Microstructure of Thermally Modified Radiata Pine Wood

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The thermal modification of wood is a potential alternative method for improving wood dimensional stability and increasing the resistance of wood to decay. However, during thermal modification, morphological changes occur within the microstructure of the cell, and these confer different properties to the wood. This study investigated the effects of the thermal modification process on the microstructure of radiata pine juvenile wood. Therefore, anatomical measurements were performed *via* optical microscopy in selected earlywood and latewood samples after each treatment, and the results were compared to untreated wood samples. In this study, two temperatures (190 °C and 210 °C) were considered for the thermal modification process. The results showed that the level of temperature of modification affected to microstructure of cell wall. The cell wall thickness decreased as treatment temperature increased, whereas the average lumen diameter increased slightly as temperature increased. Thermally modified radiata pine showed signs of damage (cracks, broken cells and deformations in the wood cell wall). The proportion of destroyed area increased as temperature increased, and significant differences were evident for the thermal treatment at 210 °C.

Keywords: Cell wall thickness; Heat treatment; Juvenile wood; Lumen diameter; Thermal modification; Wood anatomy

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INTRODUCTION

The thermal modification of wood is performed at temperatures between 160 °C and 240 °C under varying operating conditions, such as steam, vacuum, or nitrogen (Hill 2006; Militz and Altgen 2014; Sandberg *et al*. 2017). The effects of thermal modification on the physical and mechanical properties of wood have been widely published (Kubojima *et al*. 2000; Yildiz *et al.* 2006; Hill 2006; Boonstra *et al*. 2007; Esteves and Pereira 2009; Esteves *et al*. 2014; Tasdemir and Hiziroglu 2014; Uribe and Ayala 2015; Sandberg *et al*. 2017, among others); however, from an anatomical point view the effects on cell structure have not shown clear trends (Boonstra *et al*. 2006a,b; Awoyemi and Jones 2011; Kekkonen *et al*. 2010; Welzbacher *et al*. 2011; Biziks *et al*. 2013; Batista *et al*. 2015; Bernabei and Salvatici 2016; Jiang *et al*. 2017). In addition, literature on the topic has been limited due to the difficulty of studying the effects before and after heat treatment on the same piece of wood (Kekkonen *et al*. 2010; Bernabei and Salvatici 2016; Wentzel *et al*. 2020).

The main advantages of thermal modification are reduced hygroscopicity and equilibrium moisture content, increased dimensional stability and permeability (Tjeerdsma and Militz 2005; Hill 2006; Esteves and Pereira 2009; Uribe and Ayala 2015), increased resistance of wood to natural weathering, and improved durability and decay resistance

(Kamdem *et al*. 2002; Nuopponen *et al*. 2005; Welzbacher and Rapp 2007), decreased volumetric shrinkage and swelling, and hardness (Uribe and Ayala 2015).

During thermal modification, chemical changes occur within the structure of the cell wall, and these confer new characteristics and properties to the wood. Recently, Faraone *et al*. (2020) found that the thermal modification is also a strategy to improve the antioxidant activity of wood extractives. The degradation of the polymers and extractives of the cell wall occurs due to changes in chemical composition through different chemical reactions (Esteves *et al*. 2008). However, the mechanical properties of thermally modified wood tend to decrease (Kubojima *et al*. 2000; Yildiz *et al.* 2006; Boonstra *et al*. 2007). In addition, hardwoods show higher strength losses than softwood when modified under the same conditions (Hill 2006; Boonstra *et al*. 2007).

