

# Effect of Temperature and Clamping during Heat Treatment on Physical and Mechanical Properties of Okan (*Cylicodiscus gabunensis* [Taub.] Harms) Wood

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The objective was to evaluate the effect of temperature and clamping method during heat treatment on the properties of high density Okan wood. Heat treatment of sapwood and heartwood was conducted using an electric oven with a programmable controller at 160 °C, 180 °C, 200 °C, and 220 °C for 2 h. Physical and mechanical properties were compared before and after the heat treatment process. The color change ( $\Delta E^*$ ), weight loss, and volume shrinkage increased with increasing temperature, whereas the equilibrium moisture content (EMC) and water absorption (WA) decreased in both types of wood. The wood density was not affected by temperature, and the magnitude of  $\Delta E^*$  in sapwood was the highest, while the magnitude of weight loss, volume shrinkage, EMC, and WA in heartwood was higher than in sapwood. The clamping method affected  $\Delta E^*$  in heartwood only, while the weight loss, volume shrinkage, EMC, and WA was affect in both types of wood. A significant reduction in the mechanical properties occurred after heat treatment at 200 °C and 220 °C. The reduction of MOR, MOE, and shear strength in heartwood was greater than in sapwood, while the compressive strength reduction in sapwood was the highest.

*Keywords:* Okan wood; Heat treatment; Temperature; Clamping method; Physical and mechanical properties

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## INTRODUCTION

The thermal modification or heat treatment of wood can be defined as the application of heat in order to obtain a desired improvement in the performance of the wood. The heat treatment process is generally performed in the temperature range of 160 to 260 °C, because temperatures lower than 160 °C are known to result in only a slight change in wood properties (Hill 2006). Temperatures that are higher than 260 °C may result in unacceptable degradation of the wood components.

During heat treatment, the arrangement of hydrophilic polymers and plasticization of lignin occurs, resulting in a decrease of dimensional swelling and water absorption (Hakkou *et al.* 2005). Metsa-Kortelainen *et al.* (2006) reported that the heat treatment of Scots pine and Norway spruce heartwood at 170 °C, 190 °C, 210 °C, and 230 °C for 3 h decreased the water absorption, which was influenced by an increase in temperature.

Esteves *et al.* (2013) investigated the effect of heat treatment at 190 °C and 200 °C for 2 to 6 h on the equilibrium moisture content (EMC) and anti-shrinking efficiency. The cited authors found that the EMC decreased by approximately 39% to 42% and the anti-shrinking efficiency increased by approximately 50%.

Heat treatment of wood can be applied as an alternative method for improving the biological durability of the wood, without the use of toxic chemicals. Heat treatment improves the durability of the wood against fungi by the following effects: the increase of the hydrophobic character of the wood, the generation of new extractives that can act as fungicides, modification of the wood polymers leading to enzymes involved in fungal degradation, and a significant degradation of hemicelluloses, which constitute one of the main nutrient sources for the fungi (Kamdem *et al.* 2002; Weiland and Guyonnet 2003; Hakkou *et al.* 2006).

Okan, also known as Denya and African greenheart, is a tree native to west and central Africa, and it generally grows to 60 m tall with straight bole up to 24 m (Kadiri *et al.* 2005). The density of Okan heartwood can reach more than 1.0 g/cm<sup>3</sup>, and its wear resistance is generally high; therefore, this wood is commercially used for heavy-duty flooring in factories and warehouses. Studies on the heat treatment of high density Okan wood are limited. Most of the previous studies have discussed the heat treatment of low to medium density woods (Byeon *et al.* 2010; Kim *et al.* 2012; Won *et al.* 2012; Kariz *et al.* 2013; Todaro *et al.* 2013; Xing and Li 2014; Zanuncio *et al.* 2014; Aytin *et al.* 2015; Zhang *et al.* 2015).

Previous studies on the heat treatment of high density as Okan wood have been very limited and only discussed some properties such as color properties (Shi *et al.* 2011; Shi and Jiang 2011). Shi *et al.* (2011) investigated the effect of heat treatment of Okan on its color properties, and Shi and Jiang (2011) investigated the color stability of heat-treated Okan sapwood during artificial weathering. In this article, we discuss properties of Okan wood stacked with a metal clamp during the heat treatment process. Our previous study showed that this method successfully reduced the drying defects in the wood (Hidayat *et al.* 2015). However, the effect of the clamping method on the physical and mechanical properties of Okan wood has not been studied. Therefore, the objective of this study was to evaluate the effect of temperature and clamping method on the physical and mechanical properties of Okan wood.

