Effect of Mechanical Restraint on the Properties of Heat-treated Pinus koraiensis and Paulownia tomentosa Woods

By Wahyu Hidayat; Yue Qi; Jae Hyuk Jang; Fauzi Febrianto; Nam Hun Kim

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Wahyu Hidayat, a,b Yue Qi, a,c Jae Hyuk Jang, a,d Fauzi Febrianto, and Nam Hun Kim a,*

The objective of this study was to improve the properties of Korean white pine (Pinus koraiensis Sieb. & Zucc.) and royal paulownia (Paulownia tomentosa (Thunb.) Siebold & Zucc. ex Steud.) via heat treatment. The woods were treated at 160 °C, 180 °C, 200 °C, and 220 °C for 2 h. The effect of mechanical restraint through clamping during heat treatment on the dimensional stability, physical, and mechanical properties was evaluated. The results showed that increased temperature increased the weight loss and volume shrinkage, while equilibrium moisture content and wettability decreased. Royal paulownia showed higher weight loss, but lower shrinkage and equilibrium moisture content, when compared to Korean white pine. The samples with clamps in both woods had lower weight loss and volume shrinkage after heat treatment. The modulus of elasticity and modulus of rupture decreased with increased temperature followed by a noticeable decrease obtained after heat treatment at 200 °C and 220 °C. Clamping minimized strength reduction in both woods. Consequently, it was suggested that mechanical restraint was a useful method to maintain the wood properties during heat treatment.

Keywords: Clamping; Heat treatment; Korean white pine; Mechanical restraint; Physical-mechanical properties; Royal paulownia

Contact information: a: College of Forest and Environmental Sciences, Kangwon National University, Chuncheon 24341, Republic of Korea; b: Department of Forestry, Faculty of Agriculture, University of Lampung, Jl. Sumantri Brojonegoro 1, Bandar Lampung, 35145, Indonesia; c: Research Institute of Wood Industry, Chinese Academy of Forestry, Beijing 100091, China; d: Department of Forest Products, National Institute of Forest Science, Seoul 02455, Republic of Korea; e: Department of Forest Products, Faculty of Forestry, Bogor Agricultural University, Gd. Fahutan Kampus IPB Dramaga, Bogor 16680, Indonesia; * Corresponding author: kimnh@kangwon.ac.kr

INTRODUCTION

Wood production in plantations in Korea is important due to the depletion of wood resources from tropical forests and restriction in utiliation of natural forests. Korean white pine (*Pinus koraiensis* Sieb & Zucc.) and royal paulownia (*Paulownia tomentosa* (Thunb.) Siebold & Zucc. ex Steud.) are important species for use in plantation forests to improve domestic wood self-sufficiency in South Korea.

Korean white pine is a softwood species that grows in Korea, China, Russia, and a wareas on the Japanese islands of Honshu and Shikoku (Mirov 1967; Son et al. 2001). addition to high economic value of its edible seeds, the wood of Korean white pine from the tree has one been used for furniture in Northeastern Asia, especially in Korea (Son et al. 2001), where the species was extensively planted throughout the country in the last several decades (Korea Forest Service 1999).

Royal paulownia is a hardwood species that is widely grown all over the world, and it is known as a fast-growing tree (Caparros *et al.* 2008). The authors' have reported the anatomical and bioenergetics properties of royal paulownia (Qi *et al.* 2016a, 2016b) and revealed its potential application for bioenergy, particularly in the production of wood charcoal (Qi *et al.* 2016c). The wood also has a good dimensional stability, it can be easily air-dried without serious drying defects, has a low shrinkage coefficient, and does not easily warp or crack (Flynn and Holder 2001; Akhtari *et al.* 2012). However, the wood has a low density and low mechanical properties.

Some properties of wood can be improved by heat treatment. Heat treatment of wood increases the wood's value by altering its color (Hidayat *et al.* 2017), decreasing the equilibrium moisture content (Jamsa and 5 itaniemi 2001; Esteves and Pereira 2009), it proving dimensional stability (Viitaniemi *et al.* 1997; Esteves and Pereira 2009) and durability (Dirol and Guyonnet 1993; Kamdem *et al.* 2002), along with decreasing the heat transfer coefficient (Esteves *et al.* 2008). However, the treatment is detrimental to mechanical properties, especially to static bending strength, dynamic bending strength (Poncsak *et al.* 2011), and compressive strength (Unsal and Ayrilmis 2003)

In the authors' previous works, the properties of high density wood after heat treatment at different temperatures and durations were reported (Hidayat *et al.* 2015, 2016). The present work reports the results on the treatment of the low-density woods 13 rean white pine and royal paulownia. The effects of clamping and temperature during heat treatment on the dimensional stability, physical, and mechanical properties are evaluated.