The anatomical structures of wood can be modified depending on certain treatment parameters, such as temperature, process duration, heating rate, and wood species (Boonstra *et al*. 2006a; Poncsák *et al*. 2006). The degradation of the polymers and extractives of the cell wall occurs due to changes in chemical composition through different chemical reactions (Esteves *et al*. 2008). According to Jiang *et al*. (2017), differences in the distribution of surface chemical composition can produce different cell wall fractures (predominantly at the middle lamella) as well as a lost moisture content, resulting in defects, such as collapse, cracks, and deformations (Boonstra *et al*. 2006a), because wood mass, dimension, and the shape of the wood cells begin to change when increases or decreases the wood moisture content. It has also been found that these changes are related to the temperature, drying time, and drying rates, among other (Reiterer and Tschegg 2002; Redman *et al*. 2016; Jiang *et al*. 2017; Zhang *et al*. 2018, Nopens *et al.* 2019). After heat treatment, Uribe and Ayala (2015) observed differences in shape and size of the pore, as well as a significant deterioration in the cell wall appearance.

Kekkonen *et al.* (2010) observed that lumens' dimensions decrease in the three directions during thermal modification of *Pinus sylvestris*. They also indicated that around 200 °C (critical temperature), the pore size begins to increase because of the decomposition of the cell walls. Hietala *et al*. (2002), observed that increase or decrease pore size depends on the wood species and modification process.

Awoyemi and Jones (2011) reported that the destruction of tracheid walls and ray tissues occurred during the heat treatment of red cedar wood. In addition, the authors concluded that the changes in anatomical structure could have contributed to the changes in wood properties. Likewise, a scanning electron microscopic analysis of heat-treated beech and spruce wood indicated that heat-induced defects in their microstructure contribute to the strength loss of thermally modified wood (Welzbacher *et al*. 2011). This was also confirmed by Biziks *et al*. (2013), who studied the effect of heat treatment on the microstructure of birch wood. They concluded that the changes in the microstructure differ noticeably depending on the thermal treatment conditions and the type of the cell, as they observed a peak decrease of 37% of the fiber wall area after modification at 180 °C. Bernabei and Salvatici (2016) studied the anatomical characteristics of spruce wood during heat treatment using an environmental scanning electron microscope. They found that cell wall thickness decreases at high temperatures. In addition, their results indicated apparent differences between latewood and earlywood cell walls size, but the differences were not confirmed statistically. Lin *et al*. (2017) studied the effect of thermo-vacuum treatment on the anatomical characteristics of Szemao pine and alder birch wood. For Szemao pine, small radial cracks were observed, and some ray parenchyma cells were destroyed. For alder birch wood, the anatomical structures were almost unaffected. Based on these results,

the authors concluded that the thermo-vacuum treatment had a slight effect on the anatomical structure without causing serious damage. In their study of thermally modified Scots pine at 180 °C, Kymäläinen *et al*. (2018) showed that delamination and damage in the cell walls occurred, and radial cracks close to the tracheids were present in the earlywood.

In contrast, some studies, such as that performed by Andersson *et al*. (2005), on the thermally modified wood of *Pinus sylvestris*, have reported that thermal modification did not lead to a change in anatomical structure. Likewise, Batista *et al*. (2015) reported that thermal modification did not significantly affect the structure of the ray parenchyma, vessels, or fibers of *Eucalyptus grandis* wood. In addition, no significant changes were observed in fiber dimensions. Recently, Wentzel *et al*. (2020) investigated the anatomical characteristics before and after thermal modification of *Eucalyptus nitens* wood in open and closed systems. Their results showed that there were no noticeable differences in anatomical structure between the two modification systems, and there were only slight changes in the cell wall thickness and fiber and lumen areas after thermal modification.

In this study, the microstructure thermally modified radiata pine juvenile wood was analyzed focus on visualizing and quantifying exploratory the morphological changes after thermal modification.

EXPERIMENTAL

Materials

For the experiments, dried sawn wood of radiata pine (*Pinus radiata* D. Don) from a 26-year-old plantation in the Bio Bio region of Chile was utilized. The core-wood (juvenile wood) was sawn in boards of 25 mm × 100 mm × 3200 mm (width, thickness, and length, respectively). The sawn core-wood was dried in a chamber (Model Lab3.5e, Neumann, Concepcion, Chile) using a drying schedule at 100 °C / 70 °C (dry bulb / wet bulb) with an airflow speed of 6 m/s. The temperatures inside the chamber were monitored, and the temperature and moisture content of the wood were recorded according to the setup and kiln schedule.

