Effects of Temperature and Duration of Heat Treatment on the Physical, Surface, and Mechanical Properties of Japanese Cedar Wood

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This study investigated the application of heat to wood samples from Japanese cedar trees (*Cryptomeria japonica*) of small and medium diameters to evaluate the effects of both the temperature and duration of treatment on its surface, physical, and mechanical properties. The results indicate that the density, moisture content, and hygroscopicity of the wood samples decreased as the treatment temperature and duration increased, and the mass loss increased under the same conditions. Additionally, the dimensional stability of the wood improved in response to increased temperatures and prolonged durations. These results suggest that heat treatment can be used to improve the dimensional stability of wood. The surface color of the wood darkened progressively with increasing treatment temperature and duration, and the hydrophobicity of the wood sample improved as a result of the heat treatment. In contrast to the untreated wood, the heat-treated wood exhibited decreased mechanical properties with an increase in the treatment temperature and duration.

Keywords: Japanese cedar (Cryptomeria japonica); Heat treatment; Dimensional stability; Hygroscopicity; Mechanical strength

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INTRODUCTION

Japanese cedar (*Cryptomeria japonica*) is an evergreen tree species often used to make wood products for structural and decorative applications in Taiwan and Japan because of its fast growing cycle and favorable processing properties. To evaluate the mechanical properties of Taiwan-grown Japanese cedar wood, Lee *et al*. (2014) conducted relevant tests and found that the modulus of elasticity (MOE) and bending strength of a 35-year-old Japanese cedar wood were 7.1 GPa and 53.1 MPa, respectively, indicating that it is a suitable material for structural construction.

As the rotation age of these plantation forests is relatively short, the lumber produced may contain a high proportion of juvenile wood. Wang and Chen (1992) investigated Japanese cedar that was grown applying distinct plantation spacing schemes and thinning methods, and discovered that the average juvenile-to-mature-wood transition age is approximately 17 to 20 years. The findings of Csoka *et al*. (2005) further established that the lumber obtained from young Japanese cedar contains a high percentage of juvenile wood, which has unfavorable characteristics such as wide, vague growth rings; low density; low latewood percentage; large longitudinal and small tangential shrinkage tendencies; and high warping tendency during drying processes. In contrast to mature wood, juvenile wood also possesses low weathering and biodegradation resistances.

The life cycle and durability of construction materials are crucial. As an organic material, wood is vulnerable to environmental and biodegradation, which induces wood corrosion and degradation when used in outdoor conditions. In this context, wood is prone to discoloration, loss of mechanical strength, and even failure, thus endangering the safety of resulting structures. Consequently, it is important to study the potential treatments available to improve the properties of such lumber for use in future applications.

To increase the outdoor durability and life span of wood products, chemicals are typically used to treat wood in attempts to improve the dimensional stability (Papadopoulos and Pougioula 2010). However, chemical approaches can have negative impacts on human health and the environment. In response to the environmental movement, people are paying increasing attention to living environments and environmental protection (Hakkou *et al.* 2005).

As an environmentally friendly method for modifying wood properties, the heat treatment technique provides a promising alternative to conventional chemical approaches (Yildiz 2002; ThermoWood Handbook 2003). Heat treatment is typically conducted by treating wood at high temperatures (typically 150 to 230 °C) to change the chemical composition of the wood and thus improve its properties. Heat-treated wood possesses increased biological resistances (Rowell *et al*. 2009; Hadi *et al*. 2012), high dimensional stability, and low deforming and cracking tendencies (Gunduz and Aydemir 2009; Kasemsiri *et al.* 2012; Priadi and Hiziroglu 2014). In addition, heat treatment techniques can also be used to darken wood surface colors and reduce the color difference at sap-heartwood boundaries, thereby visually enhancing the natural beauty of wood (Bekhta and Niemz 2003; Esteves *et al.* 2008; González-Peña and Hale 2009; Srinivas and Pandey 2012).

Various heat treatment factors (*e.g.*, temperature, heating rate, duration, and cooling speed) can influence the properties of heat-treated wood (Kocaefe *et al*. 2009). Gündüz *et al*. (2008) investigated heat-treated Camiyani Black Pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood and suggested that the density of the wood decreased as the treatment temperature increased. Esteves *et al*. (2008) reported that prolonged treatment durations led to increased mass loss (ML) for eucalyptus (*Eucalyptus globulus*) wood.

The effect of heat treatment on wood MOE varies depending on the wood species and heat treatment temperatures and durations. For example, heat treatment has been verified to induce a MOE reduction in many wood species (Yildiz 2002; Borrega and Kärenlampi 2008; Mburu *et al*. 2008; Esteves and Pereira 2009; Korkut and Hiziroglu 2009). Furthermore, many studies have indicated that heat treatment can cause a 2% to 32% reduction in wood compression strength according to wood species and the application of different treatment conditions (Yildiz 2002; Korkut and Guller 2008).

Korkut and Hiziroglu (2009) explored the effect of heat treatment on the mechanical properties of hazelnut wood treated at 120 to 180 °C for 2 to 10 h. Their research revealed that longer treatment durations resulted in lower wood shear strength. A similar result was reported by Bakar *et al*. (2013), who evaluated the shear strength of rubber wood, Eastern red cedar wood, and red oak wood.

