Comparison of the Color and Weight Change in *Paulownia tomentosa* and *Pinus koraiensis* Wood Heat-treated in Hot Oil and Hot Air

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Color changes were tested and compared for heat-treated *Paulownia tomentosa* and *Pinus koraiensis* wood treated with hot oil or hot air for further utilization of these species. Hot oil and hot air treatments were conducted at 180, 200, and 220 °C for 1, 2, and 3 h. Heat-treated wood color changes were determined using the CIE-Lab color system. Weight changes of the wood before and after heat treatment were also determined. The weight of the oil heat-treated wood increased considerably but it decreased in air heat-treated wood. The oil heat-treated samples showed a greater decrease in lightness (L^*) than air heat-treated samples. A significant change in L^* was observed in *Paulownia tomentosa*. The red/green chromaticity (a^*) of both wood samples increased at 180 and 200 °C and slightly decreased at 220 °C. The yellow/blue chromaticity (b^*) in both wood samples increased at 180 °C, but it rapidly decreased with increasing treatment durations at 200 and 220 °C. The overall color change (ΔE^*) in both heat treatments increased with increasing temperature, being higher in *Paulownia tomentosa* than in *Pinus koraiensis*. In conclusion, oil heat treatment reduced treatment duration and was a more effective method than air heat treatment in improving wood color.

Keywords: Air heat-treatment; Color change; Oil heat-treatment; Paulownia tomentosa; Pinus koraiensis; Weight change

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INTRODUCTION

Imported wood resources meet more than 80% of the wood demand in Korea. Domestic timber resources must be developed to overcome this requirement. Fast-growing wood species, including *Paulownia tomentosa* and *Pinus koraiensis*, are becoming essential species in plantation forests to increase domestic wood resources in Korea.

Paulownia wood is a genus of hardwood that is distributed extensively worldwide and is a fast-growing tree type (Caparrós *et al*. 2008). Akyildiz and Kol (2010) reported that *Paulownia* wood can be widely used as a raw material for construction, furniture materials, pulp, and handicrafts. *Paulownia* wood can also be utilized as a material for particleboard and paper production (Lopez *et al*. 2012), acoustic materials (Kang *et al*. 2019), and as a potential resource for bioenergy applications (Qi *et al*. 2016a,b).

Pinus koraiensis is the main plantation tree among softwood species for commercial utilization in Korea (Son *et al*. 2007). The wood is suitable for particleboard and fiberboard manufacturing (Chong and Park 2008) and has long been used as a fine quality furniture material in Korea (Son *et al*. 2001).

Paulownia tomentosa wood is noted for its pale white sapwood that is not clearly divided from its heartwood, while *Pinus koraiensis* wood has a light brown color in its heartwood and pale yellow to nearly white sapwood (Chong and Park 2008). The light color of these woods can be modified to acquire a darker color by heat treatment, which has esthetic advantages for some applications (Bekhta and Niemz 2003).

Air heat treatment is the most popular and well-established method for wood modification (Kesik *et al*. 2014; Lee *et al*. 2018). Heat treatment of wood in air can be applied to improve dimensional stability, hydrophobicity, durability, color, and equilibrium moisture content (Tjeerdsma and Militz 2006; Welzbacher *et al*. 2008; Hidayat *et al*. 2015, 2017a). Heat treatment can cause desirable or undesirable discoloration depending on process parameters, based on species type, processing temperature, duration of heating, and furnace atmosphere conditions (Bekhta and Niemz 2003; Hidayat *et al.* 2016; Sandoval-Torres *et al.* 2010).

Oil heat treatment is another method for improving wood properties through the synergetic effects of oil and heat (Ma'ruf *et al.* 2021). Oil is a suitable heating medium because of its ability to transfer heat to wood more efficiently and evenly (Umar *et al*. 2016). Oil heat treatment can also be used to upgrade wood quality for outdoor uses and obtain a uniform surface color (Sailer *et al*. 2000). Dubey *et al*. (2012) treated radiata pine wood in linseed oil at 180 °C for 3 h and found that oil heat treatment significantly decreased brightness but increased redness and yellowness.