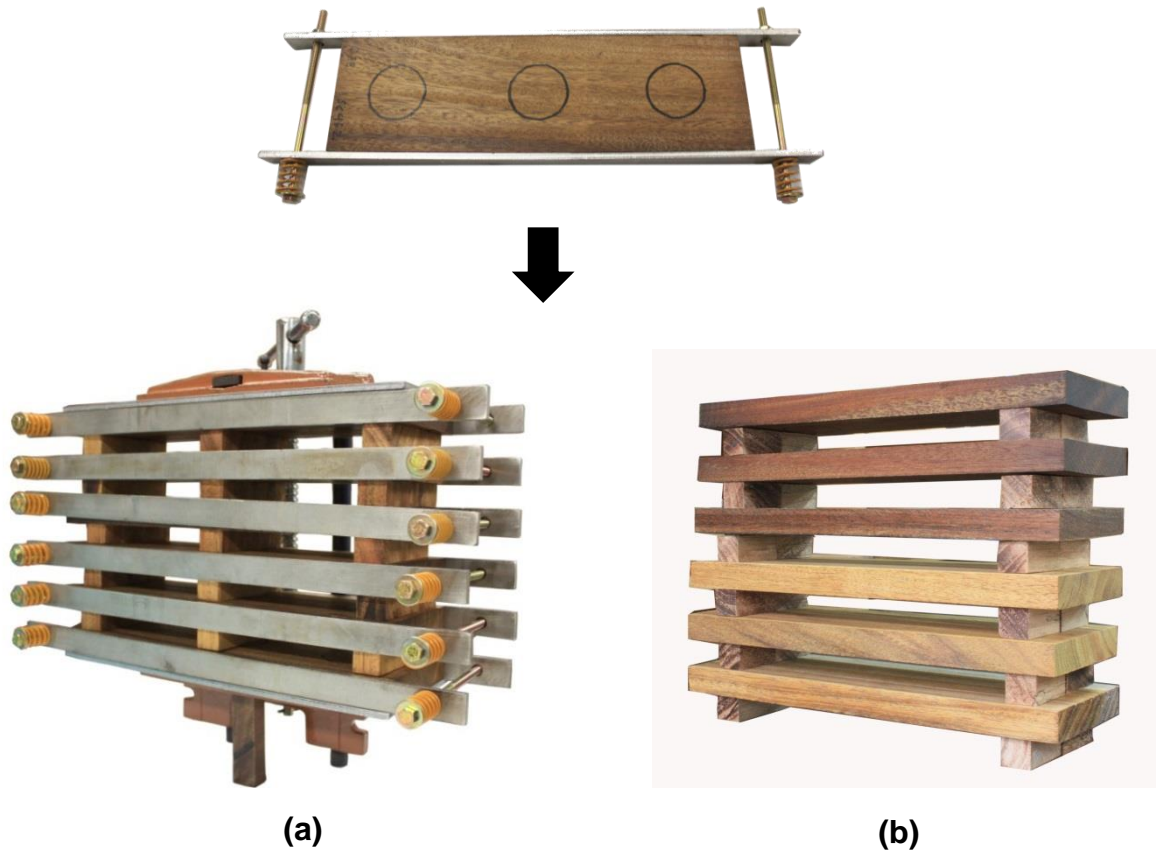
## EXPERIMENTAL

### Materials

Flat-sawn boards from sapwood and heartwood of Okan (*Cylicodiscus gabunensis* (Taub.) Harms) were prepared for heat treatment. The dimensions of the specimens were 300 mm x 90 mm x 20 mm (length x width x thickness). Boards with a small variation in density and free of defects were selected. Then, the boards were kept in a conditioning room under the relative humidity of 65 ± 3% and a temperature of 25 ± 2 °C for 2 weeks before heat treatment.

The air-dry densities of the sapwood and heartwood specimens ranged from 0.77 to 0.89 g/cm<sup>3</sup> and 1.16 to 1.23 g/cm<sup>3</sup>, respectively. Each board was clamped edge-wise using flat metal, and fastened using bolts and nuts equipped with metal springs. A total six clamped boards, *i.e.*, three sapwood and three heartwood, were stacked in a vertical clamp device. Wood stickers with the dimension of 80 mm x 20 mm x 20 mm, respectively, were

put between the boards. The vertical clamp was then fastened. For comparison, another set of samples were stacked without the clamps.



**Fig. 1.** Stacking of the samples during the heat treatment: (a) with the clamps and (b) without the clamps

## Methods

### *Heat treatment*

Heat treatment can be performed in air, in a vacuum, or under an inert atmosphere (Hill 2006). In this study, the heat treatment was performed under low presence of oxygen in an electric oven with a programmable controller (L-Series, JEIO TECH Ltd., Korea). The heat treatment began at the initial temperature of  $25 \pm 5$  °C and then was raised to the target temperatures of 160 °C, 180 °C, 200 °C, and 220 °C, with a heating rate of 2 °C/min. The target temperatures were maintained for 2 h. In the final stage of heat treatment, the oven chamber was allowed to cool naturally until they reached 30 °C. Then, the boards were taken out and kept in a conditioning room under the relative humidity of  $65 \pm 3\%$  and a temperature of  $25 \pm 2$  °C for 2 weeks until further testing.

### *Board evaluation*

The dimensions of the specimens for the evaluation of the physical and mechanical properties are summarized in Table 1. The colorimetric evaluation was performed using the CIE- $L^*a^*b^*$  system (Commission Internationale de l'Éclairage, Vienna, Austria). Three measurements of each specimen were collected before and after heat treatment. These measurements were taken using a Konica Minolta CR-400 Chroma Meter to obtain the  $L^*$ ,  $a^*$ , and  $b^*$  color values.

The color change ( $\Delta E^*$ ) was calculated as follows (Eq. 1),

$$\Delta E^* = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2} \quad (1)$$

where,  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ , and  $\Delta E^*$  represent the changes in lightness, red/green chromaticity, yellow/blue chromaticity, and the overall color, respectively.

**Table 1.** Dimensions of the Okan Specimens

Test	Dimensions of specimens (mm)		
	Radial	Tangential	Longitudinal
Color, weight loss, volume shrinkage, and air-dry density	20	90	300
Moisture content, water absorption	20	20	40
Static bending strength	20	20	300
Compression strength perpendicular to the grain*	20	10	10
Shear strength parallel to the grain	20	20	40

\* Specimen dimensions were modified with a proportional ratio to the standard dimensions according to the KS F 2206 standard (40 mm x 20 mm x 20 mm), due to board limitations.

The amount of weight loss (WL) after heat treatment was calculated with the formula,

$$WL (\%) = 100 \times (m_b - m_h) / m_b \quad (2)$$

where  $m_b$  and  $m_h$  represent the air-dry weight of the specimens before and after the heat treatment, respectively. Volume shrinkage (VS) was performed in accordance with the Korean standard KSA KS F 2203 (2009) and calculated using the equation,

$$VS (\%) = 100 \times (V_b - V_h) / V_b \quad (3)$$

where  $V_b$  and  $V_h$  represent the volume of the specimens before and after the heat treatment, respectively.

The air-dry density of the control and heat-treated specimens were determined by measuring their air-dry weight and volume in accordance with KS F 2198 standard (2011). Moisture content (MC) and water absorption (WA) were performed in accordance with KS F 2199 (2011) and KS F 2204 (2009), respectively. To measure moisture content (MC) and water absorption (WA) of control, six untreated boards were randomly selected, and a specimen with dimension of 20 x 20 x 40 mm<sup>3</sup> was prepared from each board. Six specimens for MC and WA measurement were also prepared from heat treated samples. MC and WA of control and heat treated samples were calculated using following equations,

$$MC (\%) = 100 \times (m_a - m_o) / m_o \quad (4)$$

$$WA (\%) = 100 \times (m_w - m_a) / m_a \quad (5)$$

where  $m_a$  and  $m_o$  represent air-dry weight and oven-dry weight of the specimens, respectively, and  $m_w$  represent weight of specimens after 2 weeks of water immersion.