The present study aimed to determine the appropriate heat treatment conditions, according to wood species and their intended applications, to optimize the effectiveness of heat treatment on wood. For this purpose, the present study focused on 35-year-old Taiwan-grown Japanese cedar wood to investigate the effects of particular heat treatment factors (*i.e.*, temperature and duration) on selected physical, surface, and mechanical properties.

EXPERIMENTAL

Materials

Test wood was sampled from 35-year-old Japanese cedar logs purchased from Jang Chang Lumber Industry Limited Company (Hsinchu, Taiwan). The wood was cut into 20 × 20 × 320 mm (tangential, radial, and longitudinal measurements, respectively, *i.e*., T × R × L) samples, which were randomly divided into a control group and nine heat treatment groups according to the designed treatment conditions (Table 1). Each of the nine treatment groups contained 40 samples, and another set of 40 samples was set aside as the control group to benchmark the effects of the treatment conditions. Subsequently, the wood samples were stored in a room conditioned at 20 °C and 65% relative humidity (RH) for at least four weeks to reach equilibrium moisture content (EMC).

Heat Treatment Procedure

Although distinct treatment conditions were applied to each of the groups, the conditioned Japanese cedar wood samples in the nine treatment groups (3 treatment temperatures, 170 °C, 190 °C, and 210 °C, and 3 treatment durations, 1 h, 2 h, and 4 h) were all treated using the same general heat treatment procedure.

The samples were first placed inside an oven, which was heated to 100 °C over a 6-h period with a heating rate of approximately 0.2°C/min. The temperature of the oven was then increased to 130 °C over the following 6 h with a heating rate of approximately 0.08 °C/min, during which wood drying was initiated and completed spontaneously. At this moment, samples were removed from the oven to measure the dried weight as the base to determine physical properties of heat-treated samples. Afterward, samples were placed back into the oven for the final heating phase. During the last heating phase, the treatment temperature was reached after an additional 6 h of heating with a heating rate of 0.11 to 0.22 °C/min. The temperature was then kept constant for the required time duration. After the heating process, the samples were cooled in the oven to attain room temperature.

Physical Properties

Equilibrium moisture content

The EMCs of both the heat-treated samples and the untreated control samples were determined according to the national standard for the determination of the moisture content for physical and mechanical tests (CNS452 2013). Before the EMCs of the heat-treated samples were ascertained, the samples were stored in a conditioned room at 20 °C and 65% RH for approximately four weeks to reach equilibrium. Subsequently, the EMCs of the samples were calculated as follows,

$$EMC\ (\%) = \left(m_u - m_o\right)/(m_o) \times 100 \tag{1}$$

where m_u is the mass (g) of the sample after conditioning and m_o is the mass (g) of the oven-dried sample.

Mass loss

The samples' mass losses (MLs) caused by the heat treatment were determined based on the oven-dried mass of the sample before treatment. The ML was determined as,

$$\text{ML (\%)} = \left(W_o - W_1\right)/(W_o) \times 100 \tag{2}$$

where W_o is the oven-dried mass (g) of the sample before treatment and W_1 is the oven-dried mass (g) of the same sample after heat treatment.

Density

The national standard for the determination of density for physical and mechanical tests (CNS451 2013) was applied to determine the densities of the heat-treated and control samples using the following equation,

$$\rho\ (kg/m^3) = m_u\ /\ v_u \tag{3}$$

where m_u and v_u are the mass and volume of the heat-treated or control sample, respectively, after conditioning.

Hygroscopicity

The test samples were prepared by cutting the aforementioned heat-treated wood into slices sized 20 × 20 × 10 mm (T × R × L). The test procedure was performed by first oven-drying the samples at 103 ± 2 °C and then conditioning the samples at 40 °C and 65% RH for 72 h before measuring the weight and volume of the samples. The samples were oven-dried again and then conditioned at 40 °C and 95% RH for a further 72 h. According to the measurements, the samples' EMC and the moisture excluding efficiency (MEE) values at 65% and 95% RH were calculated using the following equations, respectively:

$$EMC_{RH}\ (\%) = \left(W_\varepsilon - W_o\right)/(W_o) \times 100 \tag{4}$$

where W_e is the EMC mass of the sample conditioned at the corresponding RH and W_o is the oven-dried mass of the sample; and

$$MEE\,(\%) = \left(E_c - E_t\right)/(E_t) \times 100 \tag{5}$$

where E_c is the average EMC of the control samples and E_t is the EMC of the heat-treated sample.

Dimensional shrinkage

To determine the dimensional shrinkage induced by the heat treatment, wood samples with the dimensions of 20 × 20 × 5 mm (T × R × L) and 20 × 5 × 60 mm (T × R × L) were tailored from the heat-treated wood to determine the transverse (*i.e.*, tangential and radial) and longitudinal shrinkage, respectively. Control samples with the same dimensions were also prepared.