As mentioned above, heat treatment using hot air or hot oil could be useful for enhancing wood color. In particular, air heat treatment could improve the color properties of *Paulownia tomentosa* and *Pinus koraiensis* to suit consumer preferences (Hidayat *et al*. 2017b). However, there is still no information on comparative studies on the effects of oil or air heat treatment on wood color change. To date, there have been no trial studies on the improvement of *Paulownia tomentosa* and *Pinus koraiensis* wood quality using oil heat treatment. Therefore, this study aimed to determine and compare the effects of heat treatment in oil and air on the color changes of *Paulownia tomentosa* and *Pinus koraiensis* wood to further utilize the fast-growing wood species in Korea.

EXPERIMENTAL

Materials

Three tree samples from *Paulownia tomentosa* and *Pinus koraiensis* were harvested from the research forest at Kangwon National University, Chuncheon, Korea. The basic information on the experimental trees is presented in Table 1. Logs converted into quarter-sawn boards, and straight-grained blocks free of defects were selected. The samples were oven-dried and conditioned at 25 ± 5 °C under a relative humidity of 50% to 65% until the equilibrium moisture content was reached.

Table 1. Basic Information on the Sample Trees

Common Name	Scientific Name	Age (years)	D.B.H (cm)	Density (g/cm³)	Location
Royal paulownia	*Paulownia tomentosa* (Thunb.) Steud.	15–20	28–33	0.30 (0.05)	Chuncheon, Korea (N 37°77′, E 127°81′)
Korean white pine	*Pinus koraiensis* Siebold & Zucc.	30–35	28–32	0.42 (0.05)	

*Numbers in parentheses are standard deviations

Heat Treatment

The oil heat treatment of wood was performed in a lab-scale oil bath (C-WHT-S2; ChangShin Science, Seoul, Korea) using commercial palm oil (Lotte foods, Korea). The major components of palm oil are palmitic acids (44-45%), oleic acids (39-40%), linoleic acid (10-11%), and only a trace amount of linolenic acid (Gunstone 2002). An electric furnace (Supertherm HT16/16; Nabertherm GmbH, Lilienthal, Germany) was used for heat treatment. The wood samples were heated at 180, 200, and 220 °C for 1, 2, and 3 h. In the air heat treatment, the temperature was raised to the target temperature at a heating rate of 2 °C/min. After heat treatment, the wood samples were allowed to cool down at room temperature, and then both heat-treated wood samples were dried in an oven at 105 °C for 24 h and kept in a desiccator with silica gel for a week until further testing.

Evaluation of the Heat-treated Wood Samples

Quarter-sawn boards (5 samples per treatment, n = 90) with dimensions of 160 mm (L) × 50 mm (R) × 30 mm (T) (Fig. 1a) were used to determine color changes. Straight-grained blocks (10 samples per treatment, n = 180) with dimensions of 40 mm (L) × 20 mm (R) × 20 mm (T) were used for measuring the weight change, as shown in Fig. 1b. Detailed sample information for evaluating the color and weight changes of the heat-treated wood is summarized in Table 2.

Color change was determined using the CIE $L^*a^*b^*$ system, characterized by three parameters: L^* (lightness), a^* (red/green chromaticity), and b^* (yellow/blue chromaticity). Three-point measurements on radial surfaces for each specimen were taken before and after heat treatment using a chromameter (CR-10 Plus; Konica Minolta, Tokyo, Japan), as shown in Fig. 1a. The overall color change was calculated using the following formula,

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

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

The weight change before and after heat treatment was calculated according to the following formula,

$$WC\ (\%) = \frac{m_1 - m_0}{m_0} \times 100 \quad (2)$$

where m_0 is the weight of the samples before heat treatment (g) and m_1 is the weight of the samples after heat treatment (g).

The weight changes between oil and air heat-treated wood with the treatment temperatures were statistically analyzed by a univariate analysis of variance, and significant differences between mean values were determined using Duncan's multiple range tests (SPSS version 24; SPSS Inc., Chicago, IL, USA).