Methods

Thermal modification process

Laboratory-scale thermal modifications at 190 °C and 210 °C were made using the kiln-dried wood. An initial load of 140 samples was evenly stacked and placed in equidistant layers separated by sticks of 20 mm in thickness. The tests were realized in a flexible industrial prototype chamber with a capacity of 3.5 m^3 (Model Lab3.5e, Neumann, Concepcion, Chile) designed to withstand temperatures up to 250 °C. For each process, the chamber was loaded with 140 samples according to the schedules implemented by Herrera-Díaz *et al*. (2019). The first phase of the modification process began with a fast increase to temperature to 100 °C. In the second phase, the temperature was increased at a rate of 0.7 °C/min until reaching 190° C or 210 °C. During this stage, the wood was sprayed with steam to avoid damage. At the end of the second phase, the wood reached a moisture content of 3% to 4%. Subsequently, the temperature (190 °C or 210 °C) was maintained for approximately 3 h. In the final step, the samples were cooled and stabilized for approximately 5 h to 7 h. The total time of thermal modification was approximately 32 h, and the final wood moisture content ranged from 9% to 11%.

Anatomical measurements

For studying effects of heat treatment on wood, before the thermal modification process 152 sawn low-quality core-wood pieces (juvenile wood) were selected, 12 as control pieces (Ctrl), and 140 for the thermal treatment. From the process, 12 pieces were randomly selected when the 190 °C (T1) procedure was finished and then another 12 pieces when the process reached the temperature of 210 °C (T2). Anatomical measurements were performed at the Wood Anatomy Laboratory of the University of Bio-Bio (Concepción, Chile). Following the methodology described by Chaffey (2002), a cube from each piece was cut and soaked in water for 36 h. A transverse slice of 20 µm was obtained using a Microm Model HM325 microtome (Thermo Fisher Scientific, Tampa, FL, USA) from each one of the 12 cubes, with emphasis paid to the positions of the annual rings when they were cut. Then, 12 slice were stained in safranin and mounted on a glass slide and cover glass using Canada balsam. The wood anatomical structure images were obtained using a Lumenera (Ottawa, Ontario, Canada) Infinity digital camera and analyzed *via* WinCell Pro (Regent Instruments Inc., v.2011a, Québec, Canada).

Wood cells morphological data were measured per annual ring. The exploratory research experiment was designed, considering the intra-ring variability. The experiment involved repetition (measurements on wood cells per annual ring) instead of replicability (measurements on multiple slices). In every slice, tracheid dimensions in the radial (R) and tangential (T) directions were measured in 16 randomly cells (8 early and 8 latewood cells) at the beginning of the annual ring from each sample. Secondary wall thickness (2 R and 2 T measurements) and lumen diameter were evaluated. Length and width were measured as the horizontal and vertical size of the cell in its center of gravity position. From a grayscale image, thresholding were used to produced binary images for enhance cell contrasts when was acquired images (Fig. 1).

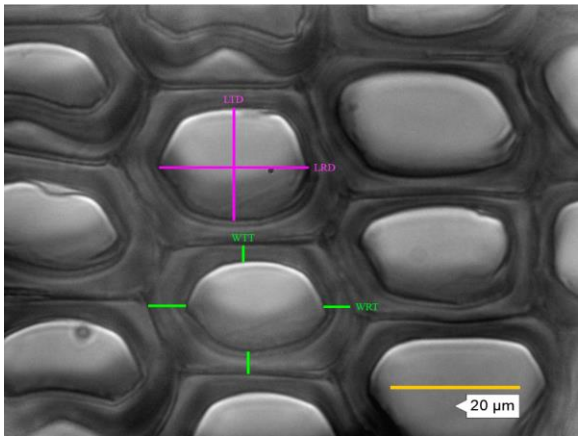

Fig. 1. Unmodified radiata pine (Pinus radiata) wood transverse surface of latewood 40x. Lumen and secondary cell wall thickness measurements of tracheid (LRD: lumen radial diameter; LTD: lumen tangential diameter; WRT: wall radial thickness WTT: wall tangential thickness)