All the samples were fully immersed in water at a temperature of 20 ± 5°C, and changes in the weight and size in the three orthotropic directions were measured at a three-day interval until the size change between two consecutive measurements in each of the orthotropic directions was less than 0.02 mm. The overall dimensional shrinkage percentage was then determined as follows,

$$\beta_{(r,t,l)max} = \left(L_{(r,t,l)1} - L_{(r,t,l)4}\right)/\left(L_{(r,t,l)1}\right) \times 100 \tag{6}$$

where L_{r1}, L_{t1}, and L_{l1} are the dimensional measurements (mm) along the radial, tangential, and longitudinal directions, respectively, at the fiber saturation point; and, L_{r4}, L_{t4}, and L_{l4} are the oven-dried dimensional measurements (mm) along the radial, tangential, and longitudinal directions, respectively.

Water absorption

To determine the water absorption (WA) of the heat-treated wood, samples sized 20 × 20 × 5 mm (T × R × L) were dried at 103 ± 2 °C to reach complete dryness before weight and volume measurements were performed. A pressure-controlled water immersing procedure was then conducted. In this procedure, the oven-dried samples were immersed in distilled water inside a tank, which was kept in a vacuum for 30 min before the air pressure of the tank was returned to atmospheric pressure and then kept constant for 1 h. The tank was placed in a vacuum for an additional 30 min and returned to atmospheric pressure for one day before the water in the tank was replaced with new distilled water. The same procedure was repeated for seven consecutive days before the samples' volume and weight measurements were taken, with the volumetric swelling coefficient (VSC), anti-swelling efficiency (ASE), and WA of the samples determined using the following equations,

$$VSC\,(\%) = \left(V_w - V_o\right)/(V_o) \times 100 \tag{7}$$

where V_w is the green-state volume of the sample and V_o is the oven-dried volume of the sample;

$$ASE\,(\%) = \left(S_c - S_t\right)/(S_t) \times 100 \tag{8}$$

where S_c and S_t are the VSCs of the sample before and after heat treatment, respectively; and,

$$WA\,(\%) = \left(W_w - W_o\right)/(W_o) \times 100 \tag{9}$$

where W_w is the green-state weight of the sample and W_0 is the oven-dried weight of the sample.

Surface Properties

Color measurement

A CM-3600D spectrophotometer (Minolta Co., USA) was used to measure the wood samples' surface color changes caused by the heat treatment. A light source of illuminant D65, a visual field of 10°, and a measuring head diameter of 8 mm were employed. Each sample was measured three times using the spectrophotometer to obtain the average surface color measurement.

Based on the spectrophotometer measurements, color parameters L^* (lightness coordinate), a^* (red/green coordinate), and b^* (yellow/blue coordinate) in the CIELAB color space for the surface color of each sample were determined. The L^*, a^*, and b^* values were then used to calculate the lightness difference ΔL^*, red/green difference Δa^*, yellow/blue difference Δb^*, and color difference ΔE^* using the following equations,

$$\Delta L^* = L_2^* - L_1^*$$

$$\Delta a^* = a_2^* - a_1^*$$

$$\Delta b^* = b_2^* - b_1^*$$

$$\Delta E^{*} = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \qquad (10)$$

where L_1*, a_1*, and b_1* are the lightness, red/green and yellow/blue coordinates of the sample's surface color, respectively, before heat treatment; and L_2*, a_2*, and b_2* are the lightness, red/green, yellow/blue coordinates of the sample's surface color, respectively, after heat treatment.

Wettability

To investigate the effect of heat treatment on the wettability of wood, a contact angle meter (Kyowa Interfacial Science, Japan) was utilized to measure the apparent contact angle of the wood samples before and after heat treatment. In the measuring procedure, distilled water was dispensed on the wood surface, and the measurements were then taken after a 15-s waiting period. The apparent contact angle measurements of the earlywood and latewood portions of the samples were also recorded.

Mechanical Properties

Bending tests

To determine the MOE and modulus of rupture (MOR) of the samples, a three-point bending test (Shimadzu AG-250KNI universal-type testing machine, Japan) was conducted with a span of 280 mm and a displacement-controlled loading speed of 5 mm/min. In this test, the samples were mounted so that the loading was applied on the radial plane. Subsequently, the MOE and MOR of each sample were determined as follows,

$$MOE\,(\text{GPa}) = (\Delta PL^3)/(4\Delta Ybh^3) \qquad (11)$$

$$MOR\,(\text{MPa}) = (3P_{MAX}L)/(2bh^2) \qquad (12)$$

where L, b, and h are the span, width, and thickness of the sample (mm), respectively; P_{MAX} is the maximum load (N); ΔP is the load difference (N) between the upper and lower boundary loads within the proportional limit; and, ΔY is the sample's mid-span deflection (mm) under ΔP.

Compression tests

To determine the compression properties of the heat-treated wood, samples sized 20 × 20 × 40 mm (T × R × L) were prepared and tested under a compression load with a displacement-controlled speed of 5 mm/min. The compression strengths of the samples were calculated as follows,

$$Compression\ strength\,(\text{MPa}) = P_{max}/A \qquad (13)$$

where $P_{\max}$ is the maximum compression load (N) and A is the cross-sectional area of the sample (mm^2).