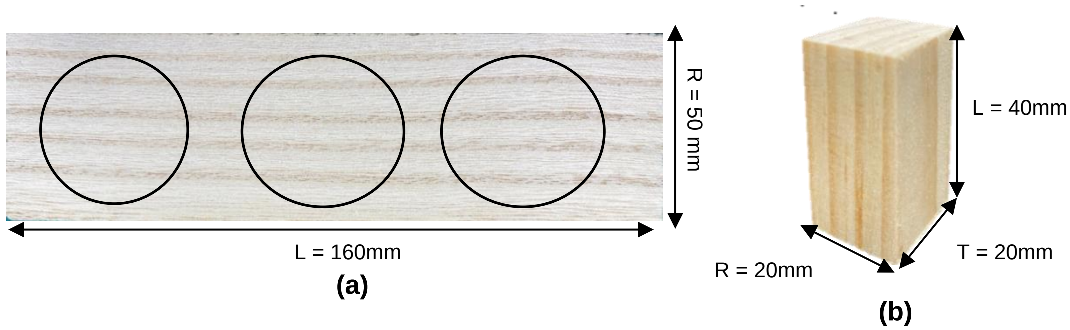


Fig. 1. Sample for color change measurement at three points (a) and weight change (b)

Table 2. Sample Information for Evaluating the Color and Weight Changes of the Heat-treated Wood Samples

Properties	Sample Dimension	Species	Treatment	Temperature and Duration	Sample Number	Total
Color change	160 (L) x 50 (R) x 30 (T) mm³	*Paulownia tomentosa*	Oil heat treatment	180 °C for 1,2,3 h	5 × 3	180
				200 °C for 1,2,3 h	5 × 3	
				220 °C for 1,2,3 h	5 × 3	
			Air heat treatment	180 °C for 1,2,3 h	5 × 3	
				200 °C for 1,2,3 h	5 ×3	
				220 °C for 1,2,3 h	5 × 3	
		Pinus koraiensis	Oil heat treatment	180 °C for 1,2,3 h	5 ×3	
				200 °C for 1,2,3 h	5 ×3	
				220 °C for 1,2,3 h	5 × 3	
			Air heat treatment	180 °C for 1,2,3 h	5 × 3	
				200 °C for 1,2,3 h	5 × 3	
				220 °C for 1,2,3 h	5 × 3	
Weight change	40 (L) x 20 (R) x 20 (T) mm³	*Paulownia tomentosa*	Oil heat treatment	180 °C for 1,2,3 h	10 ×3	360
				200 °C for 1,2,3 h	10 ×3	
				220 °C for 1,2,3 h	10 ×3	
			Air heat treatment	180 °C for 1,2,3 h	10 ×3	
				200 °C for 1,2,3 h	10 ×3	
				220 °C for 1,2,3 h	10 ×3	
		Pinus koraiensis	Oil heat treatment	180 °C for 1,2,3 h	10 ×3	
				200 °C for 1,2,3 h	10 ×3	
				220 °C for 1,2,3 h	10 ×3	
			Air heat treatment	180 °C for 1,2,3 h	10 ×3	
				200 °C for 1,2,3 h	10 ×3	
				220 °C for 1,2,3 h	10 ×3	

RESULTS AND DISCUSSION

Color Change

The appearances of *Paulownia tomentosa* and *Pinus koraiensis* wood before and after heat treatment in oil and air are shown in Figs. 2 and 3, respectively. The wood changed to a darker color with increasing temperature and duration.

Fig. 2. Appearance of *Paulownia tomentosa* (a) and *Pinus koraiensis* (b) wood during oil-heat treatment under different temperature conditions