The effect of thermal modification on the microstructure was analyzed relative to the quantity of damage under different process conditions. Every image (magnification for imaging was 10×, and the size of each image was 1468 µm^2) was subdivided into 6 subregions. Manual measurements were performed to determine the proportion (%) of cells with damage (gaps, tracheid wall destruction, cracked areas, cell breakage, and

deformation or distortion of cellular structure) according to the protocols of the WinCell Pro software.

Data analysis

Kolmogorov-Smirnov and Barlett tests were performed to verify the normality of data distribution and homogeneity of variance among treatments. Analysis of variance (ANOVA) testing was performed to analyze the significant differences, and the means were compared between treatments *via* Tukey testing with a 95% confidence level. The software Statistica (Statsoft Inc., v.10.0, Tulsa, OK, USA) was used to perform the statistical analysis.

RESULTS AND DISCUSSION

According to the F-test results of the ANOVA testing presented in Table 1, the treatments had a significant effect in cell wall thickness and lumen diameter of earlywood. For latewood, the cell wall thickness and lumen diameter of the unmodified and thermally modified samples were not statistically different, which indicates that the thermal modification treatment had no significant effect.

Table 1. ANOVA of the Treatment Levels on Cell Wall Thickness and Lumen Diameter

Source	D. F.	Result	Cell Wall Thickness		Lumen Diameter	
			EW	LW	EW	LW
Treatment	2	F-test	20.68	0.88	8.03	0.11
		p-value	< 0.0001*	0.4168	0.0004*	0.8923
Notes: D.F. = degrees of freedom; EW = earlywood; LW = latewood; *significant at p ≤ 0.05						

The average cell wall thicknesses of the earlywood and latewood are shown in Fig. 2. For earlywood (Fig. 2a), the average cell wall thicknesses were 3.55 µm, 3.36 µm, and 3.07 µm for unmodified control samples (Ctrl), 190 °C (T1), and 210 °C (T2), respectively. Cell wall thickness tended to decrease as the temperature of heat treatment increased, which was similar to the results of Biziks *et al*. (2013), who investigated changes in the microstructure of birch wood after hydrothermal treatment at three different temperatures. They found the greatest changes after treatment at 180 °C, which reached an average decrease of 32%. However, treatment T1 (190 °C) did not have a significant effect on cell wall thickness (the difference of means between Ctrl and T1 was not statistically significant). When the temperature was increased to 210 °C, cell wall thickness decreased significantly. The decreases in thickness were 5.3% and 13.5% for T1 and T2, respectively. The average cell wall thicknesses of latewood (Fig. 2b) were 4.92 µm, 4.87 µm, and 4.77 µm for Ctrl, T1, and T2, respectively, and the decrease in thickness was 1.02% for T1 and 3.05% for T2. The difference between treatments was not statistically significant at the 0.05 probability level. Bernabei and Salvatici (2016) examined the effect of heat treatment on the cell wall thickness of *Picea abies* and concluded that, from approximately 100 °C to 200 °C, the dimensions remain almost constant, but a rapid decrease in thickness occurs at temperatures over 200 °C.

