210.09 kg/cm^2) by 9.08% as compared with control. In contrast, the 180ºC: 90 min, 135ºC: 180 min 200ºC: 45 min schedules showed a significant decrease in hardness with respect to the control with greater loss percentages (18.25, 12.19, 14.36 and 20.26%, respectively), but without significant differences among them. The rest of the schedules did not show significant differences as regards control; at 120°C for all time intervals there was a 3-5% hardness gain, whereas at 160°C: 135 min and 180 min there was a 1% -5% decrease in hardness.

As opposed to hardness, density decreased by 2.50-10.00% in all HT schedules (Table 2). Despite of the decrease mentioned, three combinations (160°C: 180 min, 1.80°C: 135 min and 160°C: 90 min) did not show significant differences compared with control (Fig. 3). The 200ºC: 45 min schedule was the least effective, with the biggest density loss (10%) as compared to control (Table 2).

The density-hardness correlation showed a significant and positive correlation between both variables with r: 0.12 (in all treatment schedules), but this was not the case in each treatment considered separately in which there was no significant correlation in the 160ºC: 45 and 160ºC: 90 schedules (p>0.05). However, hardness improved while density decreased beyond the 160ºC: 45 min and the 160ºC: 90 min HT schedules. In addition, 200ºC: 45 min showed a strong negative correlation (r=-0.65, p<0.05).

Color

In general, the color (L, a, b) of control vs. heat-treated wood at 120°C and 160°C varied only slightly (Table 3, Fig. 4). However, the wood became darker with increasing HT intensity, beyond 180°C; especially at 200°C, the color darkened notably and differences were easily observed by naked eye. The reduction of L is directly related to the darkening of wood, and the increase in the values of coordinates a and b contributed to the darkening. Poplar wood has a light white to greyish yellow color, which became darker as HT became more intense (Table 3, Fig. 4).

Generally, lightness values (L) decreased with HT intensity. Especially at 120°C (for all time intervals), 160°C: 45 min and 160°C: 90 min, lightness values did not show statistical differences as regards to control, whereas in schedules ranging from 160°C: 135 min to 200°C: 45 min they did (represented by * in Table 3). As regards lightness values, at 120°C no statistical differences were observed irrespective of the time intervals; at 160°C, the 45 min and 90 min intervals were different as compared with 160°C: 180 min; and 180°C: 90 min was different from 180°C: 180 min (represented by letters in Table 3). Thus, time of exposure was significant depending on the temperature, as temperature increased, time exposure became more significant and the wood became darker. The largest reduction of L recorded was 52.5% for the 200°C: 45 min schedule. As

Table 3. Statistical values of coordinates *L*, *a*, *b* and color change (*ΔE*) for heat-treated poplar wood.

Temperature	Time	*L*[1]	*a*[1]	*b*[1]	*ΔE*[1]
Control		79.91 (2.17)	3.77 (1.2)	13.04 (2.26)	-
120	45	80.18 (1.95) a	3.72 (1.10) a	12.24 (1.21) a	2.35 (1.26)
	90	78.12 (3.20) a	4.48 (1.45) a	13.63 (1.87) a	3.32 (2.92)
	135	80.22 (2.21) a	2.51 (1.23) b	16.78*(1.12) b	4.66 (1.17)
	180	79.05 (2.61) a	4.16 (1.28) a	12.39 (1.63) a	2.96 (1.81)
160	45	79.75 (2.37) a	2.86 (1.21) a	17.32*(1.65) a	5.09 (1.72)
	90	79.78 (1.82) a	3.85 (1.06) b	14.40*(1.40) b	2.37 (1.58)
	135	77.94*(2.07) ab	4.77 (0.87) c	15.25*(1.47) b	3.49 (2.15)
	180	76.78*(2.23) b	5.11 (0.75) c	15.84*(1.47) b	4.67 (2.28)
180	90	64.52*(5.42) a	7.87*(1.39) a	22.36*(2.51) a	18.69*(5.37)
	135	61.13*(6.99) ab	8.93*(1.53) b	22.47*(2.60) a	21.97*(6.59)
	180	59.92*(4.77) b	9.38*(1.40) b	25.92*(1.24) b	24.60*(4.23)
200	45	38.00*(4.31)	10.80*(2.43)	15.44 (4.56)	42.90*(3.90)
		$p < 0.05$	$p < 0.05$	$p < 0.05$	$p < 0.05$