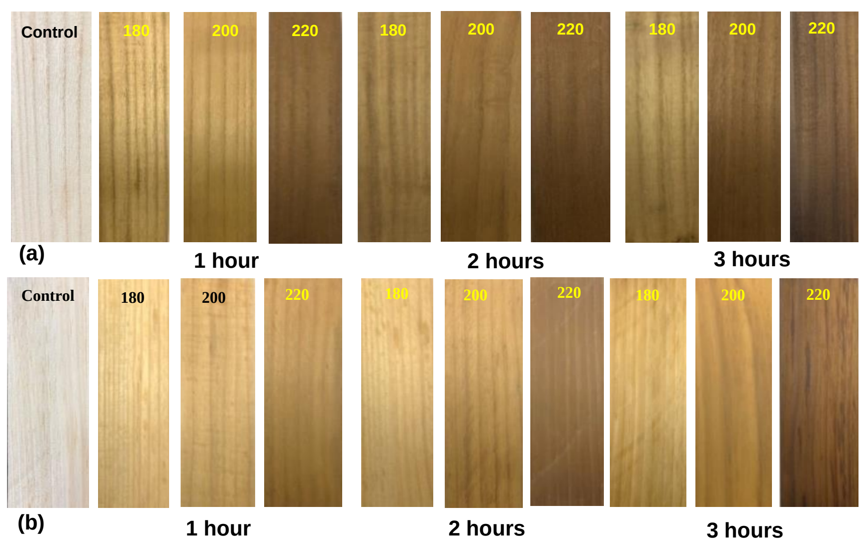


Fig. 3. Appearance of (a) *Paulownia tomentosa* and (b) *Pinus koraiensis* wood during air-heat treatment under different temperature conditions

The changes in lightness (L^*) after heat treatment in hot oil and hot air are shown in Fig. 4. In both *Paulownia tomentosa* and *Pinus koraiensis* wood, L^* decreased considerably as temperature and treatment duration increased. The air heat-treated samples showed a lower level of L^*-decrease than the oil heat-treated samples. *Paulownia* samples that were air heat-treated at 220 °C for 3 h showed a remarkably higher L^* decrease than the oil-heat-treated samples. After heat treatment, L^* also showed different responses between *Paulownia tomentosa* and *Pinus koraiensis* species, showing a higher decrease in *Paulownia tomentosa* than in *Pinus koraiensis* wood. Hidayat *et al.* (2017b) reported that L^* in air heat-treated *Paulownia tomentosa* and *Pinus koraiensis* wood gradually decreased as temperature increased. Similarly, Kim *et al.* (2018) reported that L^* in air heat-treated *Paulownia tomentosa* wood decreased with increasing temperature.

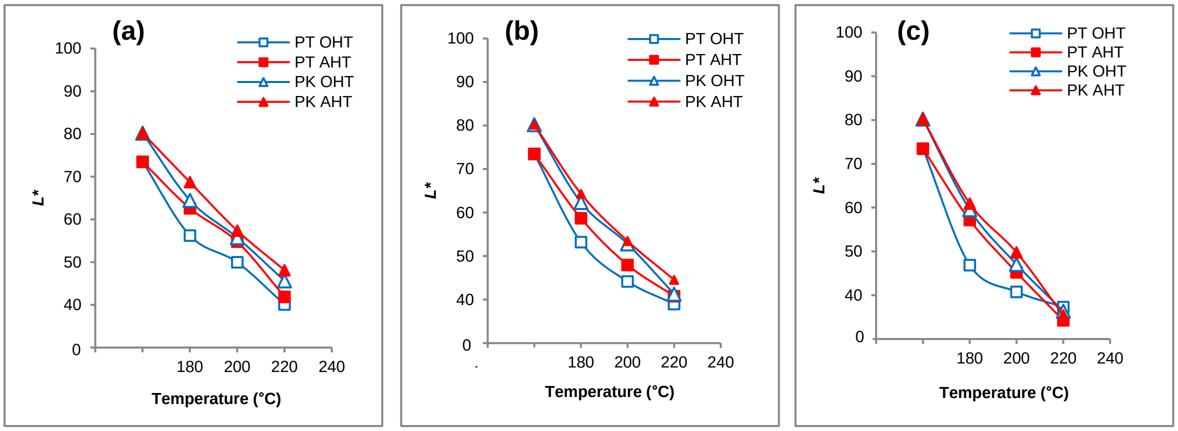


Fig. 4. Change in *L** of both wood species treated in hot oil and hot air for 1 (a), 2 (b), and 3 (c) hours. PT: *Paulownia tomentosa,* PK: *Pinus koraiensis,* OHT: oil heat treatment, AHT: air heat treatment

The changes in red/green chromaticity (*a**) after heat treatment in hot oil and hot air are shown in Fig. 5. In both wood species studied, *a** increased as the temperature and treatment duration increased. Red/green chromaticity in both wood species increased at 180 °C and 200 °C and decreased slightly at 220 °C. After 1 h and 2 h treatment time, the oil heat-treated samples exhibited a higher increase in *a** than air heat-treated samples, but the differences in *a** between the oil and air heat-treated samples at 3 h became small. The red/green chromaticity in *Pinus koraiensis* wood was higher than in *Paulownia tomentosa* wood. Bekhta and Niemz (2003) also observed that *a** of heat-treated spruce wood increased in the first 2 h of treatment and then remained practically constant with increasing time. Likewise, Kim *et al*. (2018) reported an increase in *a** in heat-treated *Paulownia tomentosa* and *Populus tementiglandulosa* wood.