Shear tests

Shear tests were conducted in accordance with the standard (CNS 455 2013) for the determination of ultimate shearing stress parallel to grain. The shear loading speed was 5 mm/min, and the equation used to calculate the parallel-to-grain shear strength (MPa) of the samples was as follows,

$$Shear\ strength\,(\text{MPa}) = V_{max}/A \qquad (14)$$

where $V_{\max}$ is the ultimate shear load (N) and A is the shear-loading area of the sample (mm^2).

Statistical Analysis

Analysis of variance, ANOVA, was conducted (α = 0.05) using SPSS 16 (IBM Corporation, USA) to evaluate the effect of temperature and duration of heat treatment on the selected properties of Japanese cedar wood. Significant differences between the mean values of the testing groups were determined using Tukey test (95% significance level).

RESULTS AND DISCUSSION

Physical Properties

The results of the EMC analysis (Table 1) indicate that the heat treatment induced a reduction in the EMC of the wood, and the samples treated at higher temperatures had lower EMC values. For example, the EMC of the samples treated at 170 °C for 4 h was found to be lower than that of the control samples, and the samples treated at 210 °C for 4 h had the lowest EMC (7.8%). Moreover, the EMC reduction of the samples treated under different temperature conditions increased with the increase in the treatment temperature and duration, and the largest MC reduction occurred at a heat treatment of 210 °C for 4 h.

The samples heat-treated under the different conditions exhibited reductions in density, and the extent of the reduction increased as the treatment temperature and duration increased (Table 1). The density reduction was primarily caused by water evaporation and wood degradation during the heat treatment process. The heat treatment induced a concurrent mass loss (ML) of the samples (Table 1). The ML was moderately low at 170 °C and was mostly caused by the evaporation of the water in cell walls and from certain wood extractives. However, the MLs of the samples suggested thermal degradation of chemical compounds (*e.g.*, hemicelluloses) as the treatment temperature increased.

Table 1. Equilibrium Moisture Content (EMC), Density, and Mass Loss (ML) of Japanese Cedar Wood Samples

Group	EMC (%)	Density (kg/m^3)	ML (%)
Control	13.1^a (0.2)	502^a (43)	0
170-1 h	12.3^a (0.1)	405^b (55)	8.2^e (1.2)
170-2 h	12.7^a (0.2)	390^{bc} (33)	8.3^e (2.2)
170-4 h	11.1^b (0.4)	407^b (58)	8.8^{de} (1.4)
190-1 h	11.3^b (0.2)	388^{bc} (50)	9.4^{de} (1.3)
190-2 h	9.4^c (0.2)	393^{bc} (41)	10.8^{cd} (3.1)
190-4 h	9.6^c (0.5)	384^{bc} (42)	12.0^c (4.0)
210-1 h	9.1^c (0.2)	375^{bc} (32)	13.0^c (4.9)
210-2 h	8.6^{cd} (0.3)	368^{bc} (40)	15.7^b (1.1)
210-4 h	7.8^d (0.2)	352^c (36)	18.5^a (0.5)

Note: The letters *a–d* denote statistically significant differences between groups having different letters, within a lengthwise difference, at the 0.05 probability level by Tukey's test. Values in parentheses represent the calculated standard deviation.

Given the anisotropic nature of wood, the heat-treated Japanese cedar wood samples exhibited different dimensional shrinkage behaviors along the three orthotropic directions (Table 2). The observed tangential shrinkage of the samples was 6.12% (control samples), 6.21% to 7.53% (170 °C samples), 5.39% to 6.60% (190 °C samples), and 4.14% to 5.21% (210 °C samples), and the lowest shrinkage (4.20%) occurred at a heat treatment of 210 °C for 4 h. This finding suggests that the heat treatment at high temperatures (> 190 °C) can induce a decline in the tangential shrinkage of wood, and higher treatment temperatures can lead to lower tangential shrinkage values. A similar trend was observed for the radial shrinkage of the samples, in that the samples treated at 210 °C for 4 h had the lowest shrinkage. Despite minor variations in the longitudinal shrinkage of the samples, the heat treatment exhibited no statistically significant effects on the longitudinal dimensions of the samples.

Table 2. Dimensional Shrinkage of Japanese Cedar Wood Samples

Group	Tangential (%)	Radial (%)	Longitudinal (%)
Control	6.12^{abc} (1.27)	4.29^{ab} (0.55)	0.25^{a} (0.37)
170-1 h	6.21^{abc} (2.30)	3.97^{bc} (0.39)	0.36^{a} (0.21)
170-2 h	7.53^{a} (0.73)	4.65^{ab} (0.25)	0.26^{a} (0.17)
170-4 h	7.01^{ab} (1.53)	5.64^{a} (0.97)	0.06^{a} (0.80)
190-1 h	6.60^{bc} (0.22)	4.04^{abc} (0.27)	0.30^{a} (0.27)
190-2 h	6.56^{bc} (0.15)	3.86^{bc} (0.17)	0.43^{a} (0.36)
190-4 h	5.39^{bc} (0.37)	4.72^{ab} (0.29)	0.28^{a} (0.29)
210-1 h	4.14^{c} (1.07)	4.29^{ab} (0.14)	0.23^{a} (0.12)
210-2 h	5.21^{bc} (0.18)	2.55^{cd} (0.47)	0.17^{a} (0.11)
210-4 h	4.20^{c} (0.22)	1.83^{d} (0.26)	0.20^{a} (0.25)

Note: The letters *a*–*d* denote statistically significant differences between groups having different letters, within a lengthwise difference, at the 0.05 probability level by Tukey's test. Values in parentheses represent the calculated standard deviation.