[1] Average of each parameter with the standard deviation in brackets. *: significant differences against control. Different letters show significant differences between time intervals for each temperature.

regards redness (*a*), in general there was a slight increase at 120°C and 160°C without significant differences respect to control (Table 3). Then, at 180°C the increase was significant relative to control; the highest value was recorded at 180 min (*a*=9.38) with an increase of 9.35% with respect to control (*a*= 3.77). Therefore, at 200°C, the a coordinate also increased significantly by 11.71%. Concerning time intervals, at 120°C there were significant differences for the 135 min interval as opposed to the 45 min, 90 min and 180 min ones. At 160°C: 45 min, a presented a statistical difference as regards to the 90 min, 135 min and 180 min intervals, and the 90 min interval was different from the 135 min and 180 min ones. At 180°C: 90 min, a was different from 135 min and 180 min. Thus, when temperature was higher, time interval became significant and the redness increased too.

Concerning the b variable, the 120°C: 45 min, 90 min and 180 min and 200°C: 45 min schedules did not present statistical differences as regards control (Table 3). The remaining schedules showed a significant increase compared with control. Thus, as temperature rose, the wood became more yellowish than control. The highest value was obtained at 180°C: 180 min (*b*= 25.92), with 20.87% increase respect to control. Although the b value at 200°C did not show differences as regards control, there was a slight increase contributing to the darkening of wood with only 45 min of exposure. The *b* coordinate at 120°C: 135 min showed more significant differences than 45 min, 90 min and 180 min, whereas the 160°C: 45 min schedule differed from the 90 min, 135 min and 180 min ones. The 180°C: 180 min schedule showed differences compared to the 90 min and 135 min time intervals at the same temperature. Hence, as temperature increased, time became significant and the heat-treated wood became more yellowish.

The total color change was not significant at 120°C and 160°C for any of the time intervals with respect to control, thus, time exposure did not affect the total color change of heat-treated *P.* × *canadensis* 'I-214' (Table 3). At 180°C, color change was significant compared with control at all time intervals; 200°C also showed significant differences as regards control. The color of poplar 'I-214' became darker as the temperature increased and it was clearly visible to the naked eye (Fig. 3), reaching 42.9 at 200°C: 45 min. As regards the classification described in materials and methods, the total color change (*ΔE*) classified as noticeable at 120°C: 45 min, 90 min and 180 min and at 160°C: 90 min, whereas at 120°C: 135 min and 160°C: 45 min, 135 min and 180 min the change classified as appreciable (Fig. 4). Besides, at 180°C for all time intervals and 200°C, *ΔE* changed completely.

Discussion

Hardness and density

According to our best results, HT combination of 160°C: 45 min increased hardness by 14.3%; however, was not enough to grade up the poplar hardness category, as it is still a "soft" category wood (Panshin & De Zeeuw, 1980).

With respect to MC and hardness, Herrera-Díaz *et al.* (2019) reported that a reduction of MC could be associated with the hygroscopic polymers degradation during HT. Therefore, according to our results, roundly 10% of MC, the chemical changes produced in the wood could be responsible for hardness values variation. Hardness gains,

Figure 4. Wood color changes during heat treatment (HT) process at 45 min and 90 min.

with few changes in MC, could represent a window to explore different combinations of temperatures and times that enhance this promising result. Contrary with our results, Hermoso *et al.* (2015) working with *Pinus radiata* wood, reported non-significant differences between the MC and hardness, although the MC of this wood showed a decrease when the intensity of HT increase.

Spavento (2015), using similar HT methodology and for the same clone, albeit with a different origin and growth site (Spain), found a decrease in hardness for all the time intervals tested at 120°C, whereas in our study there was a small improvement in hardness at 120°C for all time intervals (Table 2). In accordance with our results, the highest hardness improvement was reported for the 160ºC: 45 min schedule, whereas Spavento (2015) reported the highest increase in this mechanical parameter for the 160°C: 135 min schedule. Moreover, the same author reported an increase in hardness on heat-treated wood at 200°C both for the 45 min and the 135 min interval. In our work, the wood ignited beyond 200ºC:50 min.