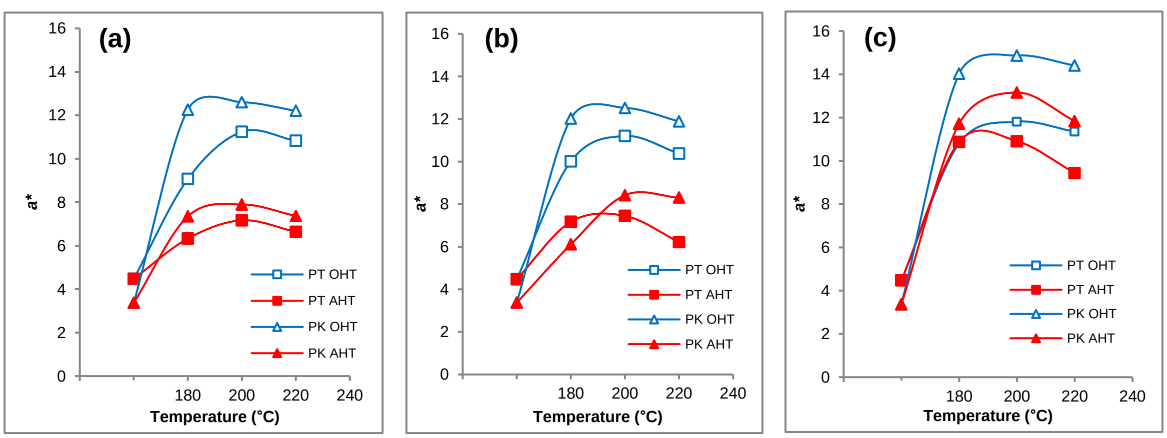


Fig. 5. Change in *a** of both wood species treated in hot oil and hot air for 1 (a), 2 (b), and 3 (c) hours. PT: *Paulownia tomentosa,* PK: *Pinus koraiensis,* OHT: oil heat treatment, AHT: air heat treatment.

Figure 6 shows the change in yellow/blue chromaticity (*b**) after heat treatment in hot oil and hot air. Yellow/blue chromaticity in both wood samples after heat-treatment increased at 180 °C but rapidly decreased in the treatments at 200 and 220 °C. In particular, the *b** values drastically decreased in the treatments after 2 and 3 h. There were considerable differences in *b** between *Pinus koraiensis* and *Paulownia tomentosa* wood. The yellow/blue chromaticity between the oil and air heat-treated samples was not remarkably different. After 1 and 3 h of treatments, the air heat-treated samples showed higher *b** than oil heat-treated samples, except for the samples after 2 h heat-treatment. Bekhta and Niemz (2003) indicated that the *b** of heat-treated *Picea abies* wood reached a maximum at 150 °C and decreased gradually until 200 °C. Correspondingly, Kim *et al*. (2018) observed that in air-treated *Paulownia tomentosa* wood, the *b** value increased from 160 to 200 °C and then decreased at 220 °C after 2 h of treatment. Furthermore, Hidayat *et al*. (2017b) examined the color change in air heat-treated *Pinus koraiensis* wood in a 160 to 220 °C temperature range and reported that *b** increased at 160 °C and gradually decreased thereafter.