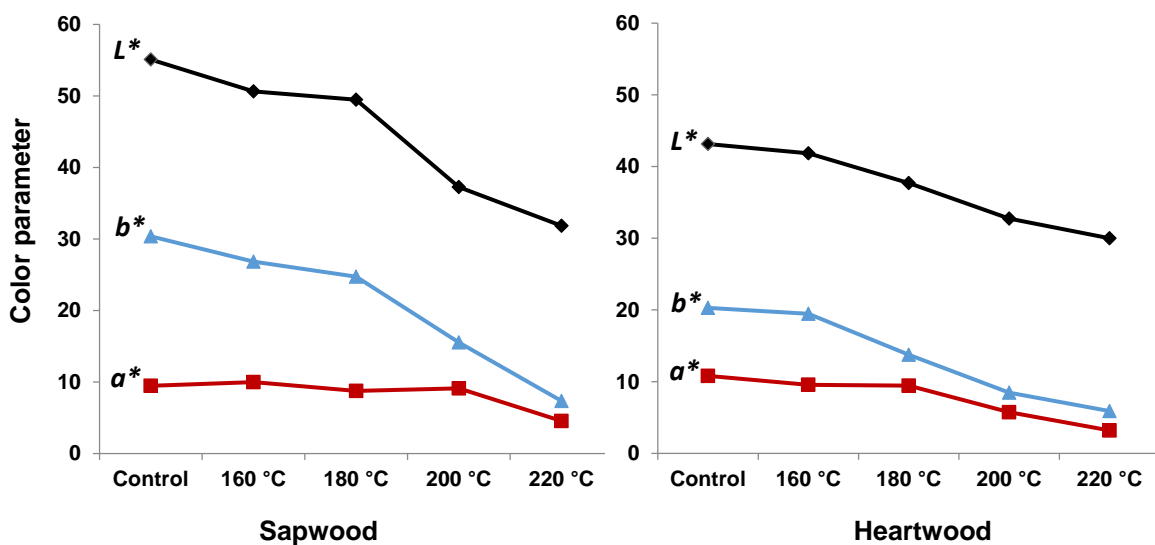
The modulus of rupture (MOR) and the modulus of elasticity (MOE) were determined by a 3-point static bending method using a universal testing machine (Model 4482, Instron, Norwood, MA, USA). The loading speed and the span length were 1.5 mm/s and 200 mm, respectively. The compression strength and the shear strength were performed in accordance with the Korean standards, KSA KS F 2206 (2009) and KSA KS F 2209 (2009).

The experimental design was a completely randomized factorial design. The results of all parameters tested were first evaluated for an overall analysis of variance (ANOVA). Then, the significant differences between the means for the control and the treated specimens were compared using Tukey's honestly significant difference (HSD) test, and the significance was accepted at  $P < 0.05$ .

## RESULTS AND DISCUSSION

### Color Changes

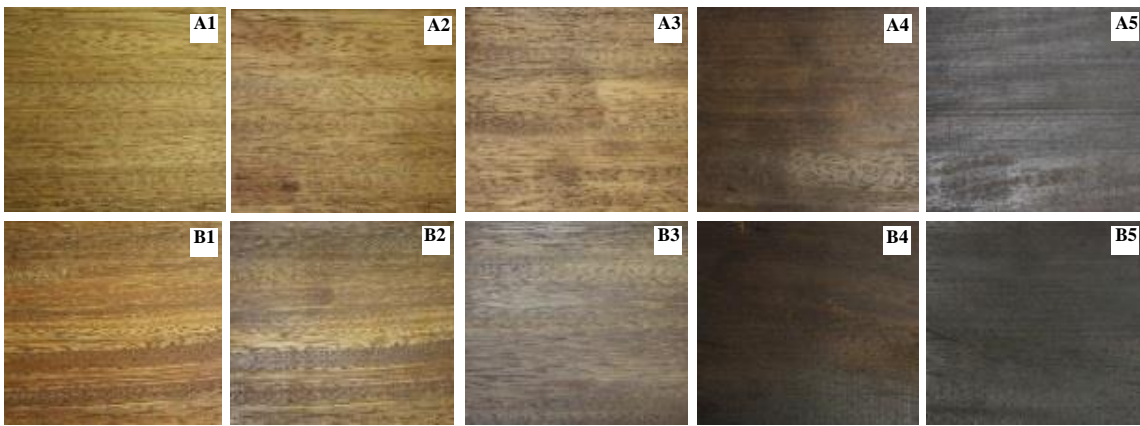
Wood color is one of the most important aesthetic aspects of wooden products. The color of wood differs not only between various wood species, but also within one species, and often within one piece of wood (Panshin and de Zeeuw 1980). The untreated Okan sapwood has a light yellow color, while heartwood has a darker yellow-brown color. The sapwood exhibited an average lightness ( $L^*$ ) value of 56.7, compared to heartwood with a  $L^*$  value of 44.5. Figure 2 shows the effect of temperature during the heat treatment using clamp on the color of sapwood and heartwood. In sapwood, increasing the temperature decreased the lightness ( $L^*$ ) and the yellow-blue chromaticity ( $b^*$ ). A similar trend as in sapwood was obtained in heartwood. The red-green chromaticity ( $a^*$ ) in sapwood was not much affected by temperature between 160 to 200 °C, after heat treatment at more than 200 °C the  $a^*$  value was obviously reduced.