EXPERIMENTAL

Materials

Trees from Korean white pine (*Pinus koraiensis* Sieb & Zucc.) and royal paulownia (*Paulownia tomentosa* (Thunb.) Siebold & Zucc. ex Steud.) were obtained from the research forest of Kangwon National University in South Korea (N 37°51' / E 127°48'). Boards from the two wood species were then prepared for heat treatment. For the evaluation of physical properties, boards with the dimensions of 300 mm (length) × 90 mm (width) × 20 mm (thickness) were 23°d. 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 with and without clamps (Fig. 1), using a similar method reported in the authors' previous studies (Hidayat *et al.* 2015, 2016).

Methods

Heat treatment

The prepared boards were heat-treated in air condition using an 10-ctric oven with a programmable coroller (L-Series, JEIO TECH Ltd., Seoul, Korea) at 48 0 °C, 180 °C, 200 °C, and 220 °C for 2 h, with a heating rate of 2 °C/min. In the final stage of the heat treatment process, the oven chamber was allowed to cool naturally until the boards reached 30 °C. Then, the boards were taken out and kept in a conditioning room under the relative humidity of $65\% \pm 3\%$ and a temperature of 25 °C ± 2 °C for 2 weeks until achievement of equilibrium moisture content.

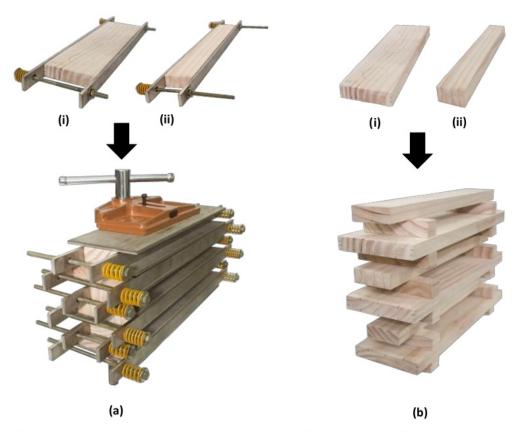


Fig. 1. Stacking of samples during heat treatment: (a) with a metal clamp, (b) without a metal clamp; samples for (i) physical properties, and (ii) mechanical properties evaluation

Board evaluation

Density, moisture content, and volume shrinkage of the samples were evaluated according to Korean standards KS F 2198 (2011), KS F 2199 (2019), and KS F 2203 (2009), respectively. Equations 1 and 2 explain how the weight loss (WL) was determined, by measuring the oven-dried weight of the sample (WL) and after heat treatment (WL), and how the volume shrinkage (WL) was determined by measuring the volume of the samples (WL) and after heat treatment (WL).

$$WL(\%) = 100 \times (m_1 - m_2) / m_1 \tag{1}$$

$$VS(\%) = 100 \times (V_1 - V_2) / V_1 \tag{2}$$

The density of the control and heat-treat 39 samples was determined by measuring its air-dry weight and volume after the boards were kept in a conditioning room with a relative humidity 29 65% at 25 °C for 2 weeks. The air-dry and oven-dry weights were measured using an attlytical balance (Sartorius AZ6101, Göttingen, Germany) with a sensitivity of 0.01 g to determine the moisture content of the control and heat-treated samples.

To measure the change in wettability, the dynamic conta 19 ingles of Korean white pine and royal paulownia before and after heat treatment were measured using a contact angle analyzer Phoenix 300+ (Surface Electro Optics, Suwon, South Korea) in the

42

National Institute of Forest Science, Seoul, Korea. The contact angle formed by a 5 mL droplet of liquid placed on the tangential surface of the samples 38 essile drop) was measured simultaneously after 1 s of drop until 60 s. The half-angle method was used to measure the contact angle (Fig. 2).

