

Effect of Treatment Duration and Clamping on the Properties of Heat-Treated Okan Wood

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Effects of treatment duration and clamping during heat treatment were evaluated relative to the color and physical and mechanical properties of okan wood. Sapwood and heartwood boards from okan wood were treated with and without clamping for 1 to 4 h at 180 °C. Changes in color were mostly due to a reduction in lightness (L^*) and yellow/blue chromaticity (b^*). These values decreased more for longer treatment durations. Red/green chromaticity (a^*) was not affected by the treatment duration. Weight loss and volume shrinkage increased with increased treatment duration. Density only slightly decreased because of a balanced reduction in weight and volume. Clamping during treatment prevented surfaces from having direct contact with the heated air, which resulted in lower weight loss and volume shrinkage than in the samples treated without clamping. Heat-treated wood absorbed less water than the control group, as suggested by the lower equilibrium moisture content and water absorption. Furthermore, the heartwood absorbed less water than the sapwood. An evaluation of the mechanical properties showed a reduction in both the modulus of rupture and modulus of elasticity after heat treatment. Clamping minimized strength reduction in both sapwood and heartwood, particularly for 1 and 2 h heat treatments.

Keywords: Heat treatment; Clamping; Color change; Okan wood (*Cylicodiscus gabunensis* (Taub.) Harms); Physical-mechanical properties; Treatment duration

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INTRODUCTION

The okan tree (*Cylicodiscus gabunensis* (Taub.) Harms) is native to tropical rain forests in West and Central Africa. It has a high density that can reach more than 1.0 g/cm³ and a very high wear resistance (Kadiri *et al.* 2005). The use of okan wood for decking and flooring in South Korea has become popular due to its exotic color and visual appearance, both of which can be improved with heat treatment. Heat treatment of wood is generally performed at temperatures ranging from 160 to 260 °C. To avoid wood combustion, heat treatment is performed in a low-oxygen environment, such as under steam conditions, nitrogen, oil, and, more recently, in a vacuum (Hakkou *et al.* 2005; Ding *et al.* 2011; Allegretti *et al.* 2012; Cao *et al.* 2012a; Dubey *et al.* 2012; Willems *et al.* 2015). Heat treatment has also been performed under air conditions (Aydemir *et al.* 2012; Jiang *et al.* 2014).

Heat treatment modifies the chemical constituents of wood, particularly the extractives and hemicelluloses compounds, which changes the wood properties (Esteves and Pereira 2009). The most obvious effect is the change in wood color, which is beneficial for lighter colored wood. These woods can obtain a natural looking dark color, which adds to the value of less attractive woods (Beckhta and Niemz 2003; Aydemir *et al.* 2012; Cao *et al.* 2012a). Heat treatment reduces the equilibrium moisture content, reduces thermal conductivity, improves durability against decay, improves dimensional stability, and slightly reduces the strength of wood (Boonstra *et al.* 2007a; Kocaefe *et al.* 2008a; Dubey *et al.* 2012; Candelier *et al.* 2013). The improved dimensional stability is due to the decrease in hydroxyl functional groups, which increases the hydrophobicity and reduces the water absorption. The degradation of hemicelluloses is reported to be the principal reason for improved decay resistance (Boonstra *et al.* 2007a; Dubey *et al.* 2012).

Many researchers have studied the effects of processing parameters during heat treatment, such as the treatment medium used to avoid wood combustion, temperature, and duration using various wood species (Metsä-Kortelainen *et al.* 2006; Ding *et al.* 2011; Dubey *et al.* 2012; Candelier *et al.* 2013). However, the application of mechanical restraint on the wood during heat treatment has not yet been studied. In the previous work, as an attempt to cope with the dimensional change, a mechanical restraint was applied using a metal clamp during heat treatment at 160, 180, 200, and 220 °C for 2 h. The results showed that the application of a metal clamp prevented cups, bows, and twists during heat treatment (Hidayat *et al.* 2015a). At the same treatment temperature, the application of a metal clamp successfully prevented strength reduction by up to 20% compared with heat treatment without clamps (Hidayat *et al.* 2015b). The results also led to the conclusion that among the temperatures studied, 180 °C was the optimal temperature to improve dimensional stability with an acceptable reduction in the mechanical strength. In the present study, the clamping method was improved by modifying the set-up of the metal clamp. In order to complete the information about heat treatment of okan wood from the previous study, the effects of heat treatment at 180 °C for different treatment durations under mechanical restraint on color changes and physical and mechanical properties were evaluated.

EXPERIMENTAL

Materials

Sapwood (SW) and heartwood (HW) boards from okan wood that were straight grain, with small variations in density, and defect-free were selected. For the evaluation of color and physical properties, boards with the dimensions of 300 mm (length) × 90 mm (width) × 20 mm (thickness) were used. Boards were cut into two pieces with the dimensions 300 mm × 45 mm × 20 mm to determine the mechanical properties before and after heat treatment. The boards were stacked using a modified metal clamping method from the previous study, as shown in Fig. 1a (Hidayat *et al.* 2015b). Each stack set consisted of six boards, *i.e.*, three boards for color and physical properties evaluation, and three boards for mechanical properties evaluation. Each board was covered by pieces of rectangular metal on their edges, which were fastened by bolts and nuts equipped with metal springs. Flat metal sheets were placed between the boards, and the screw handle on the top of the metal clamp was fastened to get a compact stack. Another set of samples

was stacked without metal clamping for comparison (Fig. 1b). Wood stickers with the dimensions 80 mm × 20 mm × 20 mm were placed between the boards.