Several researchers have found a decrease in hardness in heat-treated poplar wood (*Populus tomentosa*, *Populus tremuloides*) at temperatures beyond 200°C, with loss percentages greater than 27% (Shi *et al.,* 2007; Chu *et al.,* 2016; Ming-Li *et al.,* 2019); our highest loss percentage at 200ºC was 20.26% (Table 2). In similar research works on *Populus* spp. species, Chu *et al.* (2016) analysed the behaviour of heat-treated *Populus beijingensis* at 160ºC, 180ºC, 200ºC and 220°C for 120 min, and they found a decrease in density followed by a decrease in hardness in all schedules, with hardness losses of 2.30%, 6.87%, 8.17% and 18.42%, respectively. Gong *et al.* (2010) also recorded a slight decrease (2%) in the density of *P. tremuloides* heat-treated at 145°C for 7 min. Kozakiewicz *et al.* (2019) carried out a 160ºC, 190ºC and 220°C heat treatment for 120 min on *Populus nigra* and they noticed that its density decreased by approximately 13% at 220°C and showed no significant difference in the rest of the schedules.

During HT, the wood undergoes chemical changes which could explain the modifications of its properties, the components start to change into a plastic state by different ranges of temperature, the hemicellulose does above 127°C and lignin above 167°C, as a consequence of HT the amount of crystalline cellulose increase (Herrera-Díaz *et al.,* 2019). Like hardness and density, the chemical structure of wood differs from untreated wood as a result of HT conditions such as temperature and time of exposure (Esteves & Pereira, 2009). Several authors claim that the reason for the difference in mechanical properties among species and HT schedules lies in depolymerization reactions and changes in the hemicelluloses, which play an important role in wood strength (Kocaefe *et al.,* 2008; Elaieb *et al.,* 2015; Herrera-Díaz *et al.,* 2019). Kocaefe *et al.* (2008) also reported a slight increase in hardness at 200°C in *P. tremuloides* and *Betula papyrifera* possibly due to cellulose crystallization resulting from degradation of amorphous hemicellulose content. Generally, regardless of HT conditions, the process has an impact on the mechanical properties and density, so the chemical composition of *P. × canadensis* “I-214” might play an important role in the final properties of heat-treated wood. In furthers studies we will consider the analysis of chemical changes on heat treated poplar wood to explain the mentioned properties evaluated.

Color

The color of poplar wood became darker (decrease in *L* value, increase in *a* and *b* values) when HT conditions intensity increased. This property varied slightly when the temperatures were lower (particularly at 120ºC), but at 180°C a total color change was noticed.

Consistent with these results, Güller (2012) found a decrease in the *L* coordinate for *P. nigra*, with values of 67.84 in the control and 42.71 for the 200°C:60 min HT schedule. However, the author recorded an L value of 37.86 for the 200°C:120 min schedule, similar to our L value of 38 for the 200ºC: 45 min schedule. In line with our results, Fehér *et al.* (2014) and Yang *et al.* (2021) did not find differences at 120°C; nonetheless, they found that changes in *L* became noticeable beyond 150°C, becoming darker when the temperature increased for *Pterocarpus macrocarpus*, *Quercus robur*, *Fraxinus excelsior*, *Fagus sylvatica*, *Prunus avium* and *Acer campestre*.

As regards the a coordinate, Güller (2012) found, in opposite to the findings of this research, a decrease for *P. nigra* from 12.96 units to 11.11 for 190°C:60 min, and 10.32 for 200°C:60 min. On the other hand, Griebeler *et al.* (2018) reported an increase in a value of 18.5% for *Eucalyptus grandis*. A similar trend to our study was shown by Sikora *et al.* (2018), who reported an increase in a for *Picea Abies* and *Q. robur* at temperatures of 160ºC and 180°C, and a decrease at 210°C. Along the same line, Srinivas & Pandey (2012) reported an increase in a at the beginning and a decrease as time exposure grew for *Havea brasiliensis* and *Grevillea robusta*.