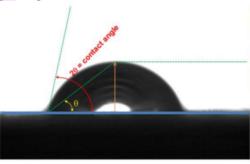


Fig. 2. Contact angle measurement using the half-angle method of drawing a line from the triphase point to the apex of the drop

Mechanical testing was conducted 12 an INSTRON 4482 universal testing machine (Instron, Norwood, MA, USA). The modulus of rupture (*MOR*) and modulus of elasticity (*MOE*) were determined by a three-point bending test in accordance with the 27 rean standard KS F 2208 (2009). Measurements were made using a constant loading speed of 1.5 mm/min and span length of 200 mm. The *MOR* and *MOE* values were determined according to Eqs. 3 and 4 below,

$$MOR (N/mm^2) = \frac{3 \times P \times L}{2 \times b \times t^2}$$
 (3)

$$MOE (N/mm^2) = \frac{P_p \times L^3}{4 \times Y_p \times h_{33}} t^3$$
 (4)

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).

The results of physical and mechanical properties evaluation were submitted to an overall analysis of variance (ANOVA) using a S16S 17 software package (New York, USA). The homogeneity of data was tested using Duncan's Multiple Range Tests with a significance level of 0.05.

RESULTS AND DISCUSSION

Physical Properties

The weight loss and volume shrinkage of Korean white pine and royal paulownia increased with increased treatment temperature (Table 1). The magnitude of weight loss in Korean white pine at the temperature of 220 °C was lower than that in royal paulownia. In contrast, the magnitude of volume shrinkage in Korean white pine was slightly higher than that in royal paulownia.

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Table 1. Physical Properties of Korean White Pine and Royal Paulownia before and after Heat Treatment

Density (g/cm³)	14/14	Clamp	0.43 ^A (0.03)	0.43 ^A (0.06)	0.43 ^A (0.01)	0.43 ^A (0.01)	0.42 ^A (0.02)	0.27 ^A (0.02)	0.27 ^A (0.01)	0.27 ^A (0.00)	0.27 ^A (0.01)	0.26 ^A
Densit	With		0.43 ^A (0.03)	0.43 ^A (0.02)	0.44 ^A (0.03)	0.43 ^A (0.01)	0.43 ^A (0.05)	0.27 ^A (0.02)	0.27 ^A (0.02)	0.27 ^A (0.02)	0.27 ^A (0.02)	0.26 ^A
	S	Without Clamp		2.00 ^A (0.20)	2.92 ^B (0.19)	4.41 ^c (0.38)	6.22 ^D (0.61)		2.10 ^A (0.29)	2.74 ^B (0.26)	4.33 ^{cb} (0.15)	5.50 ^{DE}
	NS	With		2.17 ^{AB} (0.05)	3.04 ^B (0.23)	4.60 ^c (0.16)	6.15 ^D (0.50)		2.14 ^A (0.20)	2.93 ^{BC} (0.20)	4.08 ^c (0.48)	5.17 ^D
	ST	Without		0.04 ^A (0.01)	0.14 ^B (0.00)	0.23 ^c (0.03)	0.38 ^D (0.04)		0.11 ^B (0.05)	0.15 ^{BC} (0.02)	0.26 ^c (0.05)	0.34 ^D
(%) abi		With		0.07 ^B (0.02)	0.16 ^B (0.03)	0.19 ^{BC} (0.04)	0.21 ^{BC} (0.05)		0.04 ^A (0.01)	0.11 ^B (0.04)	0.21 ^{BC} (0.06)	0.29 ^{CD}
Shrinkage (%)	RS	Without Clamp		0.37 ^A (0.12)	0.70 ^B (0.11)	1.50 ^c (0.12)	2.14 ^{DE} (0.19)		0.28 ^A (0.11)	0.68 ^B (0.14)	1.15 ^C (0.18)	1.75 ^{DE}
	<u> </u>	With Clamp		0.70 ^B (0.21)	0.99 ^{BC} (0.18)	1.99 ^D (0.26)	2.97 ^E (0.30)		0.53 ^{AB} (0.15)	0.94 ^{BC} (0.21)	1.43 ^D (0.19)	2.04€
	TS	Without		1.60 ^A (0.22)	2.09 ^B (0.27)	2.73 ^c (0.30)	3.80 ^D (0.40)		1.72 ^{AB} (0.27)	1.93 ^B (0.21)	2.97 ^{CD} (0.36)	3.49 ^D
	-	With		1.41 ^A (0.18)	1.90 ^B (0.23)	2.48 ^{BC} (0.40)	3.08 ^{CD} (0.29)		1.58 ^A (0.05)	1.90 ^B (0.19)	2.49 ^c (0.29)	2.92 ^{CD}
(%) SSOT	14/44/14	Clamp	•	1.08 ^A (0.20)	2.47 ^c (0.41)	5.06 ^E (1.10)	8.76 ^F (0.32)	,	1.27 ^A (0.14)	1.95 ^B (0.20)	5.29 ^D (0.41)	10.20F
Weight Loss	44,741	Clamp	•	1.07 ^A (0.44)	1.85 ^B (0.05)	3.67 ^D (0.26)	5.68 ^E (0.16)	,	1.40 ^{AB} (0.16)	1.78 ^B (0.19)	3.65 ^c (0.23)	7.71 ^E
	Temp.		Control	160 °C	180 °C	200 °C	220 °C	Control	160 °C	180 °C	200 °C	220 °C
	Species				Korean white pine					Royal paulownia		