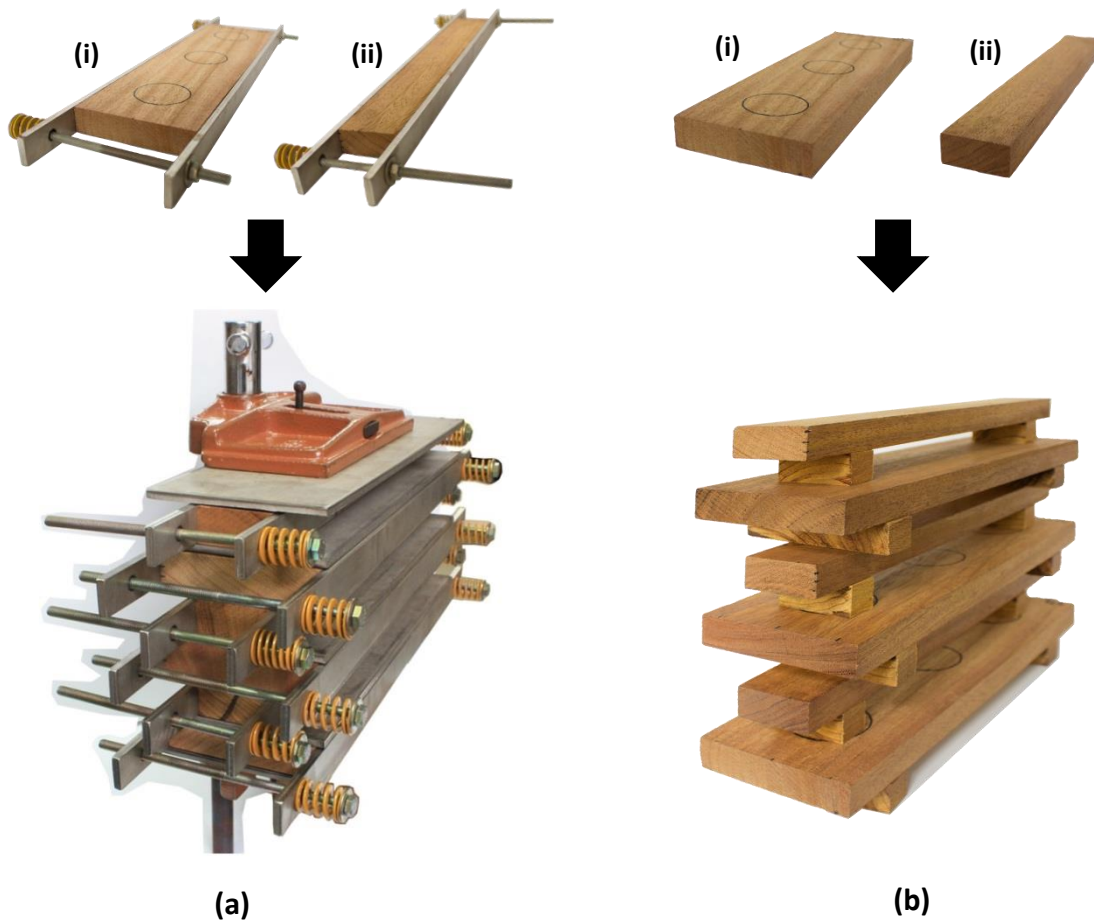


Fig. 1. Stacking of samples during heat treatment: (a) with a metal clamp, (b) without a metal clamp; (i) samples for physical properties evaluation, and (ii) samples for mechanical properties evaluation

Methods

Heat treatment

The prepared boards were heat-treated in an electric oven with a programmable controller (L-Series, JEIO TECH Ltd., Seoul, Korea). Treatment commenced at room temperature, 25 ± 3 °C, and the temperature was increased to 180 °C at a heating rate of 2 °C/min. The target temperature was selected according to the previous results (Hidayat *et al.* 2015b). The target temperature was maintained for 1, 2, 3, or 4 h. At the end of each treatment, the oven was turned off and cooled naturally to 30 °C.

Board evaluation

The surface color measurement of the samples was performed with a chromameter (CR-400, Konica Minolta Inc., Tokyo, Japan) using an illuminant C light source and an observed angle of 2°. The chromameter was calibrated with a white plate ($Y = 93.6$, $x = 0.3134$, $y = 0.3194$) prior to the measurements. Three measurements of

each sample, both before and after heat treatment, were taken to obtain the L^* , a^* , and b^* color values. The total color differences were calculated using the following formula (Aydemir *et al.* 2012),

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

where ΔL^* , Δa^* , Δb^* , and ΔE^* represent changes in lightness, red/green chromaticity, yellow/blue chromaticity, and overall color, respectively.

The levels of perceived difference in color were determined according to Cui *et al.* (2004) and Valverde and Moya (2014), which are as follows: (1) ΔE^* between 0 to 0.5 is defined as negligible; (2) ΔE^* between 0.5 to 1.5 is slightly perceivable; (3) ΔE^* between 1.5 to 3.0 is noticeable; (4) ΔE^* between 3.0 to 6.0 is appreciable; (5) ΔE^* between 6.0 to 12.0 is very appreciable; and (6) ΔE^* more than 12.0 is totally changed.

Weight loss, density, moisture content, water absorption, and volume shrinkage of the samples were evaluated according to Korean standards KS F 2204 (2009), KS F 2203 (2009), KS F 2198 (2011), and KS F 2199 (2011). Weight loss (WL) and volume shrinkage (VS) after heat treatment were estimated according to the following equations,

$$WL (\%) = 100 \times (m_1 - m_2) / m_1 \quad (2)$$

$$VS (\%) = 100 \times (V_1 - V_2) / V_1 \quad (3)$$

where m_1 and m_2 represent the oven dried weight of the samples before and after heat treatment (g), respectively. V_1 and V_2 represent the volume of the samples before and after heat treatment (cm^3), respectively.

The density of the control and heat-treated samples was determined by measuring its air dry weight and volume after the boards were kept in a conditioning room with a relative humidity of 65% and at 25 °C for 2 weeks. The air dry and oven dry weights were measured using an analytical balance with a sensitivity of 0.01 grams to determine the moisture content of the control and heat-treated samples. A water absorption (WA) test was performed by immersing the samples in water for 2 weeks. The weight of the samples before and after water immersion was measured.

The modulus of rupture (MOR) and modulus of elasticity (MOE) were determined by 3-point static bending using a universal testing machine (Model 4482, Instron, Norwood, MA, USA), in accordance with Korean standard KS F 2208 (2009). The loading speed and span length were 1.5 mm/min and 200 mm, respectively. The MOR and MOE values were calculated using the following equations,

$$MOR (\text{N/mm}^2) = \frac{3PL}{2bt^2} \quad (4)$$

$$MOE (\text{N/mm}^2) = \frac{P_p L^3}{4Y_p b t^3} \quad (5)$$

where P is the maximum load (N), P_p is the load at the proportional limit (N), Y_p is the deflection (mm), L is the span length (mm), b is the sample width (mm), and t is the sample thickness (mm).

X-ray diffractograms were collected by an X-ray diffractometer (DMAX 2100 V, RIGAKU, Tokyo, Japan) in the range of 10° to 35° at a scanning speed of 1°/s. Nickel-filtered CuK α radiation was used, and the tube was operated at 40 kV with a filament current of 40 mA. Samples with the dimensions 1 mm (radial) \times 10 mm (tangential) \times 20

mm (longitudinal) were used for measurement. The crystallinity index (*CrI*) was then calculated according to the following equation (Segal *et al.* 1959),

$$CrI (\%) = 100 \times (I_{002} - I_{am}) / I_{002} \quad (6)$$

where I_{002} is the maximum intensity of the (200) plane at $2\theta = 22.7^\circ$ and I_{am} is the intensity diffraction of the amorphous band at $2\theta = 18^\circ$.