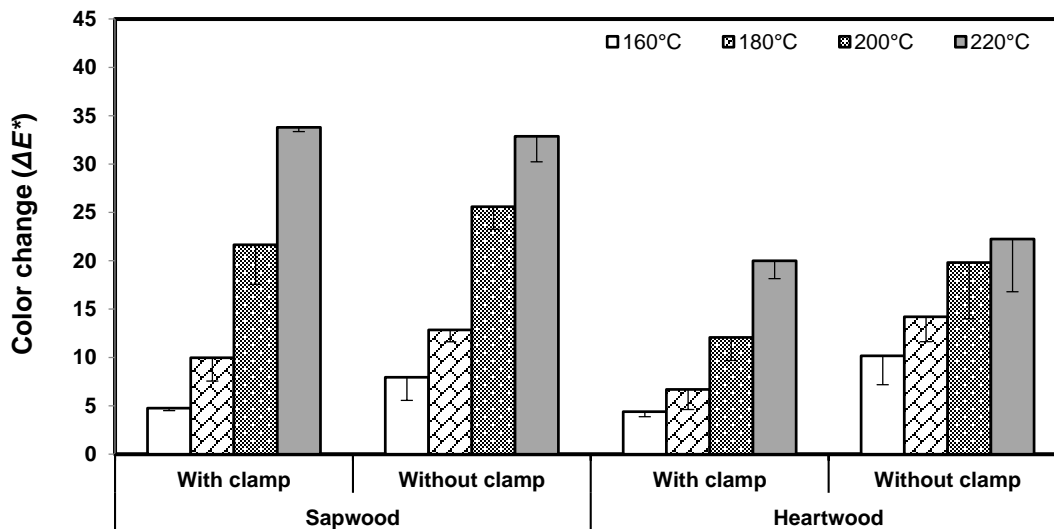
**Fig. 2.** Color parameter ( $L^*$ ,  $a^*$ ,  $b^*$ ) of Okan after heat treatment using clamp at different temperatures

Figure 3 shows the appearance of sapwood and heartwood before and after the heat treatment process at different temperatures. Darkening, as a result of heat treatment was clearly visible and it increased with increasing temperature.

Figure 4 shows the effect of the clamping method during heat treatment on color change ( $\Delta E^*$ ) in sapwood and heartwood. The  $\Delta E^*$  in sapwood slightly increased from 160 °C to 180 °C, and increased drastically from 180 °C to 200 °C. However, in heartwood, the  $\Delta E^*$  increased more gradually over the entire temperature range. This result is in a good agreement with the findings of Bekhta and Niemz (2003), who observed that the color of wood changes drastically after heat treatment at 200 °C. This implies that temperatures ranging from 180 to 200 °C are significantly critical to the change in color during the heating process.



**Fig. 3.** The appearance of sapwood (A) and heartwood (B) of Okan before and after heat treatment using clamping at different temperatures: (1) Control, (2) 160 °C, (3) 180 °C, (4) 200 °C, (5) 220 °C



**Fig. 4.** Effect of the clamping method on the color change ( $\Delta E^*$ ) during heat treatment in sapwood and heartwood of Okan

Overall, the  $\Delta E^*$  of sapwood was higher than that of heartwood. The highest  $\Delta E^*$  was 33.8 for sapwood and 22.2 for heartwood, after the heat treatment at 220 °C. Results of this study were compatible with those from other studies. For example, Shi *et al.* (2011) stated that heat treatment resulted in a darkening effect of the wood tissue and that the color became dark as the temperature increased. Their study also revealed that the effect of the treatment temperature on a color change in sapwood was more obvious than in heartwood. During heat treatment, the oxidation of phenolic compounds, the presence of reduced sugars and amino acids, the emanation of formaldehydes, the formation of quinines, or the caramelization of holocellulose components attributed the color change (Sundqvist 2002; Boonstra 2008; Sandoval-Torres *et al.* 2010). Clamping during heat treatment might prevented the oxidation of wood compound resulted in lower value color change. The  $\Delta E^*$  values of the heartwood specimens using the clamp method were lower compared to those without the clamps. In sapwood, the  $\Delta E^*$  values of samples with clamp were slightly lower compared to without clamp.

### Physical Properties

Table 2 summarizes the changes in the physical properties of Okan wood before and after heat treatment and with or without the clamp method, at different temperatures. Weight loss and volume shrinkage were significantly affected by temperature in both the sapwood and heartwood specimens. Weight loss values increased linearly as the temperature increased. The volume shrinkage did not always increase with increasing temperature. Consequently, the volume shrinkage value increased within the temperature range of 160 °C to 180 °C, then decreased slightly from 180 °C to 200 °C, and increased again from 200 °C to 220 °C. Compared to sapwood, heartwood showed a higher degree of weight loss and volume shrinkage. This might due to density difference and higher extractive content in heartwood compared to sapwood. The extractives in the wood degrade more easily, and these compounds evaporate from the wood during the heat treatment.

The clamping method also affected the amount of weight loss and volume shrinkage. The weight loss of sapwood and heartwood with the clamps at 160 to 200 °C was lower than that of the specimens without the clamps. However, for heat treatment at 200 °C, the samples with the clamps had a higher weight loss percentage. The effect of clamping on the volume shrinkage was consistent for each temperature; the volume shrinkage of the specimens with the clamps was higher than that of the specimens without the clamps.

The reduction of weight and volume during heat treatment at higher temperatures than 160 °C are generally known because of the degradation of extractives, hemicelluloses, and a part of cellulose molecules in the amorphous regions (Esteves and Pereira 2009). The degradation causes a chemical change in the wood. In other words, the basic components of the wood cell wall structure are changed in their number and dimension, leading to a reduction in the dimension and weight of the wood after the heat treatment.

The density of the Okan wood before and after heat treatment at all temperatures seemed to be unchanged. Statistical analysis also revealed that the density change in sapwood and heartwood was not affected by the temperature or the clamping method. This may have occurred because of a proportional reduction in weight and volume during the heat treatment process.