Notes: The means are averages of 5 replicates. National parenthesis are standard deviations. Means within a physical property (including samples with and without clamp 422 by treatment temperature) followed by the same capital letter are not significantly different at 5% significance level using Duncan's multiple range test. TS = tangential shrinkage; RS = radial shrinkage; LS = longitudinal shrinkage; and VS = volume shrinkage

Previous studies reported that royal paulownia had a good dimensional stability and low shrinkage coefficient (Flynn and Holder 2001; Akhtari *et al.* 2012). The results also showed that samples with a clamp in both woods had a low 37 weight loss than in samples without a clamp. The results showed similar trends with heat-treated okan wood (Hidayat *et al.* 2015). The extent of thermal decomposition is often measured by mass loss. In accordance to the ThermoWood patent (Viitaniemi *et al.* 1997), a mass loss of 3% is needed to improve wood dimensional stability and at least 5% to improve urability.

In both woods, the volume shrinkage of samples with a clamp after heat treatment at 160 °C and 180 °C had slightly higher volume shrinkage (total shrinkage) compared to samples without a clamp. However, increasing the temperature to 200 °C and 220 °C, samples with a clamp resulted in slightly lower volume shrinkage than without a clamp. The results also showed that radial shrinkage in samples with a clamp were higher than without a clamp. During heat treatment with a clamp, low pressure applied during the fastening of that stacked boards might have affected the radial shrinkage. Esteves and Pereira (2009) stated that the reduction of dimension in heat treatment is caused by the chemical changes of the wood; or in other words, the basic components of the wood cell wall structure are changed in their number and dimension. The density of Korean white pine and royal paulownia was not noticeably affected by heat treatment. This might have been due to a balance reduction between weight and volume during the heat treatment.

Equilibrium moisture contents (EMC) for untreated Korean white pine and royal paulownia were 8.69% and 7.68%, respectively. Increased heating temperature remarkably reduced the EMC, which reached minimum values of 3.23% and 2.54% for Korean white pine and royal paulownia, respectively. The results showed that the EMC in Korean white pine samples before and after heat treatment were remarkably higher than that in royal paulownia (Fig. 3).

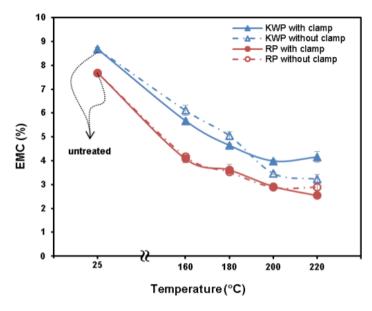


Fig. 3. Effect of temperature and clamping method on the change of EMC (KWP = Korean white pine; RP = royal paulownia)

A previous study by Metsa-Kortelainen and Viitanen (2012) reported that the EMC for spruce decreased after heat treatment from 10.6936 5.9% and from 10.1% to 5.4% for Scots pine. This decrease may have been a result of the increase in hydrophobicity of the cell wall, as a result of a decrease in the number of hydroxyl groups from the chemical reactions occurring in the heat treatment, resulting in less water adsorption (Jämsä and Viitaniemi 2001).

The clamping method affected the ENGC, particularly in Korean white pine. In Korean white pine, samples with the clamp and heat-treated at lower temperatures of 160 °C and 180 °C exhibited lower EMC than without the clamp. When the temperature increased to 200 °C and 220 °C, samples without a clamp showed lower EMC than samples with a clamp. The decrease in EMC might have been related to weight loss after heat treatment.

The results showed that the maximum reduction in EMC values were obtained 35 or heat treatment at 200 °C, which was associated to a weight loss between 3% and 5%. When the temperature increased to 220 °C the EMC values were constant, or not noticeably decreased (Fig. 3).