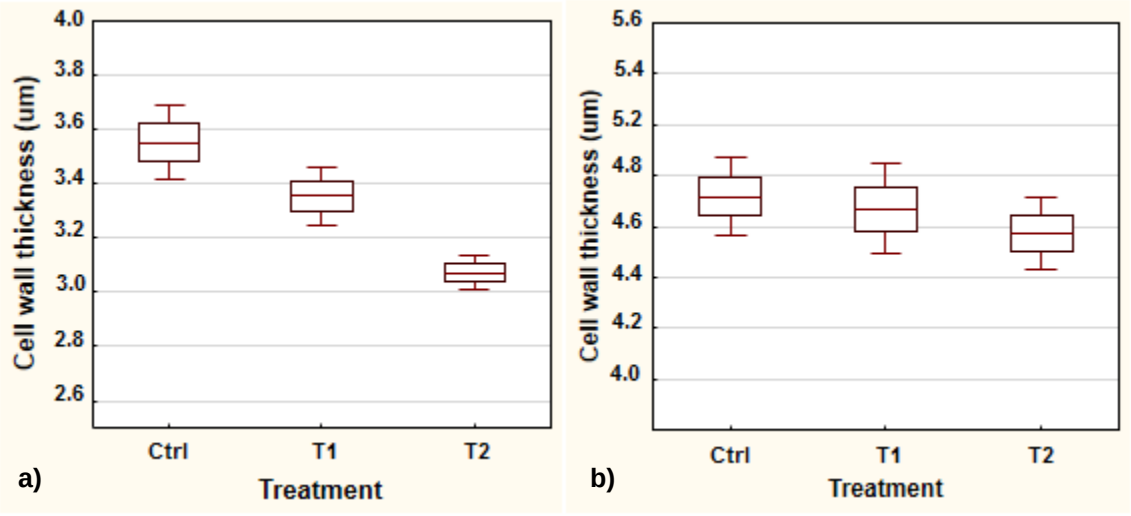

Fig. 2. Average values of cell wall thickness of unmodified control samples and thermally modified wood samples of (a) earlywood and (b) latewood

The results of the lumen diameter measurements showed a slight increase in diameter as temperature was increased (Fig. 3). For earlywood (Fig. 3a), the average lumen diameters were 32.46 µm for Ctrl, 33.54 µm for T1, and 35.09 µm for T2, which corresponded to an increase of 3.3% and 8.1% for T1 and T2, respectively. Kekkonen *et al*. (2010) found similar results, which were attributed to the decomposition of the cell walls.

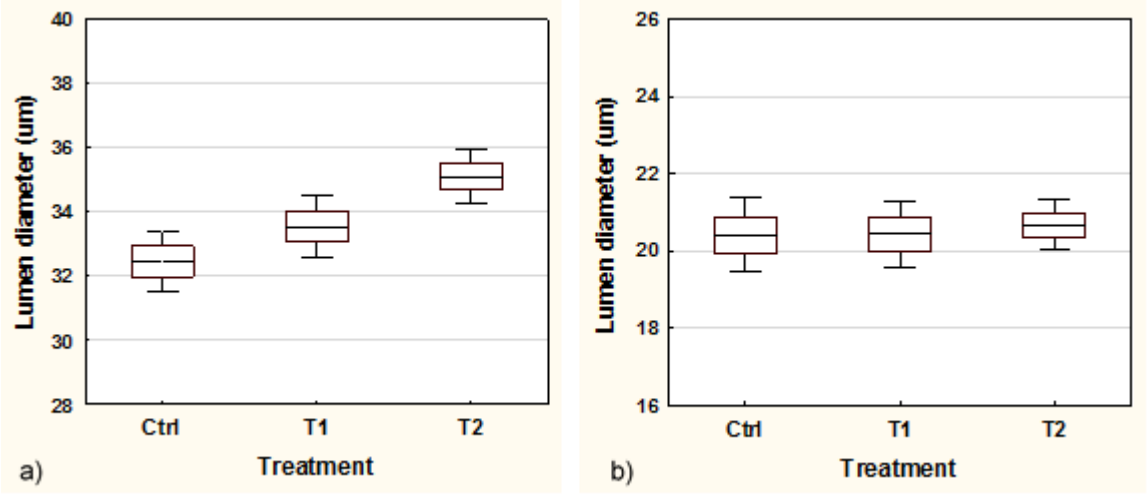

Fig. 3. Average values of lumen diameter of unmodified and thermally modified wood samples of (a) earlywood and (b) latewood