**Table 2.** Effect of Temperature and Clamping Method on the Physical Properties of Sapwood and Heartwood

Wood Part	Temp. (°C)	Weight loss (%)		Volume shrinkage (%)		Density (g/cm <sup>3</sup> )		EMC (%)		Water Absorption (%)	
		With clamp	Without clamp	With clamp	Without clamp	With clamp	Without clamp	With clamp	Without clamp	With clamp	Without clamp
Sapwood	Control	-	-	-	-	0.88 <sup>A</sup> (0.03)	0.78 <sup>A</sup> (0.07)	8.99 <sup>A</sup> (0.03)	8.99 <sup>A</sup> (0.03)	32.18 <sup>A</sup> (2.57)	32.18 <sup>A</sup> (2.57)
	160	6.04 <sup>A</sup> (0.60)	7.85 <sup>A</sup> (0.44)	2.62 <sup>A</sup> (0.40)	3.12 <sup>A</sup> (0.58)	0.84 <sup>A</sup> (0.00)	0.74 <sup>A</sup> (0.08)	6.98 <sup>B</sup> (0.29)	6.57 <sup>B</sup> (0.28)	29.62 <sup>A</sup> (2.90)	32.08 <sup>A</sup> (2.95)
	180	7.18 <sup>AB</sup> (0.83)	7.92 <sup>A</sup> (0.67)	4.17 <sup>B</sup> (0.95)	2.83 <sup>A</sup> (0.66)	0.85 <sup>A</sup> (0.05)	0.74 <sup>A</sup> (0.09)	6.74 <sup>B</sup> (0.11)	5.39 <sup>C</sup> (0.20)	26.81 <sup>AB</sup> (1.90)	29.55 <sup>A</sup> (2.18)
	200	8.28 <sup>B</sup> (0.56)	8.66 <sup>AB</sup> (0.64)	3.94 <sup>B</sup> (0.19)	3.45 <sup>A</sup> (0.49)	0.84 <sup>A</sup> (0.02)	0.75 <sup>A</sup> (0.09)	5.12 <sup>C</sup> (0.25)	4.45 <sup>D</sup> (0.42)	21.46 <sup>B</sup> (2.61)	24.36 <sup>B</sup> (2.36)
	220	10.39 <sup>C</sup> (0.83)	9.90 <sup>B</sup> (0.96)	6.81 <sup>C</sup> (0.89)	4.60 <sup>B</sup> (0.70)	0.86 <sup>A</sup> (0.04)	0.74 <sup>A</sup> (0.07)	4.50 <sup>D</sup> (0.04)	4.09 <sup>D</sup> (0.26)	18.91 <sup>C</sup> (1.89)	21.31 <sup>AB</sup> (2.63)
Heartwood	Control	-	-	-	-	1.20 <sup>A</sup> (0.03)	1.16 <sup>A</sup> (0.03)	9.68 <sup>A</sup> (0.14)	9.68 <sup>A</sup> (0.14)	13.74 <sup>A</sup> (1.00)	13.74 <sup>A</sup> (1.00)
	160	5.59 <sup>A</sup> (2.20)	7.54 <sup>A</sup> (0.35)	4.08 <sup>A</sup> (0.35)	4.70 <sup>A</sup> (0.06)	1.18 <sup>A</sup> (0.01)	1.13 <sup>A</sup> (0.05)	7.05 <sup>B</sup> (0.59)	6.23 <sup>B</sup> (0.42)	10.61 <sup>B</sup> (1.01)	12.16 <sup>A</sup> (1.30)
	180	6.87 <sup>A</sup> (1.01)	8.75 <sup>AB</sup> (1.34)	5.48 <sup>B</sup> (0.17)	5.13 <sup>AB</sup> (0.60)	1.18 <sup>A</sup> (0.04)	1.11 <sup>A</sup> (0.04)	6.56 <sup>BC</sup> (0.17)	6.80 <sup>BC</sup> (0.25)	9.71 <sup>B</sup> (1.85)	11.76 <sup>AB</sup> (0.51)
	200	8.09 <sup>AB</sup> (1.13)	9.87 <sup>BC</sup> (0.38)	5.22 <sup>B</sup> (0.34)	5.13 <sup>AB</sup> (0.40)	1.20 <sup>A</sup> (0.03)	1.10 <sup>A</sup> (0.05)	5.61 <sup>C</sup> (0.44)	5.62 <sup>BC</sup> (1.20)	7.84 <sup>C</sup> (1.01)	9.41 <sup>B</sup> (0.84)
	220	11.26 <sup>B</sup> (0.65)	11.21 <sup>C</sup> (1.16)	7.06 <sup>C</sup> (0.49)	6.66 <sup>C</sup> (0.92)	1.12 <sup>A</sup> (0.05)	1.10 <sup>A</sup> (0.03)	4.11 <sup>D</sup> (0.21)	4.69 <sup>C</sup> (0.29)	6.58 <sup>C</sup> (0.89)	7.95 <sup>BC</sup> (1.01)

Notes: Means within a column followed by the same capital letter are not significantly different at 5% significance level using Tukey's HSD test. Numbers in parenthesis are standard deviations. Means are average of 3 replications.



In contrast to weight loss and volume shrinkage, the equilibrium moisture content EMC and water absorption (WA) decreased with increasing temperature of treatment. This decrease may have been a result of the increase in hydrophobicity of the cell wall as a result of a decrease in the number of hydroxyl groups by the chemical reactions occurring during the heat treatment, resulting in less water absorption (Jämsä and Viitaniemi 2001).

Overall, the reduction of the EMC and WA in heartwood was higher than in sapwood during the heating process. The results are in agreement with the increase of weight loss in heartwood which was higher than that in sapwood. However, no significant differences were found between the EMC values of sapwood and heartwood. Similar results were reported by Metsä-Kortelainen (2006).

The clamping method also affected the EMC and WA. The heartwood with the clamps exhibited a slightly lower EMC than the heartwood without clamping. However the use of clamp in sapwood samples did not improve EMC as shown by higher EMC values compared to without clamping. The sapwood and heartwood with the clamps tended to have a lower WA, compared to non-clamping. The drying set may have occurred because of the compression on both the radial sections by clamping. The occurrence of the drying set may prevent water intake from the radial direction, resulting in a lower WA value.