A completely randomized factorial design was used for the experimental design. The results of the changes in color, and physical and mechanical properties were submitted to an overall analysis of variance (ANOVA). The homogeneity of the means among combinations was tested using Duncan's Multiple Range Tests at a P-value of 0.05. IBM SPSS Statistics 21 (New York, USA) was used for statistical analysis.

RESULTS AND DISCUSSION

Color Changes

Color is an attribute of visual perception, which is determined by the spectral makeup of light reflected from an object's surface (Sandoval-Torres 2010). It is one of the important factors that influence customers when choosing wood products. Dark brown colors are currently in demand and widely appreciated by customers in the flooring, furniture, and decoration markets (Cao *et al.* 2012a). Figure 2 shows changes to L^* , a^* , b^* , and ΔE^* values after heat treatment at 180 °C for 1, 2, 3, and 4 h. In both sapwood and heartwood boards, with and without clamping, L^* and b^* decreased significantly with increased treatment duration, while a^* in sapwood and heartwood boards with clamping was not affected by the treatment duration. In the previous study, heat treatment of okan wood at different temperatures revealed no differences of a^* values after heat treatment at 160, 180, and 200 °C as well. The a^* values decreased after heat treatment at 220 °C (Hidayat *et al.* 2015b). A previous study by Bekhta and Niemz (2003) showed that the L^* and b^* values were reduced with increased heat treatment duration, and that the a^* value increased after heat treatment for 2 h and remained constant thereafter.

The ΔE^* increased with an increase in treatment duration. Salca *et al.* (2016) studied the effects of heat treatment on black alder and beech at 190 °C for different durations and revealed similar results. Additionally, they reported that the changes in color appeared mostly due to a reduction in lightness, which was related to the degradation of hemicelluloses during heat treatment. Gonzalez-Pena and Hale (2009a) also revealed that ΔE^* was highly influenced by the behavior in lightness. Their study proposed that ΔE^* occurred during heat treatment results from chemical changes in lignin due to the darkening of the lignin which was associated with the generation of chromophoric groups, mainly the increase in carbonyl groups. McDonald *et al.* (2000) stated that when wood was exposed to high temperatures, aldehydes and phenols were formed, which led to the formation of colored compounds as a result of the chemical reactions that occurred.

In the samples with a clamp, the ΔE^* of sapwood was higher than for heartwood. Shi *et al.* (2011) also revealed that the effect of heat treatment on the color change in sapwood from okan was more obvious than for heartwood. However, in the samples without a clamp, sapwood and heartwood had similar ΔE^* values. Figure 2 clearly shows

that the samples with a clamp exhibited a lower magnitude of ΔE^* than the samples without a clamp. Clamping during heat treatment protected the wood sample surfaces (radial and tangential surfaces) from direct contact with the heated air, and allowed only the cross section surfaces to be exposed. This may have resulted in a lower oxidation of wood components than for the samples without clamping.

The results also showed that the samples with the modified clamping method exhibited lower ΔE^* values compared to the previous clamping method (Hidayat *et al.* 2015b). The ΔE^* after heat treatment at 180 °C for 2 h using the modified clamping method was 5.86 and 3.60 for sapwood and heartwood, respectively. On the other hand, for the previous clamping method ΔE^* was 9.98 and 6.69 for sapwood and heartwood, respectively. This may have been due to the less direct contact of the wood surfaces with the heated air.