Table 3 presents the WA, VSC, and ASE values of the wood samples. In contrast to the control samples, the heat-treated samples exhibited lower WA values. Moreover, the WA of the samples showed an evident decreasing trend with increased treatment temperatures and durations. The samples treated at 210 °C for 4 h had the lowest WA (173.2%).

As presented in Table 3, the VSC of the samples was found to be 11.1% (control samples), 8.5% to 9.5% (170 °C samples), 7.3% to 7.9% (190 °C samples), and 5.7% to 6.7% (210 °C samples). The lowest VSC occurred to the samples treated at 210 °C for 4 h, indicating that the increase in the treatment temperature led to a decline in the wood's VSC.

The ASE data of the samples indicate that the treatment temperature had a significant effect on the ASE values. Specifically, the higher treatment temperatures caused a desirably higher ASE of the wood, and the highest ASE was occurred at a heat treatment of 210 °C for 4 h.

According to the hygroscopicity analysis, the EMC of the wood samples, which were conditioned at 65% and 95% RH for 72 h, decreased with increased treatment temperature, whereas the MEE increased under the same conditions (Table 4). For example, the samples treated at 210 °C and conditioned at 65% and 95% RH had significantly higher MEE values than those treated at 170 and 190 °C. In addition, the samples treated at 210 °C for 4 h showed the highest MEE values (*i.e.*, 37.95% and 31.24% for 65% and 95% RH, respectively). These results indicate that the heat treatment had a significant effect on improving the hygroscopicity of the wood and consequently enhancing the wood's dimensional stability.

Table 3. Water Absorption (WA), Volumetric Swelling Coefficient (VSC), and Anti-Swelling Efficiency (ASE) of Japanese Cedar Wood Samples

Group	WA (%)	VSC (%)	ASE (%)
Control	326.5^a (11.7)	11.1^a (0.2)	-
170-1 h	225.1^{bc} (3.9)	9.5^b (0.2)	20.3^f (0.2)
170-2 h	229.1^b (5.6)	9.3^b (0.1)	21.8^f (0.9)
170-4 h	225.1^{bc} (3.7)	8.5^c (0.1)	21.5^f (2.1)
190-1 h	205.0^{cd} (7.1)	7.9^{cd} (0.1)	28.5^e (0.7)
190-2 h	196.1^{de} (1.3)	7.6^d (0.2)	31.5^{de} (0.7)
190-4 h	188.8^{def} (6.8)	7.3^{de} (0.2)	36.0^c (1.4)
210-1 h	187.6^{def} (8.0)	6.7^{ef} (0.4)	48.0^b (1.4)
210-2 h	176.0^{ef} (5.7)	6.3^{fg} (0.1)	53.5^a (1.8)
210-4 h	173.2^f (1.6)	5.7^g (0.3)	54.0^a (1.1)

Note: The letters *a–d* denote statistically significant differences between groups having different letters, within a lengthwise difference, at the 0.05 probability level by Tukey's test. Values in parentheses represent the calculated standard deviation.

The depolymerization of carbohydrates in the wood samples, particularly the dehydration reaction of hemicelluloses, took place during the heat treatment, which reduced the number of hydroxyl groups in the wood (Boonstra and Tjeerdsma 2006). Because hemicelluloses have low molecular weights, the pyrolysis of hemicelluloses often occurs at low temperatures (167 to 187 °C) during the heat treatment process (Elder 1991). As a result, the water absorption capacity of the wood was reduced.

Furthermore, when the heating temperature reached the glass transition temperature (T_g) of the wood, the lignin present in the wood began to soften, thereby further reducing the inner stresses and producing a favorable anti-swelling performance in the wood (Hanhijärvi 1999).

Table 4. Equilibrium Moisture Content (EMC) and Moisture Excluding Efficiency (MEE) of Japanese Cedar Wood Samples