### Mechanical Properties

As discussed in the previous section, the EMC decreased with increasing heating temperature. Ross (2010) and Shmulsky and Jones (2011) both demonstrated that a decrease in the EMC exhibited a positive effect on the mechanical properties of the wood. However in this study, the mechanical properties of the Okan wood before and after the heat treatment at different temperatures were investigated after acclimatization in the same environmental condition. This option was considered to be the best for this practical evaluation, as the timber in service will be in hygroscopic equilibrium with the environment.

Table 3 shows the effect of the temperature and the clamping method during heat treatment on the MOR, MOE, compressive strength, and shear strength of sapwood and heartwood specimens. The MOR and MOE decreased with increasing temperature in both the sapwood and heartwood specimens. In particular, both values remarkably decreased after the heat treatment at 200 °C. Similar results were reported by Poncsak *et al.* (2006). The authors reported that the MOR decreased as the treatment temperature increased, yet significantly decreased after 200 °C.

The reduction in the MOR and MOE of sapwood and heartwood with clamping was lower than without clamping at an overall temperature range. This is because in both hardwood and sapwood, samples with clamping exhibited lower weight loss values compared to without clamping. The highest reduction in the MOR was 37.5% and 49.0% for heartwood, with and without the clamps, respectively. The highest reduction in the MOE was 22.2% and 27.0% for heartwood, with and without the clamps, respectively. In the case of sapwood, the highest reductions in the MOR after heat treatment with and without clamping was 29.2% and 31.6% respectively, while the highest reduction in MOE after heat treatment with and without the clamps was 12.4% and 15.3%, respectively.

**Table 3.** Effect of Temperature and Clamping Method on Mechanical Properties in Sapwood and Heartwood of Okan

Wood Part	Temp. (°C)	MOR (N/mm <sup>2</sup> )		MOE (N/mm <sup>2</sup> )		Compressive strength (N)		Shear strength (N)	
		With clamp	Without clamp	With clamp	Without clamp	With clamp	Without clamp	With clamp	Without clamp
Sapwood	Control	120 <sup>A</sup> (7)	117 <sup>A</sup> (8)	10,740 <sup>A</sup> (441)	9,723 <sup>A</sup> (515)	20.15 <sup>A</sup> (1.48)	18.36 <sup>A</sup> (1.76)	32.18 <sup>A</sup> (4.69)	31.41 <sup>A</sup> (2.29)
	160	118 <sup>A</sup> (8)	109 <sup>A</sup> (8)	10,476 <sup>AB</sup> (549)	9,431 <sup>AB</sup> (483)	19.06 <sup>A</sup> (1.53)	16.64 <sup>AB</sup> (2.36)	29.39 <sup>A</sup> (3.95)	28.12 <sup>A</sup> (1.45)
	180	110 <sup>A</sup> (9)	100 <sup>AB</sup> (13)	9,963 <sup>AB</sup> (351)	9,373 <sup>ABC</sup> (357)	18.02 <sup>A</sup> (2.48)	16.05 <sup>AB</sup> (0.68)	29.89 <sup>A</sup> (7.71)	29.28 <sup>A</sup> (1.84)
	200	102 <sup>AB</sup> (10)	95 <sup>AB</sup> (9)	9,837 <sup>AB</sup> (588)	8,307 <sup>BC</sup> (289)	16.66 <sup>A</sup> (2.67)	13.74 <sup>BC</sup> (0.97)	24.37 <sup>B</sup> (4.39)	25.53 <sup>AB</sup> (3.74)
	220	85 <sup>B</sup> (10)	80 <sup>B</sup> (9)	9,403 <sup>B</sup> (460)	8,234 <sup>C</sup> (465)	16.15 <sup>A</sup> (1.42)	10.96 <sup>C</sup> (1.38)	25.42 <sup>B</sup> (4.17)	20.73 <sup>B</sup> (3.19)
Heartwood	Control	192 <sup>A</sup> (14)	200 <sup>A</sup> (23)	15,573 <sup>A</sup> (578)	15,610 <sup>A</sup> (670)	48.27 <sup>A</sup> (3.18)	47.67 <sup>A</sup> (1.59)	32.49 <sup>A</sup> (3.39)	34.18 <sup>A</sup> (4.83)
	160	185 <sup>A</sup> (13)	191 <sup>A</sup> (35)	15,015 <sup>A</sup> (834)	15,387 <sup>A</sup> (515)	46.73 <sup>A</sup> (3.86)	45.13 <sup>A</sup> (2.19)	31.79 <sup>A</sup> (5.59)	32.38 <sup>A</sup> (7.49)
	180	169 <sup>AB</sup> (37)	165 <sup>AB</sup> (41)	15,301 <sup>A</sup> (686)	14,564 <sup>AB</sup> (723)	46.02 <sup>AB</sup> (4.32)	44.77 <sup>A</sup> (2.18)	28.29 <sup>A</sup> (4.29)	33.94 <sup>A</sup> (6.35)
	200	156 <sup>AB</sup> (21)	150 <sup>AB</sup> (7)	13,811 <sup>AB</sup> (594)	13,329 <sup>B</sup> (832)	39.96 <sup>B</sup> (3.41)	41.79 <sup>B</sup> (3.73)	20.24 <sup>B</sup> (6.52)	27.49 <sup>AB</sup> (4.15)
	220	120 <sup>B</sup> (6)	102 <sup>B</sup> (15)	12,117 <sup>B</sup> (636)	11,387 <sup>C</sup> (760)	35.75 <sup>B</sup> (4.88)	34.03 <sup>B</sup> (2.67)	18.43 <sup>B</sup> (8.12)	17.64 <sup>B</sup> (3.53)

Notes: Means within a column followed by the same capital letter are not significantly different at 5% significance level using Tukey's HSD test. Numbers in parenthesis are standard deviations. Means are average of 3 replications.