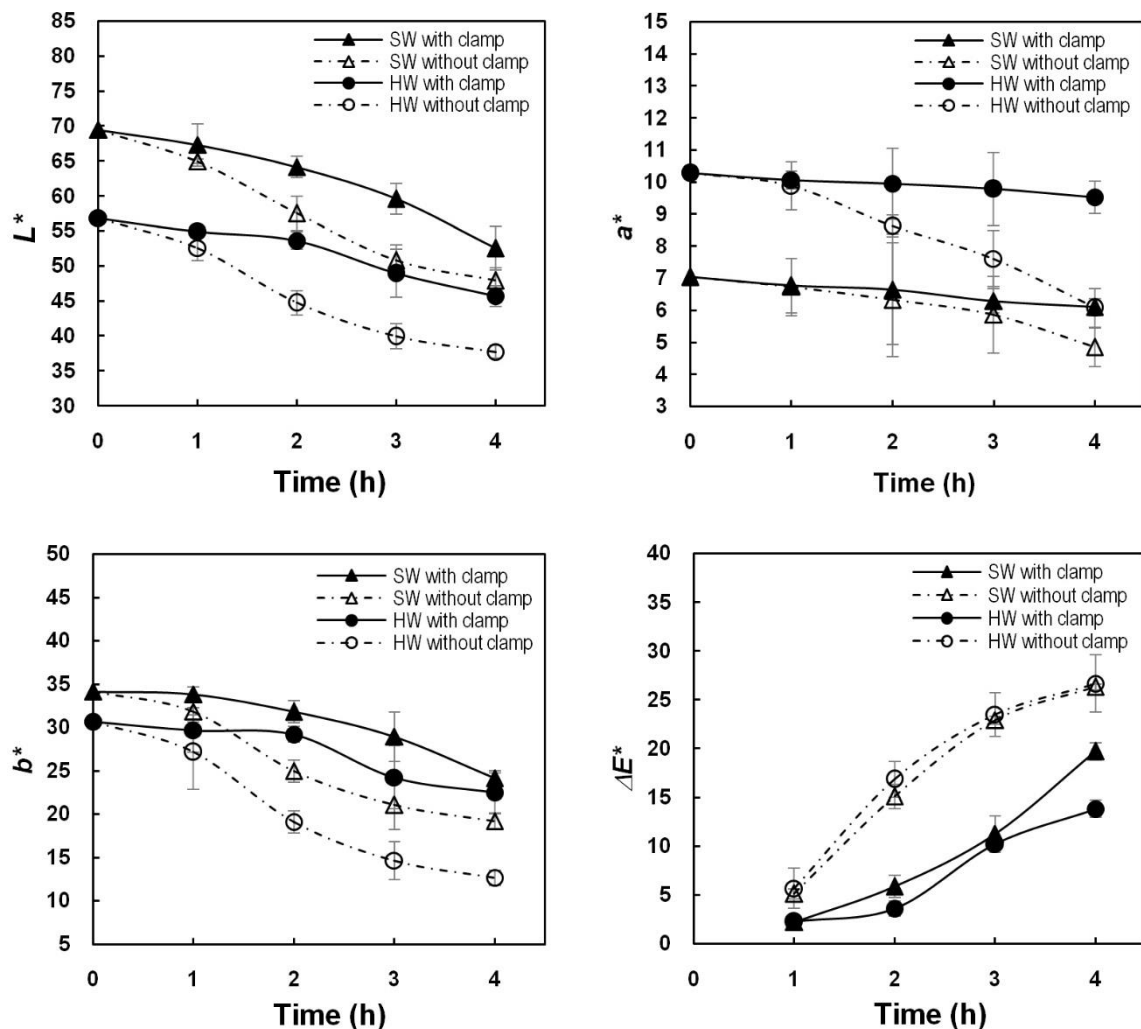


Fig. 2. Effect of treatment duration and clamping method during heat treatment on the change of L^* , a^* , b^* , and ΔE^* (SW= sapwood; HW= heartwood)

Table 1 shows that those samples subjected to clamping and heat treatment for 1, 2, and 3 h for both sapwood and heartwood boards only resulted in noticeable ($\Delta E^* = 1.5$ to 3.0), appreciable ($\Delta E^* = 3.0$ to 6.0), and very appreciable ($\Delta E^* = 6.0$ to 12.0) changes,

respectively. The color was totally changed ($\Delta E^* = > 12.0$) after heat treatment for 4 h. While in the samples without clamping, the color was totally changed at the shorter treatment duration of 2 h.

Table 1. The Levels of Perceived Difference in Color after Heat Treatment

Wood Part	Time	ΔE^* Level	
		With clamp	Without clamp
Sapwood	1 h	Noticeable	Appreciable
	2 h	Appreciable	Totally changed
	3 h	Very appreciable	Totally changed
	4 h	Totally changed	Totally changed
Heartwood	1 h	Noticeable	Appreciable
	2 h	Appreciable	Totally changed
	3 h	Very appreciable	Totally changed
	4 h	Totally changed	Totally changed

*According to the classification of the level color change by Cui *et al.* (2004) and Valverde and Moya (2014)

Physical Properties

Weight loss and volume shrinkage increased with increased treatment duration (Table 2). The results showed similar trends with increases in temperature (Hidayat *et al.* 2015b). However, the treatment temperature seemed to have a more significant effect than the treatment duration, as shown by the higher magnitude of weight loss and volume shrinkage. The samples with clamping exhibited significantly lower weight loss and volume shrinkage compared to the samples without clamping. Among the samples with clamping, sapwood had a higher degree of weight loss than heartwood. This effect was consistent among the samples without clamping, but the difference was not significant. A previous study on the heat treatment of *Pinus pinaster* showed that in the earlier stages of treatment (1 h treatment duration), heartwood lost more mass than sapwood, and as the treatment duration increased to 3 h, this reversed and the weight loss of sapwood was significantly higher than for heartwood, increasing to 10.7% and 5.1%, respectively (Esteves *et al.* 2014).

The weight loss after heat treatment may have occurred due to the thermal oxidation of wood components, which was mostly the degradation of hemicellulose. For example, Yildiz *et al.* (2006) reported that the heat treatment of spruce wood under air conditions at 180 °C decreased the hemicellulose content of 21.43% to 8.21, 6.02, and 2.66% for heat treatments with durations of 2, 6, and 10 h, respectively. The cellulose and lignin contents were unchanged after heat treatment. Kocaefe *et al.* (2008b) reported that heat treatment at temperatures ranging from 180 to 260 °C started to degrade hemicelluloses and soften lignin, which caused a reduction in mass. Yang *et al.* (2007) also stated that hemicellulose was the first component to degrade during the thermal treatment of wood, followed by cellulose at 315 to 400 °C, and the maximum decomposition of lignin occurred above 900 °C.

The density of wood after heat treatment decreased with increased treatment duration. However, these results were not significantly different compared to untreated

samples. Kasemsiri *et al.* (2012) reported that heat-treated eastern red cedar had similar responses, and that there was an insignificant decrease in density after heat treatments with different durations. This insignificant decrease may have been due to a balanced amount of weight loss and volume shrinkage from the heat treatment.

Table 2. Effect of Treatment Duration and Clamping Method During Heat Treatment on Physical Properties of Okan Wood

Wood Part	Time	Weight Loss (%)		Volume Shrinkage (%)		Density (g/cm ³)	
		With clamp	Without clamp	With clamp	Without clamp	With clamp	Without clamp
Sapwood	Control	-	-	-	-	0.66 ^A (0.08)	0.66 ^A (0.08)
	1 h	5.18 ^A (0.31)	7.10 ^B (0.82)	2.81 ^A (0.27)	3.58 ^B (0.28)	0.64 ^A (0.08)	0.63 ^A (0.03)
	2 h	7.07 ^B (0.11)	8.03 ^{BC} (1.03)	4.04 ^{BC} (0.54)	4.58 ^C (0.23)	0.63 ^A (0.05)	0.62 ^A (0.04)
	3 h	8.03 ^C (0.19)	9.09 ^{CD} (0.88)	3.94 ^B (0.25)	4.72 ^C (0.29)	0.62 ^A (0.03)	0.60 ^A (0.06)
	4 h	8.34 ^C (0.29)	9.60 ^D (0.79)	5.08 ^{CD} (0.60)	5.71 ^D (0.31)	0.60 ^A (0.04)	0.58 ^A (0.05)
Heartwood	Control	-	-	-	-	0.94 ^A (0.06)	0.94 ^A (0.06)
	1 h	3.50 ^A (0.31)	6.62 ^{BC} (1.00)	2.45 ^A (0.13)	3.25 ^{BC} (0.44)	0.92 ^A (0.04)	0.91 ^A (0.07)
	2 h	5.50 ^B (0.16)	7.72 ^D (0.45)	3.01 ^B (0.17)	4.38 ^{CD} (0.58)	0.92 ^A (0.13)	0.90 ^A (0.05)
	3 h	6.08 ^C (0.35)	8.45 ^{DE} (0.57)	3.75 ^C (0.53)	5.30 ^E (0.21)	0.90 ^A (0.05)	0.91 ^A (0.04)
	4 h	6.58 ^C (0.36)	9.71 ^E (0.60)	4.47 ^D (0.17)	6.86 ^F (0.08)	0.90 ^A (0.05)	0.89 ^A (0.07)

* The means are averages of 5 replicates. Numbers in parenthesis are standard deviations. Means within a physical property (including samples with and without clamp and by treatment durations) followed by the same capital letter are not significantly different at 5% significance level using Duncan's multiple range test.