For latewood (Fig. 3b), the values were 20.41 µm, 20.43 µm, and 20.66 µm for Ctrl, T1, and T2, respectively. No significant differences were found between treatments. In all treatments, the difference between earlywood and latewood results were significant ($p < 0.0001$). Similarly, Bernabei and Salvatici (2016) reported average increases of lumen diameter in *Picea abies* wood of 6.2% and 1.8% for earlywood and latewood respectively, during heat treatment at 200 °C.

The anatomical structures of thermally modified and unmodified radiata pine wood are shown in Fig. 4. The structures showed changes from the unmodified state. After

modification at 190 °C, a slight crushing of the cellular lumen in earlywood was observed (Fig. 4b). However, when the temperature was increased to 210 °C, more intense crushing of the cellular lumen was observed (Fig. 4c). In addition, small cracks, broken cells, and cell deformations in some areas on the cell wall of earlywood were observed after modification. In this direction, Boonstra *et al.* 2006a, found radial cracks along the tracheids of early wood in Scots pine, as well as distortion of cell structure has also been reported.

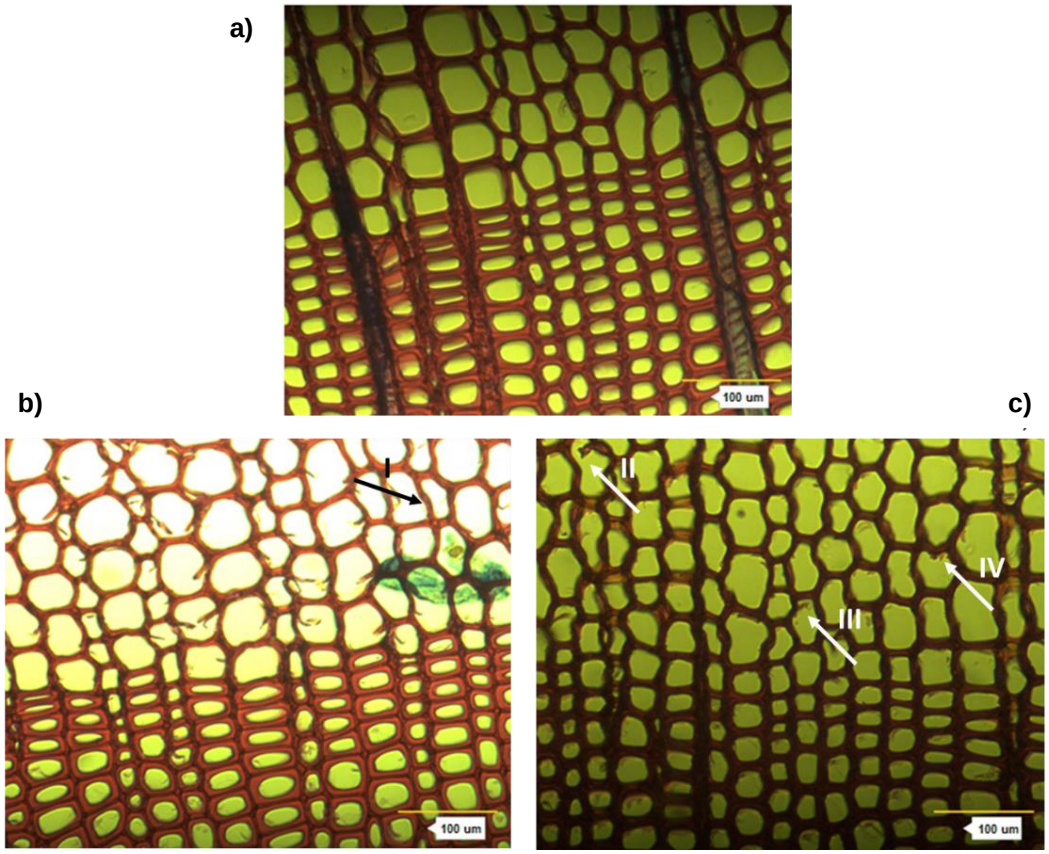

Fig. 4. Anatomical structure of thermally modified and unmodified radiata pine wood, with the arrows pinpointing the structural changes of the thermally modified wood: (a) unmodified wood; (b) thermally modified wood at 190 °C (I: crushing); (c) thermally modified wood at 210 °C (II: broken cells, III: small cracks, IV: cell deformations)

In quantitative terms and based on the measurements and the identified proportions of damaged cells, the damaged area increased as the temperature of thermal treatment increased (Fig. 5). The unmodified wood had a damaged area of 80.2 μm^2. According to Boonstra *et al.* (2016a), the earlywood tracheids of Norway spruce were slightly deformed for unmodified wood. In this context, damaged such as tracheid wall destruction, cracked areas, and cell breakage or deformation of cellular structure could be directly attributable to the preparation of the specimen, boiling and cut that can affect the condition of the wood (Bernabei and Salvatici 2016).