Group	40 °C, 65% RH		40 °C, 95% RH	
	EMC (%)	MEE (%)	EMC (%)	MEE (%)
Control	8.78^a (0.13)	—	19.3^a (0.14)	—
170-1 h	7.57^b (0.06)	13.85^d (0.70)	17.79^{cd} (0.12)	7.83^{de} (0.62)
170-2 h	7.47^b (0.12)	15.00^d (1.30)	18.71^b (0.10)	7.74^{de} (0.12)
170-4 h	7.34^b (0.25)	16.47^d (2.80)	18.12^c (0.14)	6.12^e (0.7)
190-1 h	7.42^b (0.16)	15.50^d (1.84)	17.46^d (0.03)	9.54^d (0.15)
190-2 h	6.81^c (0.02)	22.42^c (0.23)	16.27^e (0.06)	15.73^c (0.33)
190-4 h	6.45^{cd} (0.12)	26.57^{bc} (1.36)	16.51^e (0.29)	14.47^c (1.20)
210-1 h	6.13^d (0.20)	30.24^b (2.33)	15.33^f (0.32)	20.54^b (1.68)
210-2 h	6.28^d (0.04)	28.45^b (0.44)	14.86^f (0.13)	23.03^b (0.69)
210-4 h	5.45^e (0.36)	37.95^a (4.07)	13.27^g (0.35)	31.24^a (1.79)

Note: The letters *a–d* denote statistically significant differences between groups having different letters, within a lengthwise difference, at the 0.05 probability level by Tukey's test. Values in parentheses represent the calculated standard deviation.

The degradation temperature of the amorphous region of celluloses is approximately 200 to 240 °C. During the heat treatment process, the degradation of celluloses led to an increase in cellulose crystallinity (Bhuiyan and Sobue 2000) and consequently reduced the water absorption tendency of the wood. In addition, the naturally hydrophobic lignin started to exhibit a flow behavior at temperatures of 127 to 193°C and spontaneously formed a lignin film on the surface of cell walls, thereby further reducing the hygroscopicity of the wood (Hakkou *et al*. 2005).

Heat treatment can facilitate the formation of ether bonds between lignin-derived compounds and celluloses (Boonstra and Tjeerdsma 2006; Kocaefe *et al*. 2009), thereby reducing the amount of hydrophilic hydroxyl groups in wood. Rowell *et al*. (2009) also indicated that, during heat treatment process, the hemicellulose decomposition produces volatiles and furans, and polymerization reactions generate water-insoluble compounds, thereby inducing the reduction of wood hygroscopicity.

Surface Properties

The results of the color tests are summarized in Table 5. The results indicate that the negative ΔL^* values, which represent darkened wood surface colors, are associated with high treatment temperatures and long durations (as shown in Fig. 1). The samples heat-treated at 210 °C exhibited statistically significant L^* changes compared with the samples treated at other temperatures.

Table 5. Color Changes of Japanese Cedar Wood Samples

Group	Color parameters			
	ΔL^*	Δa^*	Δb^*	ΔE^*
170-1 h	-2.0^{c} (4.2)	-1.5^{a} (1.7)	1.8^{b} (2.3)	5.2^{b} (2.4)
170-2 h	-4.2^{bc} (2.8)	-0.4^{a} (2.4)	1.2^{b} (2.1)	5.7^{b} (1.8)
170-4 h	-5.5^{bc} (3.5)	-1.2^{a} (1.8)	1.1^{b} (2.3)	6.5^{b} (3.3)
190-1 h	-8.1^{bc} (5.2)	-1.4^{a} (2.0)	1.7^{b} (2.9)	9.6^{b} (3.7)
190-2 h	-10.5^{b} (4.6)	-0.9^{a} (1.7)	0.1^{ab} (2.5)	9.7^{b} (4.5)
190-4 h	-7.6^{bc} (4.5)	-2.7^{a} (2.1)	1.2^{b} (2.5)	9.2^{b} (3.4)
210-1 h	-18.1^{a} (6.3)	-2.3^{a} (4.4)	-1.4^{ab} (3.6)	19.0^{a} (6.5)
210-2 h	-20.9^{a} (6.8)	-1.1^{a} (2.1)	-2.7^{a} (2.5)	21.4^{a} (6.5)
210-4 h	-22.1^{a} (5.1)	-1.8^{a} (2.3)	-3.3^{a} (1.7)	22.6^{a} (4.9)

Note: The letters *a*–*d* denote statistically significant differences between groups having different letters, within a lengthwise difference, at the 0.05 probability level by Tukey's test. Values in parentheses represent the calculated standard deviation.

Most of the heat-treated samples exhibited negative a^* and b^* values, indicating that such samples had green and blue surface colors. The samples treated at 170 °C had relatively high b^* values, which could be attributed to the phenols on the wood surface turning into a grayish yellow color as a result of the low-temperature heat treatment (Yao *et al*. 2012). The color parameters (*i.e.*, L^*, a^*, and b^*) of the samples treated at temperatures greater than 190 °C had higher values because of chemical changes of wood components (Sundqvist and Morén 2002).

According to the ΔE^* analysis, the samples treated at the lower temperatures *(i.e.*, 170 and 190 °C) had $\Delta E^* < 10$, indicating no evident color changes, which was mainly attributable to the structural alteration of the polar extractives in the wood (Yao *et al.* 2012). The ΔE^* of the samples implied that the treatment duration had a moderate effect on the surface color change. Sundqvist (2004) reported that darkened wood colors at heat treatments of >210 °C, and concluded that such color changes were because of the hemicellulose degradation which produced particular components (*e.g.*, furfural) that can darken wood colors.