Kubojima *et al.* (2000) reported that the density of Sitka spruce decreased after heat treatment at 160 °C for 0.5, 1, and 2 h, and a more significant density reduction occurred after heat treatments with durations of 4, 8, and 16 h. Boonstra *et al.* (2007b) attributed the reduction in density after heat treatment to the degradation of hemicelluloses into volatile products that evaporated during treatment, the evaporation of extractives, and a lower equilibrium moisture content of the boards since heat-treated wood is more hydrophobic.

In sapwood, the weight loss during heat treatment may have been due to a decrease in density and an increase in volume shrinkage, which resulted in strong linear relationships with coefficient of determinations (R^2) of 0.83 and 0.85 for density and volume shrinkage, respectively (Fig. 3). Weight loss and density exhibited not as strong linear relationships, particularly in heartwood, with R^2 values of 0.55. The regression analysis of ΔE^* showed similar results when it came to weight loss (Fig. 4). Furthermore,

the overall results showed that the ΔE^* had a stronger relationship with density and volume shrinkage than with weight loss.

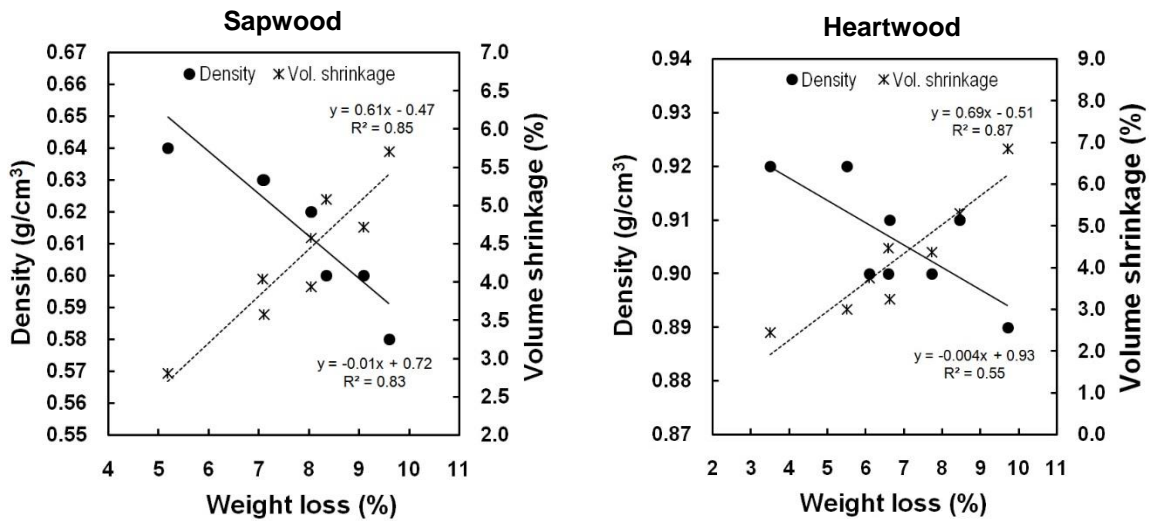


Fig. 3. Relationships between weight loss and density/volume shrinkage in sapwood and heartwood of heat-treated okan wood

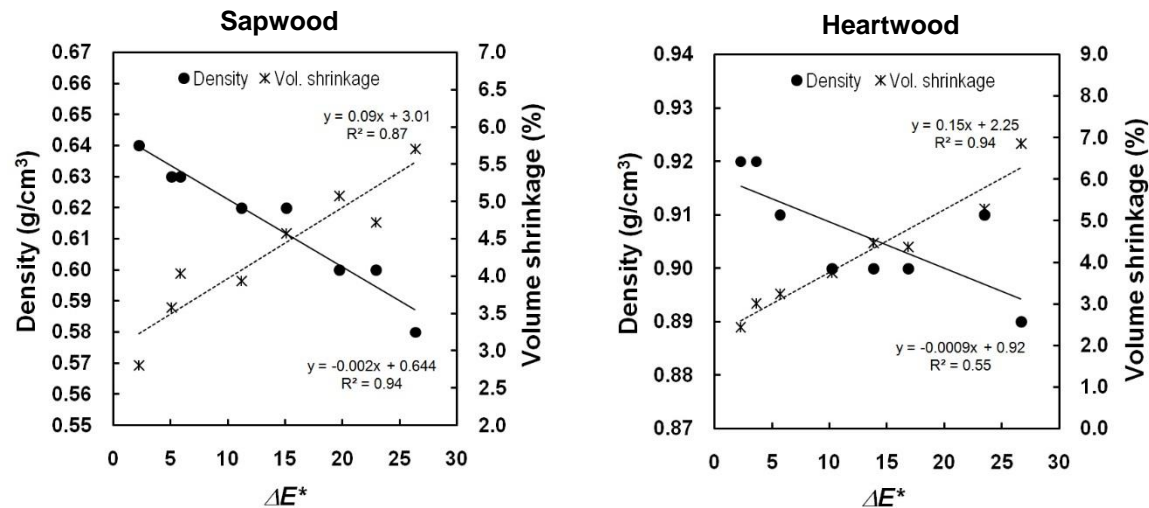


Fig. 4. Relationships between ΔE^* and density/volume shrinkage in sapwood and heartwood of heat-treated okan wood

The results showed that the equilibrium moisture content (EMC) and WA decreased after heat treatment (Fig. 5). Increased treatment duration resulted in lower EMC and WA values. The samples without clamping exhibited lower EMC and WA values compared to those with clamping. The EMC of heartwood was greater than that of sapwood. The WA in heartwood was lower than in sapwood. The results for WA compared well with the previous heat treatment study of sapwood and heartwood from spruce and pine wood (Metsä-Kortelainen *et al.* 2006). That study revealed that the WA of sapwood was greater than for heartwood in both wood species, and that the difference was significant, particularly for pine.