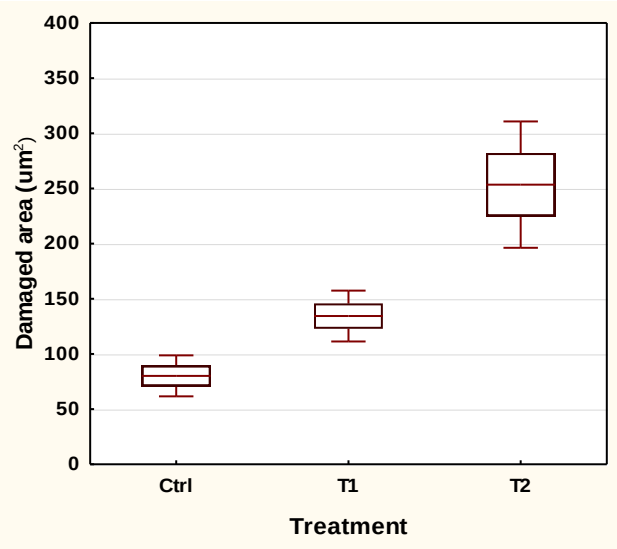

Fig. 5. Damaged area in the anatomical structure of thermally modified radiata pine wood for the unmodified wood (Ctrl), wood modified at 190 °C (T1) and wood modified at 210 °C (T2)

The damaged areas of the thermal treatments at 190 °C and 210 °C reached values of 134.4 μm^2 and 253.5 μm^2, respectively, which corresponded to an increase of 9.2% for T1 and 17.3% for T2. Similar results were found by Kymäläinen *et al*. (2018), who reported that the cell wall was damaged and delaminated at 180°C and Gao (2017), who observed formation of small cracks in latewood tracheids at 220°C.

The difference between Ctrl and T1 was not statistically significant. Hietala *et al*. (2002) have reported similar trends, where the measurements showed no clear change in the cell dimensions of the thermally modified samples compared with control samples.

CONCLUSIONS

1. Thermally modified radiata pine showed signs of damage after the thermal modification process, as small cracks, broken cells, and deformations in the wood cell wall were present after modification. The cell wall thickness decreased and the average lumen diameter increased as the modification temperature increased.
2. The changes in the structure of the modified wood were more noticeable at modifications at 210 °C. This was made evident by an analysis of the damaged area after modification that showed a statistically significant increase at this temperature, which means that the cellular structure was more affected as the modification temperature increased.
3. Further research should focus on defining whether the speed of the increase in temperature to reach the modification temperature could be a factor in the development of the changes in the cellular structure and to further analyze the development of cracks in the wood cell wall structure.

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