The cleavage of β-O-4 bonds and reduction of methoxyl contents of lignin, during the heat treatment process induced the production of condensation and oxidation products (*e.g.*, quinones), which also darken the wood colors (Bekhta and Niemz 2003; Wikberg and Maunu 2004).

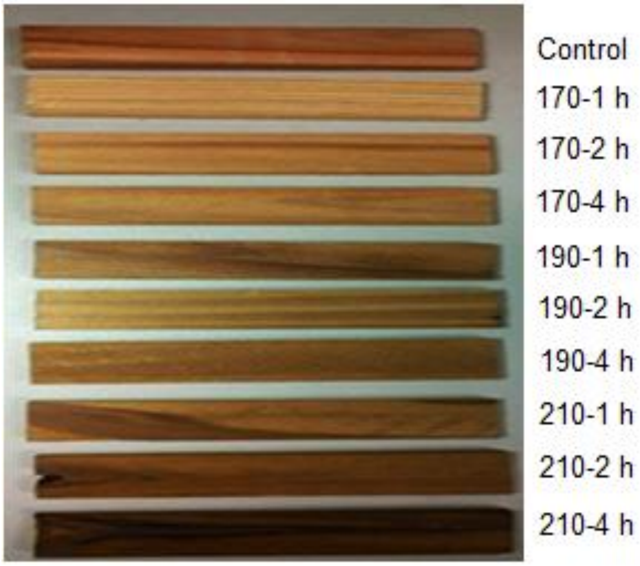

Fig. 1. Color Changes of the Japanese cedar wood induced by heat treatment

The heat treatment also promoted the optical absorption and color formation behaviors of the wood (Yamauchi *et al*. 2005; Windeisen and Wegener 2008; Yao *et al*. 2012). During the heat treatment process, the degradation of hemicelluloses released organic acids, which facilitated polysaccharide degradation (Sivonen *et al*. 2002). Minor wood charring also occurred during heat treatment with high temperatures, and the occurrence of charring further darkened the wood color.

During apparent contact angle analysis, no substantial differences were observed between the apparent contact angles of the earlywood and latewood samples of the untreated wood (Table 6). However, the heat-treated samples had greater apparent contact angles than the control samples did, and the increase in treatment temperature and duration had no substantial effects on altering the apparent contact angles of the samples, thereby indicating that low-temperature heat treatment can be used to effectively enhance the hydrophobicity of the wood.

This change of wettability may not be directly related to important chemical modifications like hemicelluloses decomposition (Hakkou *et al.* 2005). However, Hakkou *et al*. (2005) also revealed that the plasticization of lignin occurs at 160 °C in a heating process, and such plasticization can improve the hydrophobicity of wood. This finding was confirmed in this study. In the wood samples treated at low temperatures, the earlywood had slightly larger apparent contact angles than the latewood did. No statistically significant differences were discovered between the apparent contact angles of the earlywood and latewood samples.

Table 6. Apparent Contact Angle of Japanese Cedar Wood Samples

Group	Apparent contact angle(°)	
	Earlywood	Latewood
Control	57.5^b (2.1)	52.4^b (1.6)
170-1 h	87.5^a (1.3)	82.7^a (4.5)
170-2 h	90.0^a (1.2)	86.2^a (0.6)
170-4 h	83.3^a (5.1)	86.3^a (1.8)
190-1 h	89.7^a (0.8)	88.0^a (2.8)
190-2 h	87.4^a (0.6)	88.0^a (1.6)
190-4 h	87.6^a (0.8)	85.1^a (4.9)
210-1 h	87.0^a (2.3)	87.7^a (2.3)
210-2 h	83.7^a (7.5)	88.2^a (1.9)
210-4 h	87.7^a (2.4)	88.4^a (1.6)

Note: The letters *a*–*d* denote statistically significant differences between groups having different letters, within a lengthwise difference, at the 0.05 probability level by Tukey's test. Values in parentheses represent the calculated standard deviation.

Mechanical Properties

The results of the mechanical tests were presented in Table 7. Among all treatment groups, the samples treated at 170 °C for 1 h and 190 °C for 2 h had the highest MOE values, which were not significantly different from those of the samples in the control group, whereas the samples in the other treatment groups exhibited lower MOE values. This result agreed with several previous studies (Yildiz 2002; Borrega and Kärenlampi 2008; Mburu *et al.* 2008; Esteves and Pereira 2009; Korkut and Hiziroglu 2009).