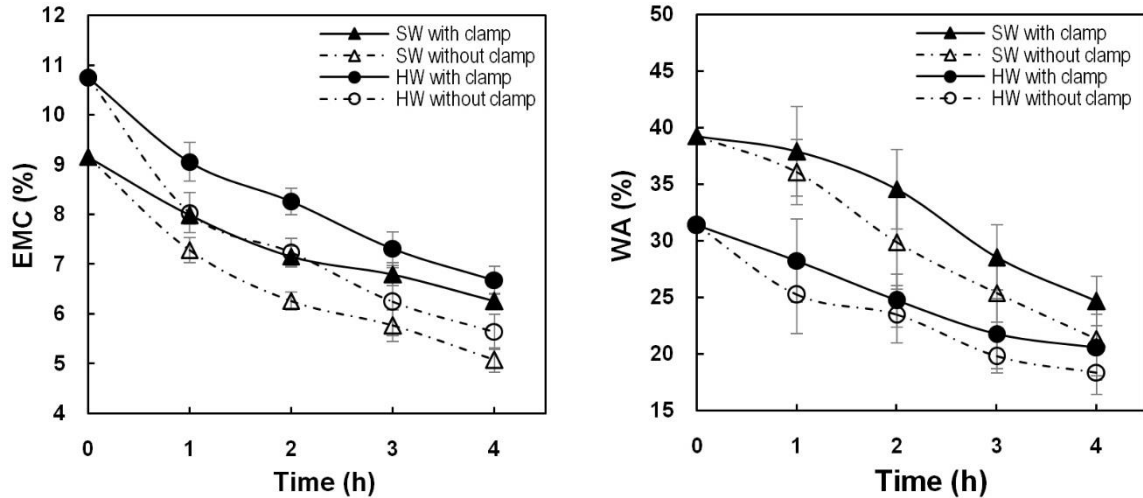


Fig. 5. Effect of treatment duration and clamping method during heat treatment on the EMC and water absorption. (SW= sapwood; HW= heartwood)

The results suggested that the weight loss during heat treatment was attributable to the decrease in the EMC and WA (Fig. 6). The higher weight loss resulted in lower EMC and WA values. The decrease of hemicellulose during the heat treatment of wood may have been by caused the weight loss and increased hydrophobicity of the cell walls because of a decrease in hydroxyl groups, which resulted in less WA (Jamsa and Viitaniemi 2001). The causes of the decrease of free hydroxyl groups were summarized by Boonstra *et al.* (2007b) and are as follows: (1) depolymerization of hemicelluloses, causing a reduction in the total amount of hydroxyl groups; (2) an increase in the relative proportion of crystalline cellulose, in which the hydroxyl groups are not easily accessible to water molecules; and (3) crosslinking of the lignin network, which may hinder the accessibility of free hydroxyl groups to water. The ΔE^* also exhibited a strong correlation with the decrease in EMC and WA (Fig. 7). Hence, ΔE^* could be a good predictor for changes in the physical properties of heat-treated wood.

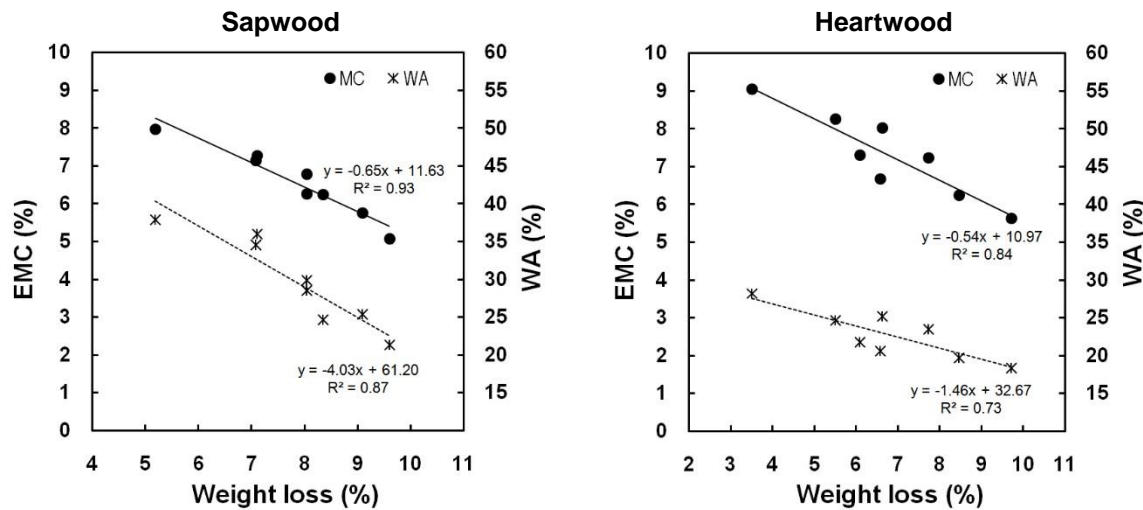


Fig. 6. Relationships between weight loss and EMC/WA in sapwood and heartwood of heat-treated okan wood

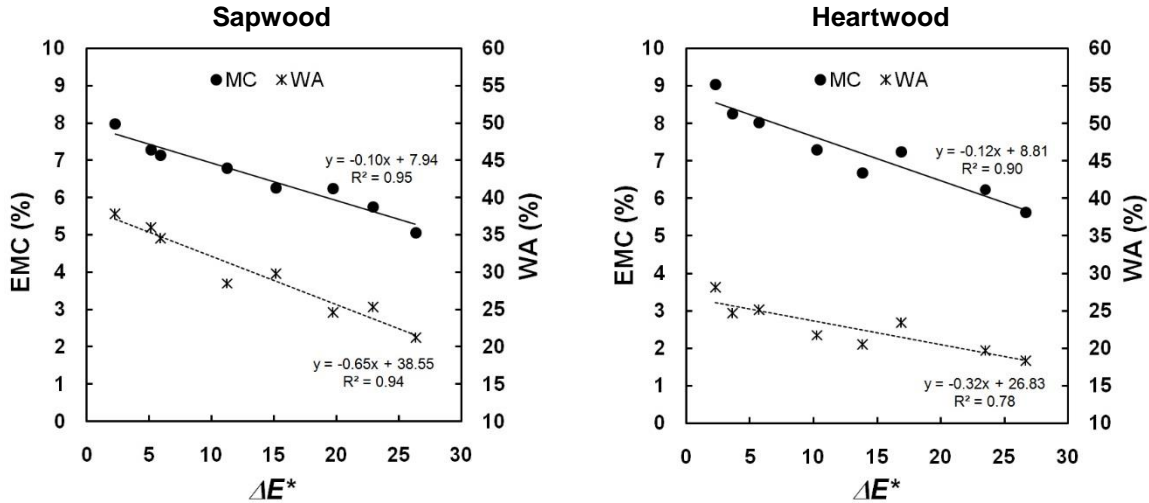


Fig. 7. Relationships between ΔE^* and EMC/WA in sapwood and heartwood of heat-treated okan wood