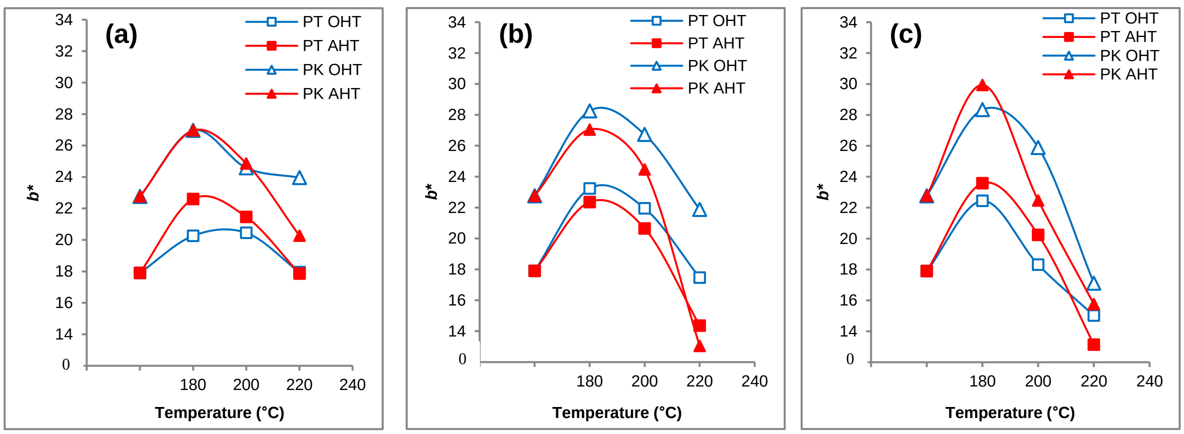


Fig. 6. Change in *b** of both wood species treated in hot oil and hot air for 1 (a), 2 (b), and 3 (c) hours. PT: *Paulownia tomentosa,* PK: *Pinus koraiensis,* OHT: oil heat treatment, AHT: air heat treatment.

Figure 7 shows the overall color change (Δ*E**) of both wood species treated in hot oil and hot air for 1, 2, and 3 h periods. The color change in both wood species increased with increasing temperature and treatment duration.

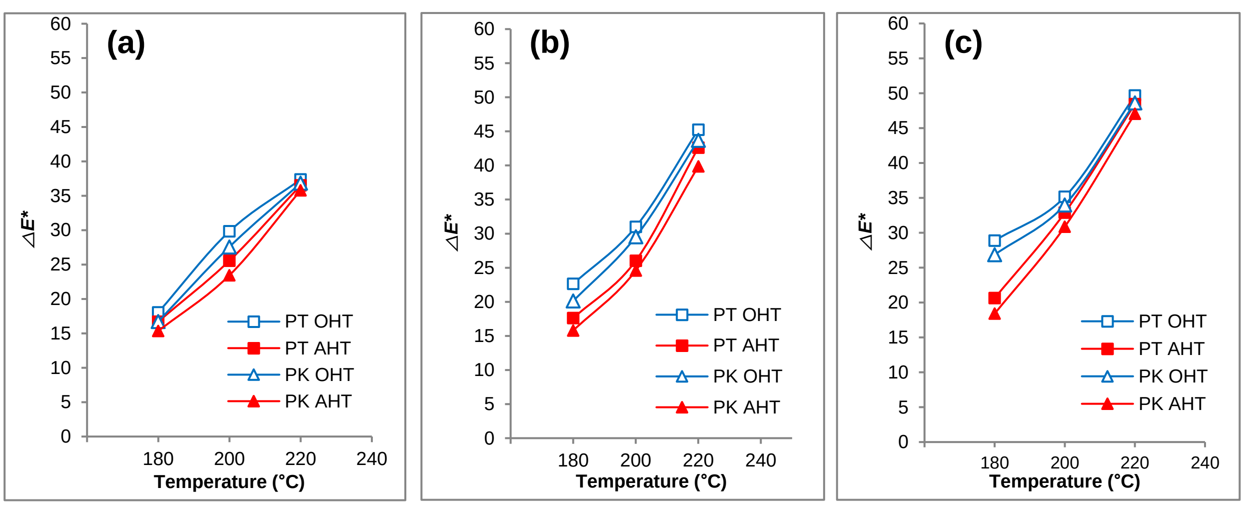


Fig. 7. Change in △*E** of both wood species in hot oil and hot air for 1 (a), 2 (b), and 3 (c) hours. PT: *Paulownia tomentosa,* PK: *Pinus koraiensis,* OHT: oil heat treatment, AHT: air heat treatment

The color change of oil-heat-treated wood was greater than in air-heat-treated wood. During oil heat treatment, the wood absorbs the oil and is exposed to high temperatures due to high boiling point of oil, causing some changes in the chemical properties of wood components (Umar *et al*. 2016). This might be one of the factors leading to darker color changes in oil heat treatment. Color change appears primarily due to decreased brightness during heat treatment associated with hemicellulose degradation, changes in extractive contents, and the formation of oxidation products (Bekhta and Niemz 2003; Dubey *et al.* 2012).