The highest reductions in the MOR and MOE in both heartwood and sapwood specimens were obtained after heat treatment at 220 °C. The decreasing degree of both values were higher in heartwood than in sapwood. These results were comparable with the increase in weight loss values, *i.e.*, the heartwood lost more weight compared to the sapwood during the heat treatment process. Poncsak *et al.* (2006) stated that the MOR reduction of hardwood during heat treatment probably occurred because of the degradation of the hemicelluloses and cellulose. Awoyemi and Jones (2011) also stated that the degradation of the hemicellulose was primarily responsible for the reduction in weight and density, resulting in the decrease of mechanical properties. The MOR and MOE reductions of sapwood and heartwood specimens after the heat treatment at 160 to 200 °C were less than 25%. Esteves *et al.* (2013) reported that a reduction of mechanical properties of less than 30% is acceptable for most practical applications, such as cladding, sound barriers, or decking. According to this report, Okan treated at 160 to 200 °C could be applied for practical use; however, Okan treated at 220 °C might be not suitable for structural applications because of a high reduction in mechanical strength.

The compressive strength decreased with increasing temperatures. Treatments in the range 160 to 180°C did not significantly reduce the compressive strength; however, treatments above 200 °C did reduce the compressive strength. The highest reduction in the compressive strength for sapwood after heat treatment with and without the clamps was 19.8% and 40.3%, respectively, while for the heartwood after heat treatment with and without the clamps was 25.9% and 28.6%, respectively. The greatest reductions in strength were obtained at 220 °C. The reduction in the compressive strength was highest for sapwood. The clamping method affected the compressive strength for sapwood, but did not affect the heartwood due to density difference between samples with and without clamp in sapwood was more obvious than that in heartwood.

The shear strength showed a similar tendency as that of the compressive strength. The shear strength decreased with increasing temperatures, and significantly decreased after 200 °C. The highest reduction in the shear strength was in the sapwood with and without clamping (21.0% and 34.0%, respectively), whereas the heartwood with and without clamping was 43.3% and 48.4%, respectively. The greatest reductions in strength were obtained at 220 °C. The results showed that the reduction of the shear strength in heartwood was highest. The clamping method negatively affected the shear strength after the heat treatment at 180 °C and 200 °C in both the sapwood and heartwood specimens; no positive effects were shown at 160 °C and 220 °C.

## CONCLUSIONS

1. Increasing the heat treatment temperature increased the  $\Delta E^*$ , weight loss, and volume shrinkage, and decreased the EMC, WA, MOR, MOE, compressive strength, and shear strength. A significant decrease in the mechanical properties was obtained after heat treatment at 200 °C and 220 °C.
2. A color change in the sapwood was more obvious than in the heartwood, whereas the magnitude of weight loss, volume shrinkage, EMC, WA, and the reduction of MOR, MOE, and shear strength in heartwood were higher than in sapwood.

3. The clamping method contributed positively to the physical and mechanical properties of the wood. However, the clamping method decreased the EMC and WA, and decreased the reduction of MOR, MOE, compressive strength, and shear strength.
4. This study showed the feasibility of heat treatment using the clamping method for high density wood of Okan to improve the physical and mechanical properties.

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## REFERENCES CITED

- Awoyemi, L., and Jones, I. P. (2011). "Anatomical explanations for the changes in properties of western red cedar (*Thuja plicata*) wood during heat treatment," *Wood Sci. Tech.* 45(2), 261-267. DOI: 10.1007/s00226-010-0315-9
- Aytin, A., Korkut, S., Ünsal, Ö., and Çakıcıer, N. (2015). "The effects of heat treatment with the ThermoWood® method on the equilibrium moisture content and dimensional stability of wild cherry wood," *BioResources* 10(2), 2083-2093. DOI: 10.15376/biores.10.2.2083-2093
- Bekhta, P., and Niemz, P. (2003). "Effect of high temperature on the change in colour, dimensional stability and mechanical properties of spruce wood," *Holzforchung* 57(5), 539-546. DOI: 10.1515/HF.2003.080
- Boonstra, M. J. (2008). "A two-stage thermal modification of wood," Ph.D. dissertation in cosupervision Ghent University and Universite' Henry Poincare', Nancy 1.
- Byeon, H.- S., Park, J.- H., Hwang, K.- K., Park, H.- M., Park, B.- S., and Chong, S.- H. (2010). "Sound absorption property of heat-treated wood at a low temperature and vacuum conditions," *J. Korean Wood Sci. & Tech.* 38(2), 101-107. DOI: 10.5658/WOOD.2010.38.2.101
- Esteves, B. M., and Pereira, H. (2009). "Wood modification by heat treatment: A review," *BioResources* 4(1), 340-404. DOI: 10.15376/biores.4.1.340-404
- Esteves, B. M., Nunes, L., Domingos, I., and Pereira, H. (2013). "Comparison between heat treated sapwood and heartwood from *Pinus pinaster*," *Euro. J. Wood Prod.* 72(1), 53-60. DOI: 10.1007/s00107-013-0751-y
- Hakkou, M., Pétrissans, M., Zoulalian, A., and Gérardin, P. (2005). "Investigation of wood wettability changes during heat treatment on the basis of chemical analysis," *Polym. Degrad. Stab.* 89(1), 1-5. DOI:10.1016/j.polymdegradstab.2004.10.017
- Hakkou, M., Pétrissans, M., Gérardin, P., and Zoulalian, A. (2006). "Investigations of the reasons for fungal durability of heat-treated beech wood," *Polym. Degrad. Stab.* 91(2), 393-397. DOI: 10.1016/j.polymdegradstab.2005.04.042