Mechanical Properties

The *MOR* and *MOE* values increased slightly after heat treatment for 1 and 2 h, particularly in the samples with clamping (Fig. 8). However, for the longer treatment durations of 3 and 4 h, the values significantly decreased. An increase in the mechanical properties for a short time after heat treatment was similarly reported in other previous studies. Kubojima *et al.* (2000), for example, treated Sitka spruce at 160 °C for 0.5, 1, 2, 4, 8, and 16 h and revealed that the *MOR* and *MOE* increased slightly after heat treatment for 0.5 to 4 h, and then decreased thereafter. Cao *et al.* (2012b) reported that the *MOE* of sapwood from Chinese fir increased by 1% and 3% after heat treatment at 170 °C for 1 h and 2 h, respectively, and then decreased. Shi *et al.* (2007) reported that the *MOR* of birch increased by 6% after heat treatment at 200 °C for 3 h.

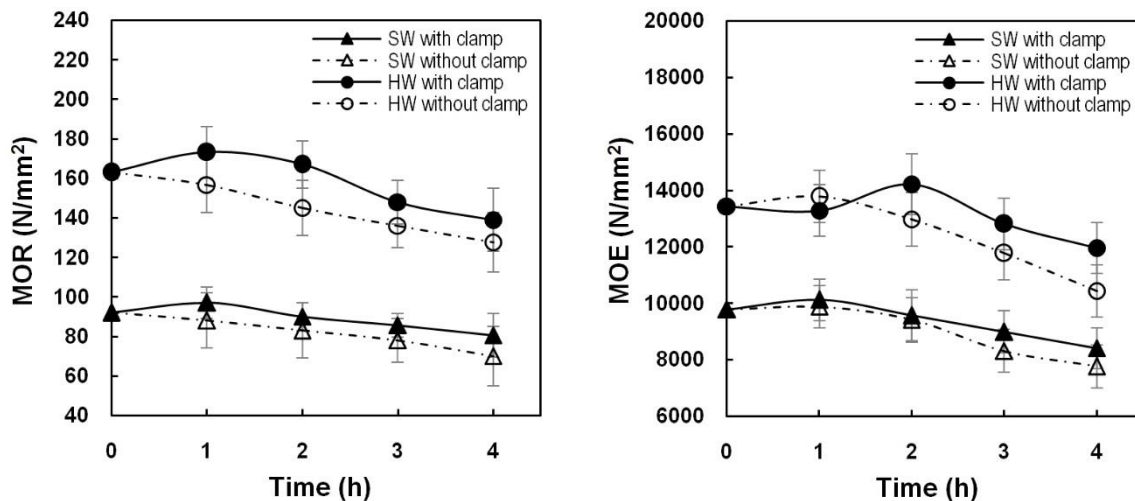


Fig. 8. Effect of treatment duration and clamping method during heat treatment on the *MOR* and *MOE*. (SW= sapwood; HW= heartwood)

The change in the *MOR* and *MOE* after heat treatment might have been caused by the change in the crystallinity of okan wood. The X-ray diffractograms showed that the intensity of the three peaks at 2θ values of 14.7° , 16.2° , and 22.7° , which related to the $(\bar{1}\bar{1}0)$, (110) , and (200) diffraction profiles of cellulose in okan wood, were changed after the heat treatment at 180°C for different treatment durations (Fig. 9). The relative amount of crystalline cellulose of the control sample increased from 70.6% to 74.1% after heat treatment for 1 h and remained constant after heat treatment for 2 h, with a value of 70.6%. On the other hand, the heat treatments with longer durations decreased the relative crystallinity to 66.0% and 64.6% for the 3 and 4 h heat treatments, respectively. These results revealed that the heat treatments with shorter treatment durations had a stimulating effect on the crystallization of amorphous cellulose, which resulted in the slight increase of the *MOR* and *MOE*. These results agreed with previous studies. Kubojima *et al.* (1998) reported an increase in relative crystallinity and a slight increase of the mechanical properties for short heat treatment durations. Kocafe *et al.* (2008b) stated that the slight increase of mechanical properties of aspen wood (*Populus tremuloides*) after heat treatment was due to the degradation of amorphous cellulose content and the increase in relative crystallinity.

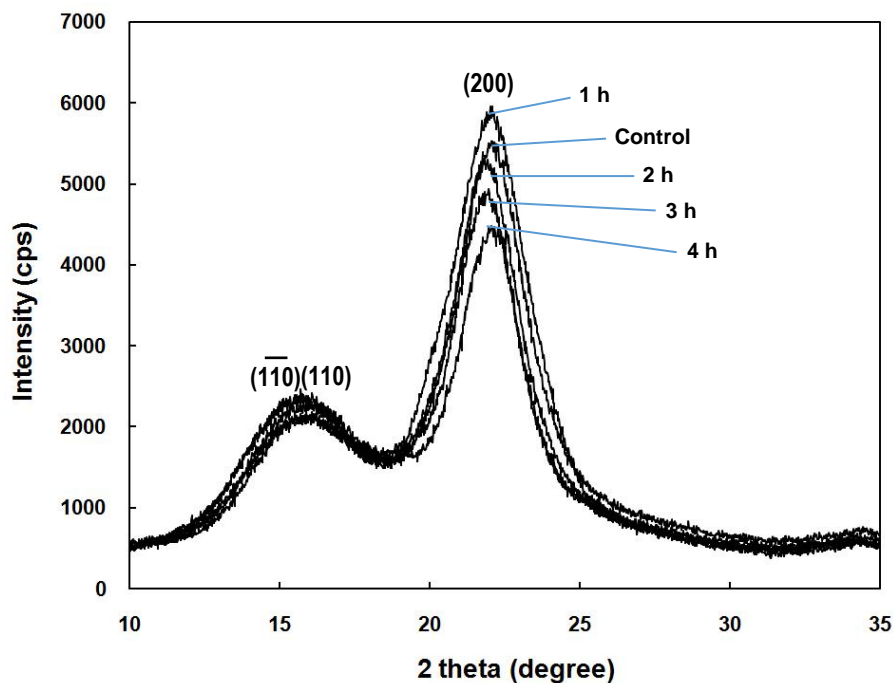


Fig. 9. Equatorial X-ray diffractograms of okan wood before and after heat treatment

The samples with clamping exhibited less *MOR* and *MOE* reduction compared to the samples without clamping, showing higher *MOR* and *MOE* values (Fig. 8). But overall, the difference was not statistically significant. These results were comparable to the results for weight loss during heat treatment, *i.e.* the samples with clamping experienced lower weight loss than the samples without clamping. A strong linear relationship between weight loss and *MOR* was observed in both sapwood and heartwood, with R^2 values of 0.93 and 0.83, respectively (Fig. 10). The relationship between weight loss and *MOE* tended to be linear with R^2 values of 0.65 and 0.54 for sapwood and

heartwood, respectively. The *MOR* and *MOE* of untreated and heat-treated heartwood samples were higher than for sapwood due to the higher density of heartwood (Fig. 8).