Table 7. Mechanical Strength of Japanese Cedar Wood Samples

Group	MOE (GPa)	MOR (MPa)	Compression strength (MPa)	Shear strength (MPa)
Control	8.1^a (1.8)	70.7^a (10.2)	32.1^a (3.5)	8.2^a (1.4)
170-1 h	7.4^{ab} (2.9)	61.9^{ab} (20.3)	32.0^a (7.1)	7.5^a (0.7)
170-2 h	6.0^{cd} (1.5)	54.8^{bc} (12.3)	35.1^a (5.4)	7.1^{ab} (0.7)
170-4 h	6.4^{bcd} (2.4)	55.0^{bc} (21.1)	41.9^a (11.1)	6.7^{abc} (0.9)
190-1 h	6.0^{bcd} (2.4)	63.4^{ab} (17.4)	34.6^a (5.9)	5.3^{bcd} (0.8)
190-2 h	7.4^{abc} (1.8)	52.7^{bcd} (12.8)	32.5^a (2.5)	5.3^{bcd} (1.0)
190-4 h	5.4^c (1.4)	42.6^{cde} (13.8)	32.0^a (4.7)	5.1^{cd} (0.9)
210-1 h	5.8^c (1.6)	42.6^{cde} (14.6)	32.9^a (4.7)	5.3^{bcd} (0.9)
210-2 h	6.0^{cd} (1.2)	40.7^{de} (11.9)	38.7^a (3.8)	5.3^{bcd} (0.8)
210-4 h	6.2^{bcd} (1.5)	36.0^e (11.0)	35.8^a (5.7)	4.5^d (1.2)

Note: The letters *a*–*d* denote statistically significant differences between groups having different letters, within a lengthwise difference, at the 0.05 probability level by Tukey's test. Values in parentheses represent the calculated standard deviation.

The MOR values of the samples showed a declining trend as the treatment temperature and duration increased. The loss of strength may be attributed to embrittlement of fibers (Kamdem *et al.* 2002). Tomak *et al.* (2014) suggested that hemicellulose degradation during heat treatment processes induces MOR reduction of wood. Release of considerable organic acids such as formic and acetic acids during heat treatment processes also reduces celluloses in wood, thereby triggering decline in the MOR of wood.

According to Kocaefe *et al*. (2009), an, higher treatment temperatures (e.g., > 160 °C but < 210 °C) are associated with higher MOR and MOE values of wood; however, when the treatment temperatures reaches 210 °C, the MOR and MOE of wood decline substantially with increasing temperatures. Conversely, the present study revealed a declining trend in the MOE and MOR of the wood treated at the temperatures greater than 160 °C and lower than or equal to 210 °C.

The changes in the compression strength of the heat-treated wood samples exhibited no trends, but the average compression strength in each of the treatment groups was higher than that of the control group, indicating that the heat treatment effectively improved the compression strength of the wood. ThermoWood Handbook (2003) reported that the compression strength of spruce wood treated at 195 °C for 3 h increased by 30%, which is attributable to increasing crystallinity of celluloses and to crosslinking reactions occurring during the heat treatment process. Moreover, the shear strength of the heat-treated wood exhibited a declining trend as the treatment temperature and duration increased, and the lowest shear strength occurred at the heat treatment at 210 °C for 4 h. Generally, the effect of the heat treatment on the mechanical properties of the wood appeared to be very complex and varied depending on the temperature, duration, and medium of heat treatment as well as the MC and load-bearing conditions of the wood.

The overall effects of the heat treatment temperature and duration on the physical, surface, and mechanical properties of Japanese cedar wood are summarized in Table 8, associated with the ANOVA results. These results demonstrated that the treatment temperature imposes significant effects on the contact angle and compression strength of wood. Concurrently, the treatment duration influences a less number of properties than the treatment temperature. The results also indicated that significant effects of interaction of the temperature and duration may exist.

Table 8. Results of ANOVA for the Effect of Temperature and Duration of Heat Treatment on Various Properties of Japanese Cedar Wood

Properties	*p*-value		
	Temperature	Duration	Temperature x Duration
EMC	< 0.001**	< 0.001**	0.003**
Density	< 0.001**	0.456	0.260
ML	< 0.001**	< 0.001**	0.002**
Tangential shrinkage	< 0.001**	0.046*	0.290
Radial shrinkage	< 0.001**	0.476	< 0.001**
ASE	< 0.001**	< 0.001**	0.031*
MEE (65%)	< 0.001**	< 0.001**	0.003**
MEE (95%)	< 0.001**	< 0.001**	< 0.001**
ΔE^*	< 0.001**	0.345	0.627
Apparent contact angle (earlywood)	0.572	0.631	0.315
Apparent contact angle (latewood)	0.454	0.850	0.833
MOR	< 0.001**	< 0.001**	0.013*
MOE	0.015*	0.034*	< 0.001**
Compression strength	0.228	0.147	0.027*
Shear strength	< 0.001**	0.177	0.921
*: means significant effect; **: means highly significant effect			

CONCLUSIONS

This study investigated the changes in the properties of 35-year-old Japanese cedar wood induced by heat treatment processes conducted at various temperatures and for varying durations. The following conclusions were made:

1. The results of this study indicate that the heat treatment can effectively enhance the dimensional stability of wood.
2. Significant differences were found between the apparent contact angles of the heat-treated samples and those of the control samples.
3. Heat treatment at temperatures over 190 °C induced darkened surface colors, and the largest color difference ("ΔE^*") values occurred with the samples treated at 210 °C.
4. The heat treatment exhibited a statistically significant effect on the MOE and MOR values of the samples decreased with increasing treatment temperatures and durations.
5. Heat treated samples showed no significant difference from the untreated sample in compression strength. Furthermore, the heat treatment caused a decline in the shear strength of the wood, and the samples treated at 210 °C for 4 h had the lowest shear strength.

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