Consistent with our results, for the *b* parameter, Fehér *et al.* (2014) and Sikora *et al.* (2018) found an increase in the values as temperature increased for *A. campestre* and *Q. robur*, respectively. Especially, Fehér *et al.* (2014) reported a 45% decrease at higher temperatures (210°C). On the other hand, Griebeler *et al.* (2018) found that the mean of the *b* coordinate decreased at 140°C and 180°C but increased at 160°C, that agree well with our results for 160°C (Table 3).

Generally, the performance of color change (ΔE) founded in this work was similar to those in literature. In agreement with our results, Van Nguyen *et al.* (2018) reported an increase in ΔE for *Populus alba* as temperature grew, reaching 41.76 at 220°C:8 hours. Also, our results for poplar at 180°C were similar than those found by the aforementioned authors, with values ranging from 22.58 to 27.03. Several authors have reported an increase in this parameter as temperature and time increased. For instance, Cao *et al.* (2012) found an increase in ΔE for *Cunninghamia lanceolata*, particularly ranging from 185°C to 230°C in heartwood. Hidayat *et al.* (2016) reported higher values of ΔE when temperature increased for *Cylicodiscus gabunensis*. They also reported total color change for sapwood and heartwood after 2 hours of exposure at 180°C. Moreover, Dzurenda (2018) found significant color changes for *Q. robur* at a maximum temperature of 140°C after 7.5 hours of exposure.

In line with our results, Hidayat *et al.* (2016) found an appreciable change in sapwood and heartwood color at 180°C after 1 hour of exposure and a total color change after 2, 3 and 4 hours of exposure for *Cylicodiscus gabunensis*. On the other hand, Dzurenda (2018) reported a total color change for *Q. robur* at 140°C for 7.5 hours with a ΔE value of 20.1. For the 130°C:6.5 hour schedule, the author recorded a big color difference and for the 115°C:5.5 hour schedule, the color difference became visible. During HT, the chemical changes not only affected the hardness and density as described, but also wood color due to the reactions of its compounds. Discoloration could be linked to polymerization or oxidation reactions (Chen *et al.,* 2012; Wei *et al.,* 2017). Hemicellulose, lignin and extractives play a role in the color changes of heat-treated wood. However, it varies according to wood species, part of the tree, environmental conditions and HT intensity (Kučerová *et al.,* 2016; Wei *et al.,* 2017). A decrease in lightness (*L*) indicates that many compounds that absorb visible light are formed, thus, the degradation of hemicelluloses and the formation of low molecular sugars and quinones are responsible for the darkening of the wood. Also, an increase in polyphenol content, from a condensed lignin and extractives, results in an increase of red tones in the wood (Kučerová *et al.,* 2016). Moreover, Chen *et al.* (2012) concluded that heat-treated wood in the presence of an oxygen-rich atmosphere induces hydrolysis reactions in the wood, which form yellow low-molecular-weight phenolic substances. Therefore, the *P.* × *canadensis* 'I-214' could be subjected to the reactions mentioned due to the darkening, redding and yellowing of the color which came about after the HT process. As mentioned above, a future analysis should be performed to explain the changes that the color of poplar wood undergoes.

Conclusions

P. × *canadensis* 'I-214' improved its hardness under two HT schedules: 160°C: 45 min and 160°C: 90 min. However, was not enough to extend the feasible applications, it remains a "soft" wood. Although hardness and density are strongly related, during HT their behaviour in some cases was different. Therefore, an increase in hardness did not mean an increase in density for this heat-treated wood. Also, density decreased as treatment intensity increased. The color of poplar wood became darker as HT schedules intensity increased. This property varied slightly when the temperatures were lower, particularly at 120°C; at 160°C the color became as darker as exposure increased, and when temperature was higher than 180°C, a total color change was noticed.

In further studies, the chemistry of heat-treated 'I-214' poplar wood will be analysed to better understand the HT process studied in this research, because the chemistry of wood plays an important role during HT due to the reaction of compounds, which might change hardness, density and color of the wood analysed.

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