The results were in a good agreement with a previous study. Esteves *et al.* (2008) reported that after heat treatment of maritime pine (*Pinus pinaster* Aiton) at different temperatures, a mass loss between 4% and 6% was enough to obtain the maximum reduction in EMC, and a higher treatment severity did not benefit the EMC of wood. Hidayat *et al.* (2016) also reported that the weight loss after heat treatment is attributable to the decrease in the EMC, showing a strong linear relationship with the coefficient of determinations (R²) of 0.85, in which the higher weight loss resulted in lower EMC values.

The dynamic contact angles of Korean white pine and royal paulownia before and after heat treatment are shown in Fig. 4.

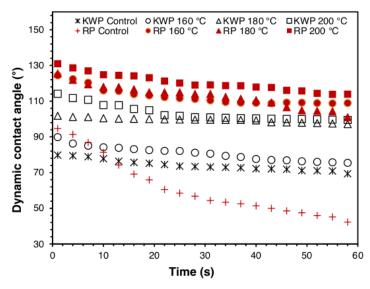


Fig. 4. Dynamic contact angle of Korean white pine (KWP) and royal paulownia (RP) before and after heat treatment

The results showed that the untreated royal paulownia had a higher contact angle at the initial stage of the drop test (1 s to 10 s), but decreased more rapidly than untreated Korean white pine, and showed the lowest contact angle of 42° at the end stage. While untreated Korean white pine showed a remarkably higher contact angle of 69° at the end stage of the drop test.

The higher contact angle in untreated Ko28 in white pine might have been due to extractive (resin) existence in the wood, similar to a previous study on maritime pine by Esteves *et al.* (2008) who stated that the extractive composition in the wood plays an important role on wood wettability (contact angle). In 11 mparison to the untreated samples, the dynamic contact angles of both woods after heat treatment showed a more gradual decrease from the beginning to the end stage of the drop test.

The heat treatment affected the secondary wettability of Korean white pine and royal paulownia. Wettability of both woods decreased after heat treatment, as shown by the increase of the contact angle after heat treatment (Fig. 4). A more remarkable increase in the contact angle was observed in royal paulownia compared to Korean white pine (Fig. 5 and Fig.6).

In Korean white pine, the contact angles gradually increased with the increase of temperature. The 14 ttability decrease (or the increase in contact angle) after heat treatment was due to the degradation of the most hygroscopic compounds, hemicelluloses, and amorphous cellulose, as well as dehydration reactions (Hakkou et al. 2005; Es 25 es et al. 2008). Esteves et al. (2008) also stated that in the heat treatment of pinewood, most of the original extractives disappeared and new ones were formed. The new extractives formed are mainly some phenolic compounds and anhydrosugars, resulting from the decrease of wettability, or the increase of the contact angle.

Time (s)	Control	160 °C	180 °C	200 °C
1		4		0
13	A	4	_	
28	A	4	1	
43	_	4	4	0
58		4	4	0

Fig. 5. Contact angles of Korean white pine before and after heat treatment

Time (s)	Control	160 °C	180 °C	200 °C
1	4		0	0
13	4		0	0
28	_		0	0
43			9	0
58			9	

Fig. 6. Contact angles of royal paulownia before and after heat treatment

Mechanical Properties

The results showed that Korean white pine had higher MOR values than royal paulownia, which was consistent with its higher density (Fig. 7). In Korean white pine imples with the clamp, the MOR increased after heat treatment at 160 °C and then decreased gradually with the increase of temperature, and reached the highest reduction of 10% at 220 °C. The reduction in samples without the clamp were higher, with 6% (160 °C), 12% (180 °C), 15% (200 °C), and 22% (220 °C). In royal paulownia, the MOR values linearly decreased with 10 reased temperature and reached 4%, 8%, 13%, and 23% reduction after heat treatment at 160 °C, 180 °C, 200 °C, and 220 °C, respectively. In royal paulownia, samples with and without clamp showed almost similar values.