Moreover, there are some differences in color change between the species in the present study, showing higher ΔE^* in *Paulownia tomentosa* wood than in *Pinus koraiensis* wood. Sandoval-Torres *et al.* (2010) determined that hardwoods are more easily discolored than softwoods even at low temperatures.

Weight Change

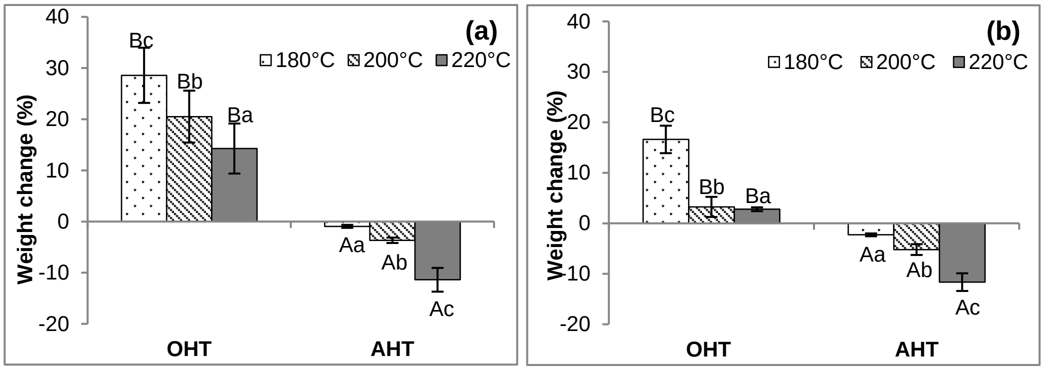
The weight changes in *Paulownia tomentosa* and *Pinus koraiensis* wood before and after heat treatments are shown in Figs. 8 and 9, respectively. The weights of *Paulownia tomentosa* and *Pinus koraiensis* wood after oil heat treatment significantly increased, whereas they significantly decreased following air heat treatment. The weight gain could be caused by the oil uptake by the wood sample after oil heat-treatment (Bazyar 2012; Dubey *et al.* 2012; Bal 2015).

Dubey *et al*. (2012) observed an increase in the weight of *Pinus radiata* wood attributed to oil absorption during oil heat treatment and a gradual decrease in weight with increasing temperature and heating duration. Bazyar (2012) also reported a weight percentage gain of 83.9% to 86.2% in *Populus tremula* wood following oil heat treatment at 190 to 220 °C for 4.5 and 6 h. Additionally, Bal (2015) reported similar findings in *Fagus orientalis* wood, showing a weight increase in oil heat treatment and weight loss in air heat treatment.

In the oil heat treatment, weight gain was highest at 180 °C and it significantly decreased with increasing temperature and treatment duration. A slight decrease of 2.9% in weight was only observed in *Paulownia tomentosa* wood subjected to oil heat treatment at 220 °C for 3 h.

In contrast, in the air heat treatment, weight loss was lowest at 180 °C and increased significantly as the temperature and time were increased. The highest weight loss was 17% in *Paulownia tomentosa* wood after treatment at 220 °C after 3 h heat treatment. Dubey *et al*. (2012) stated that the weight reduction following oil heat treatment indicated a change in the chemical composition of wood, with a remarkable decrease of 70% in hemicellulose content.

Srinivas and Pandey (2012) reported weight loss in *Hevea brasiliensis* and *Grevillea robusta* wood after air heat treatment with a maximum weight loss of approximately 18% following treatment at 240 °C for 8 h. They attributed the weight loss to the removal of bound water and extractives from wood. Hidayat *et al.* (2015) further ascribed wood weight loss to hemicellulose degradation during heat treatment.