- Hidayat, W., Jang, J. H., Park, S. H., Febrianto, F., and Kim, N. H. (2015). "Drying defects of Okan wood (*Cylicodiscus gabunensis*) heat-treated at different temperatures," in: *Proceedings from the 65<sup>th</sup> Annual Meeting of the Japan Wood Research Society*, Tokyo, Japan, pp. 144.
- Hill, C. A. S. (2006). "Wood Modification: Chemical, Thermal and Other Processes," *Wiley Series in Renewable Resources*, John Wiley & Sons, Ltd., West Sussex, England.
- Jämsä, S., and Viitaniemi, P. (2001). "Heat treatment of wood: Better durability without chemicals," in: *Proceedings of special seminar held in Antibes, France*.
- Kadiri, A. B., Olowokudejo, J. D., and Ogunipe, O. T. (2005). "Some aspects of foliar epidermal morphology of *Cylicodiscus gabunensis* (Taub.) Harms (*Mimosaceae*)," *J. Sci. Res. Dev.* 10(2005), 33-38.
- Kamdem, D., Pizzi, A., and Jermannaud, A. (2002). "Durability of heat-treated wood," *Holz. Roh-Werkst.* 60(1), 1-6. DOI: 10.1007/s00107-001-0261-1
- Kariz, M., Kuzman, M. K., and Sernek, M. (2013). "The effect of heat treatment on the withdrawal capacity of screws in spruce wood," *BioResources* 8(3), 4340-4348. DOI: 10.15376/biores.8.3.4340-4348
- Kim, C. H., Kang, C. W., Kang, S. G., and Kang, H. Y. (2012). "Effect of pretreatments on reducing surface cracks of heat-treated western hemlock roundwoods," *J. Korean Wood Sci. & Tech.* 40(5), 343-351. DOI:10.5658/WOOD.2012.40.5.343
- KSA. KS F 2198 (2011). "Determination of density and specific gravity of wood," Korean Standards Association, Seoul, Korea.
- KSA. KS F 2199 (2011). "Determination of moisture content of wood," Korean Standards Association, Seoul, Korea.
- KSA. KS F 2203 (2009). "Method of shrinkage test for wood," Korean Standards Association, Seoul, Korea.
- KSA. KS F 2204 (2009). "Method of test for water absorption of wood," Korean Standards Association, Seoul, Korea.
- KSA. KS F 2206. (2009). "Method of compression test for wood," Korean Standards Association, Seoul, Korea.
- KSA. KS F 2209. (2009). "Method of shear test for wood," Korean Standards Association, Seoul, Korea.
- Metsä-Kortelainen, S., Antikainen, T., and Viitaniemi, P. (2006). "The water absorption of sapwood and heartwood of Scots pine and Norway spruce heat-treated at 170 °C, 190 °C, 210 °C and 230 °C," *Holz. Roh-Werkst.* 64(2006), 192-197. DOI: 10.1007/s00107-005-0063-y
- Panshin, A. J., and de Zeeuw, C. (1980). *Textbook of Wood Technology: Structure, Identification, Properties, and Uses of the Commercial Woods of the United States and Canada*, 4<sup>th</sup> Ed., McGraw-Hill, New York.
- Poncsak, S., Kocaeffe, D., Bouazara, M., and Pichette, A. (2006). "Effect of high temperature treatment on the mechanical properties of birch (*Betula papyrifera*)," *Wood Sci. Tech.* 40(8), 647-663. DOI: 10.1007/s00226-006-0082-9
- Ross, R. J. (2010). "Wood handbook: Wood as an engineering material," *General Technical Report FPL-GTR-190*, Centennial Ed., Forest Products Laboratory, United States Department of Agriculture Forest Service, Madison, WI, ([http://www.fpl.fs.fed.us/documnts/fplgtr/fpl\\_gtr190.pdf?](http://www.fpl.fs.fed.us/documnts/fplgtr/fpl_gtr190.pdf?)).

- Sandoval-Torres, S., Jomaa, W., Marc, F., and Puiggali, J.-R. (2010). "Causes of color changes in wood during drying," *For. Stud. China* 12(4), 167-175. DOI: 10.1007/s11632-010-0404-8.
- Shi, Q., Bao, F. C., Lu, J. X., and Jiang, J. H. (2011). "Effect of heat treatment temperature on the colour of Okan wood," *Adv. Mater. Res.* 214(1), 531-534. DOI: 10.4028/www.scientific.net/AMR.214.531
- Shi, Q., and Jiang, J. H. (2011). "Colour stability of heat-treated Okan sapwood during artificial weathering," *Adv. Mater. Res.* 197-198, 13-16. DOI: 10.4028/www.scientific.net/AMR.197-198.13
- Shmulsky, R., and Jones, P. D. (2011). *Forest Products and Wood Science: An Introduction*, 6<sup>th</sup> Ed., Wiley-Blackwell, England.
- Sundqvist, B. (2002). "Color response of Scots pine (*Pinus sylvestris*), Norway spruce (*Picea abies*) and birch (*Betula pubescens*) subjected to heat treatment in capillary phase," *Holz Roh Werkst* 60,106-114. DOI: 10.1007/s00107-001-0273-x
- Todaro, L., Dichicco, P., Moretti, N., and D'Auria, M. (2013). "Effect of combined steam and heat treatments on extractives and lignin in sapwood and heartwood of turkey oak (*Quercus cerris* L.) wood," *BioResources* 8(2), 1718-1730. DOI: 10.15376/biores.8.2.1718-1730
- Weiland, J. J., and Guyonnet, R. (2003). "Study of chemical modifications and fungi degradation of thermally modified wood using DRIFT spectroscopy," *Holz. Roh-Werkst.* 61(3), 216-220. DOI: 10.1007/s00107-003-0364-y
- Won, K.- R., Kim, T.- H., Hwang, K.- K., Chong, S.- H., Hong, N.- E., and Byeon, H.- S. (2012). "Effect of heat treatment on the bending strength and hardness of wood," *J. Korean Wood Sci. & Tech.* 40(5), 303-310. DOI:10.5658/WOOD.2012.40.5.303
- Xing, D., and Li, J. (2014). "Effects of heat treatment on thermal decomposition and combustion performance of *Larix* spp. wood," *BioResources* 9(3), 4274-4287. DOI: 10.15376/biores.9.3.4274-4287
- Zanuncio, A. J. V., Motta, J. P., da Silveira, T. A., Farias, E. D. S., and Trugilho, P. F. (2014). "Physical and colorimetric changes in *Eucalyptus grandis* wood after heat treatment," *BioResources* 9(1), 293-302. DOI: 10.15376/biores.9.1.293-302
- Zhang, T. T., Tu, D., Peng, C., and Zhang, X. (2015). "Effects of heat treatment on physical-mechanical properties of *Eucalyptus regnans*," *BioResources* 10(2), 3531-3540. DOI: 10.15376/biores.10.2.3531-3540

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