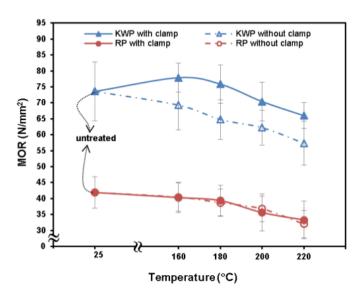


Fig. 7. Effect of temperature and clamping method on the change of MOR (KWP = Korean white pine; RP = royal paulownia)

The MOE values in Korean white pine samples without a clamp increased slightly after heat treatment at 160 °C and 180 °C, and reaching an overall increa 33 of 5% and 1%, respectively (Fig. 8). The values were then remarkably decreased after heat treatment at 200 °C and 220 °C, and reached a 13% and 14% reduction, respectively. From the results, 200 °C was a critical temperature to decrease the MOE of Korean white pine noticeably after heat treatment. Similar results were observed in the heat treatment of okan wood, showing a remarkable increase of MOR and MOE after heat treatment at 200 °C (Hidayat et al. 2015). Poncsak et al. (2006) treated birch wood (Betula papyrifera) and reported that the bending strength remarkably decreased after 200 °C. The results revealed that in the Korean white pine samples with a clamp was a similar trend but with a slightly lower magnitude of MOE reduction due to the smaller amount of weight loss in comparison to without a clamp. Clamping minimized the reduction of MOE, particularly after heat treatment at higher temperatures such as 200 °C and 220 °C.

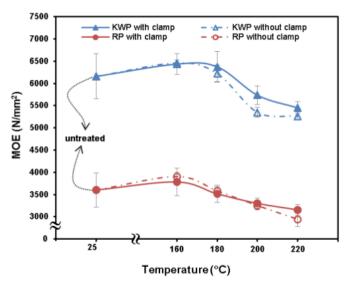


Fig. 8. Effect of temperature and clamping method on the change of MOE (KWP = Korean white pine; RP = royal paulownia)

Royal paulownia showed a similar trend of MOE changes as observed in 51e Korean white pine. The MOE values increased slightly after heat treatment at 160 °C, and then decreased at higher temperatures. The MOE reduction in royal paulownia was higher than in Korean white pine, which reached the highest reduction of 12% and 18%, after heat treatment at 220 °C with and without a clamp, respectively. Overall, the reduction of MOE in Korean white 15 ine and royal paulownia after heat treatment at 200 °C was still acceptable. Esteve 11 t al. (2008) stated that at the mass loss necessary to obtain the maximum improvement on the equilibrium moisture content and dimensional stability (between 4% to 6%), the decrease of MOE under 10% is not significant.

Overall, the reduction of MOR and MOI 32h Korean white pine and royal paulownia was lower in comparison to heat-treated okan wood (Hidayat *et al.* 2015) 21h the heat treatment of okan wood, the highest reduction of MOR was 31% and 49% for sapw 21 and heartwood, respectively, while the highest reduction of MOE was 15% and 27% for sapwood and heartwood, respectively. The lower strength reduction in Korean

white pine and royal paulownia might have been related to its lower wood density. The sapwood and heartwood of okan reported to have densities of 0.77 g/cm³ and 1.16 g/cm³, respectively (Hidayat *et al.* 2015), which were remarkably higher than the densities of Korean white pine and royal paulownia that had densities of 0.43 g/cm³ and 0.27 g/cm³, respectively.

CONCLUSIONS

31

- 1. The weight loss and volume shrinkage increased with increased heat treatment temperature. It was found that the weight loss and volume shrinkage in samples with a clamp were lower than without a clamp.
- 2. Heat-treated wood evidently absorbed 44 ss water than the control, which showed lower equilibrium moisture content. Heat treatment decreased the wettability of wood, and royal paulownia showed a higher degree of change in contact angles than Korean white pine.
- 3. An evaluation of the mechanical properties showed that the modulus of rupture and modulus of elasticity in both woods decreased with increased temperature. Clamping minimized the weight loss and strength reduction in both woods.
- Overall, the results showed that the application of mechanical restraint by clamping during heat treatment enhanced the properties of such woods for added value products.

ACKNOWLEDGEMENTS

This work was performed with the support of a research grant provided by the National Research Foundation of Korea (NRF-2016R1D1A1B01008339).

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Article submitted: April 4, 2017; Peer review completed: June 1, 2017; Revised version received and accepted: August 21, 2017; Published: August 30, 2017. DOI: 10.15376/biores.12.4.7539-7551

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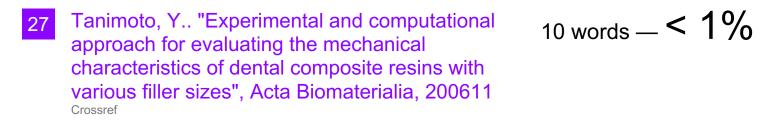
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