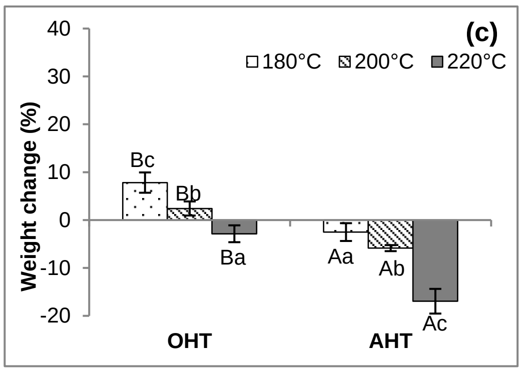
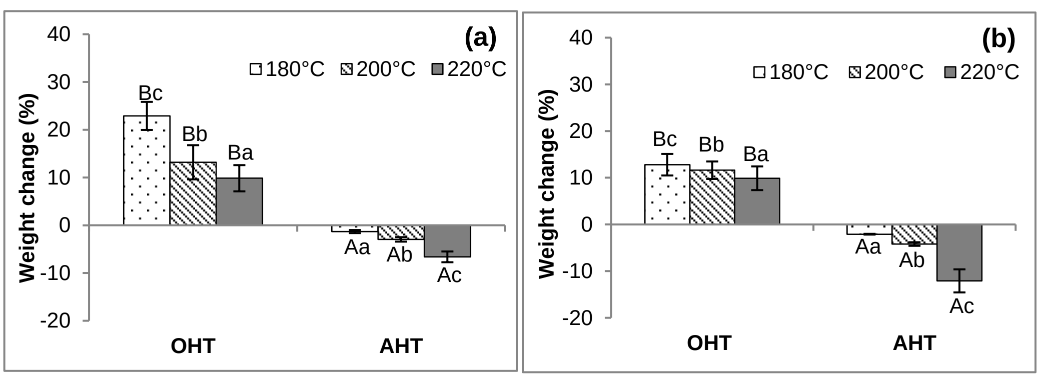


Fig. 8. Effect of temperature and treatment duration on the weight changes of *Paulownia tomentosa* after oil (OHT) and air heat treatment (AHT) for 1 (a), 2 (b), and 3 (c) hours. The different capital letters and lowercase letters indicate significant differences at the 5% significance level for comparisons between oil and air heat treatment and among the treatment temperatures, respectively, using Duncan's multiple range tests.



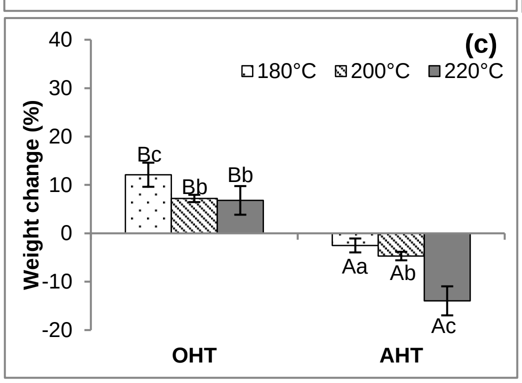


Fig. 9. Effect of temperature and treatment duration on the weight changes of *Pinus koraiensis* after oil (OHT) and air heat treatment (AHT) for 1 (a), 2 (b), and 3 (c) hours. The different capital letters and lowercase letters indicate significant differences at the 5% significance level for comparisons between oil and air heat treatment and among the treatment temperatures, respectively, using Duncan's multiple range tests.

CONCLUSIONS

1. The oil heat-treated samples showed a higher L^* decrease than the air heat-treated samples, showing higher values in *Paulownia tomentosa* than in *Pinus koraiensis*.
2. Red/green chromaticity in both wood species increased at 180 and 200 °C and slightly decreased at 220 °C, showing higher values in *Pinus koraiensis* than in *Paulownia tomentosa* wood.
3. Yellow/blue chromaticity in both wood samples treated in hot oil and hot air increased at 180 °C but rapidly decreased at 200 and 220 °C. The yellow/blue chromaticity drastically decreased after the 2 h and 3 h heat treatments, showing considerably higher values in *Pinus koraiensis* than in *Paulownia tomentosa* wood.
4. The oil heat-treated wood showed higher ΔE^* than the air heat-treated samples, with higher values in *Paulownia tomentosa* species than in *Pinus koraiensis* wood.
5. The weights of *Paulownia tomentosa* and *Pinus koraiensis* wood significantly increased after oil heat treatment but significantly decreased after air heat treatment. With increasing temperature and treatment durations, weight gain decreased in the oil heat treatment, whereas it increased with air heat treatment.

In summary, it was demonstrated that oil heat treatment is a more effective method than air heat treatment in reducing the duration of heat treatment and improving wood color even at low temperatures.

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