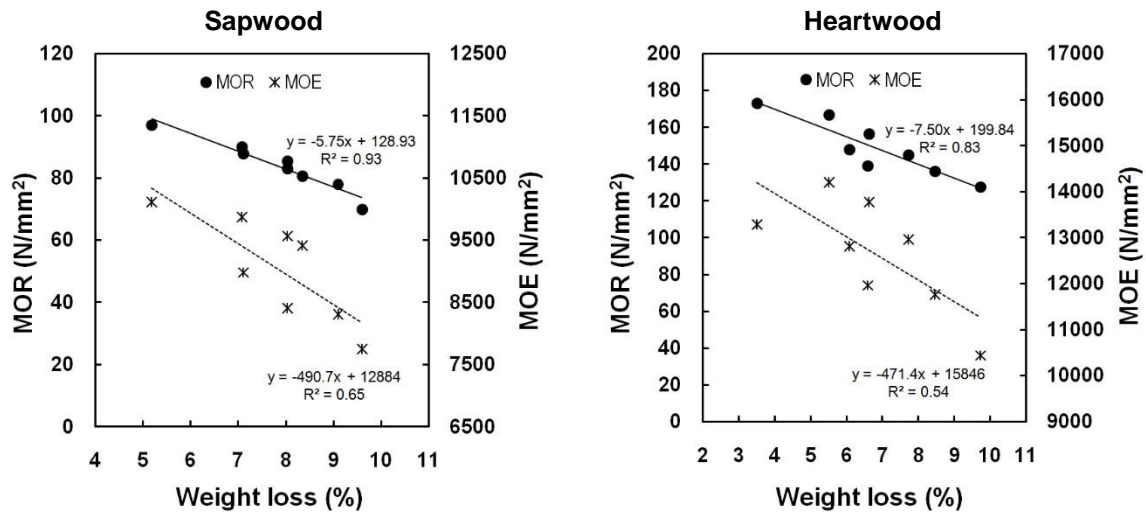


Fig. 10. Relationships between weight loss and bending properties in sapwood and heartwood of heat-treated okan wood

Besides weight loss, the color change in wood is one of the most important factors in heat treatment and is often considered an indication of the quality of the treatment (Matsuo *et al.* 2011; Candelier *et al.* 2016). The results in this study revealed a linear relationship between the ΔE^* and mechanical properties (Fig. 11). In comparison to weight loss, ΔE^* was associated with bending properties, in both sapwood and heartwood. Overall, heartwood showed less strong relationship compared to sapwood. Similar results in sapwood and heartwood of pine wood were observed (Brischke *et al.* 2007). Previous studies have reported a correlation between color change and bending strength. Bekhta and Niemz (2003) reported a strong linear relationship ($R^2 = 0.99$) between ΔE^* and both *MOE* and *MOR*. Gonzalez-Pena and Hale (2009b) found that ΔE^* was a good predictor of the strength of heat-treated wood.

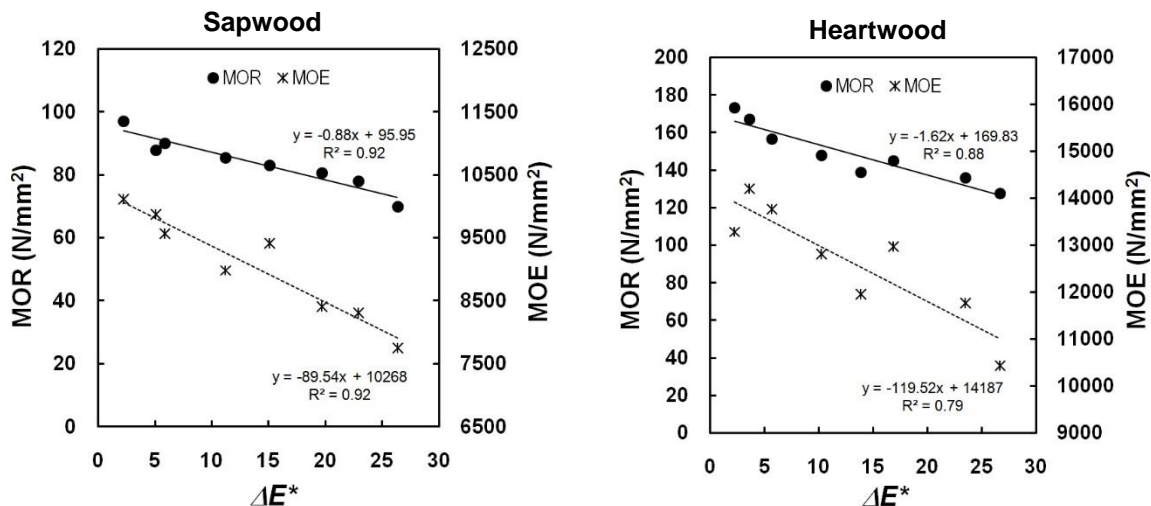


Fig. 11. Relationships between ΔE^* and bending properties in sapwood and heartwood of heat-treated okan wood

CONCLUSIONS

1. Longer treatment durations increased the ΔE^* , which was mostly due to a reduction in the L^* and b^* chromaticity. Boards with the application of a metal clamp exhibited lower ΔE^* than for boards without a clamp.
2. The weight loss and volume shrinkage increased with increased treatment times. ΔE^* , weight loss, and volume shrinkage in the samples with clamping were lower than in the samples without clamping. Sapwood showed a higher magnitude of weight loss and ΔE^* than heartwood, particularly in the samples without clamping.
3. Heat-treated okan wood absorbed less water compared to the control. In addition, the heartwood of heat-treated okan absorbed less water than the sapwood.
4. The modified clamping showed better results in minimizing the *MOR* and *MOE* changes compared to the previous clamping method, especially for the short heat treatment durations of 1 and 2 h.

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