Effects of Heat Treatment on Physical-Mechanical Properties of *Eucalyptus regnans*

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Eucalyptus regnans was subjected to thermal treatment. The samples were placed in superheated vapors at 120, 130, 140, 150, 160, 170, 180, 190, and 200 °C at ambient pressure to determine the effect of heat treatment on the physical-mechanical properties of *Eucalyptus regnans*. The results showed that heat treatment played an important role in the impact toughness, nail-holding ability, surface hardness, bending strength, and bending modulus of elasticity of *E. regnans*. The nail-holding ability, surface hardness, bending strength, and bending modulus of elasticity of *E. regnans* treated at 120 and 130 °C increased related to untreated wood, but decreased at 140 to 200 °C. The impact toughness decreased after heat treatment from 120 to 200 °C, while the bending modulus of elasticity increased. Consequently, heating at 120 and 130 °C was found to be more suitable to ensure sufficient strength.

Keywords: Eucalyptus regnans; Heat treatment; Physical-mechanical properties

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INTRODUCTION

In Australia, *Eucalyptus regnans* is an important timber tree and is harvested from both natural and cultivated stands. The species is also cultivated overseas. Mountain ash (*E. regnans*) grows in cool-temperate parts of southeastern Australia and is arguably the world's tallest flowering plant, reaching a height of up to 110 metres (Peter and Jane 2001; Peter 2002). Its application is very extensive and can be used for heavy construction building, shipbuilding, pile structure, papermaking, door frames, and furniture production. Because of its light color and beautifully plain grain, the furniture made from this wood is attractive and popular with consumers. One disadvantage in the use of this species is its large deformation problem. Eucalyptus grows in tropical areas and the wood cell division is very active, so the growth stress is greater than for other trees commonly used as a source for furniture production. In addition, if the moisture content of *Eucalyptus* wood changes, the stress inside becomes uneven and leads to dimensional change that largely limits use of the wood (Huang *et al.* 2010). Consequently, increasing the dimensional stability of *E. regnans* is a useful way to improve the production quality.

Heat treatment is a type of green technology used to improve the dimensional stability of wood. However, it is well known that overly high temperatures may reduce the mechanical strength of wood based on the finding in previous studies (Korkut *et al.* 2008, 2013; Kesik *et al.* 2014; Pavel *et al.* 2014). For example, Pavel *et al.* (2014) determined a significant decrease of 47% in value of shear strength for thermally treated wood with natural wood when compared with natural oak – oak bonding. A similar result was pointed by Esteves *et al.* (2007) that heat treatment for 2 to 12 h at 190 to 210 °C

affected the mechanical strength of eucalyptus (the bending strength was reduced by 50% at 9% mass loss). The mechanical strength of *Eucalyptus regnans* has a direct influence on its use, so it is very important to choose a suitable temperature to ensure sufficient strength.

In this study, the effects of heat on the physical and mechanical properties of *E. regnans* were determined. Samples were placed in superheated vapors at temperatures from 120 to 200 °C at atmospheric pressure, and the effects of heat treatment on the physical-mechanical properties of *E. regnans* were analyzed. The results provide a theoretical reference to enhance the added value of *E. regnans* and to select the best heat treatment temperature in different situations.

EXPERIMENTAL

Materials

E. regnans trees, harvested in Tasmania, Australia, were 30 years old, with a 30-cm diameter at breast height and straight trunks with no eccentricity. Boards were cut from a 4-meter log from the bottom of the tree, and samples cut from the center of the quarter-sawn boards.

The dimensions of the samples were 450 mm x 120 mm x 25 mm. Eight replicates were treated at each heating temperature.

Methods

Determination of moisture content

Two 20 mm-wide pieces were cut from the ends of each 450 mm x 120 mm x 25 mm sample; each of the two pieces was cut into five test samples (20 mm x 20 mm x 20 mm). The test samples (20 mm x 20 mm x 20 mm) and the remainder of the corresponding boards (410 mm x 120 mm x 25 mm) were appropriately labelled. The test samples and remainder of the boards were deburred. The test samples were completely dried at a temperature of 103 ± 2 °C for 16 h in a 101-1 electric blower drying oven (Tianjin Taisite Instrument Co., Ltd., China). After drying, the moisture content of the test samples and the remainder of boards were calculated, as well as the absolute dry weight of the remaining samples (Tu 2010).

Heat treatment of wood

During the drying stage, the remaining samples (410 mm x 120 mm x 25 mm) were dried in a drying oven at 60, 70, 80, 90, and 100 °C for 10 h and then weighed. The equilibrium moisture content of the samples was calculated (Tu 2010). The final moisture content of the treated samples was reduced to 3 to 5% during the drying stage.

After the drying stage, the test samples (20 mm x 20 mm x 20 mm) and the remainder of the corresponding boards (410 mm x 120 mm x 25 mm) were preheated. Then the samples were heated to the target temperature (120, 130, 140, 150, 160, 170, 180, 190, or 200 °C) by a small, non-commercial coking furnace (Jiangyin Xing-Nan Drying Equipment Co., Ltd., China) during the heating stage. The samples were maintained at the target temperature for 3 h during the heat preservation stage, and then naturally cooled to 60 °C. All the parameters of the heat treatment are shown in Table 1.

Table 1. Parameters of Heat Treatment

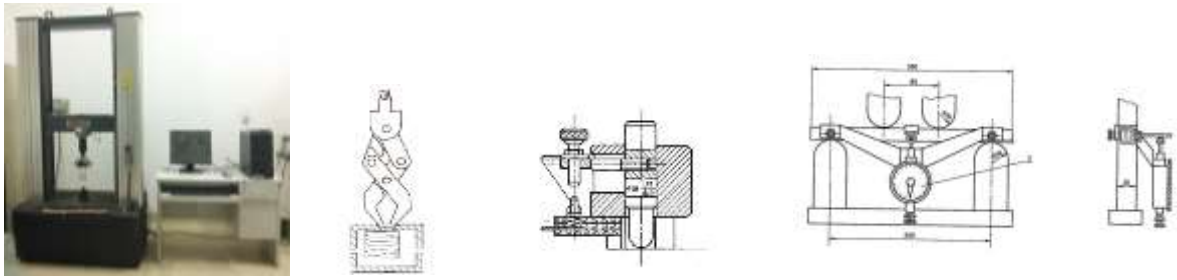
Procedure	Dry bulb temperature	Wet bulb temperature	Description
Drying stage	60 °C	45 ℃	The wood moisture content was reduced to 3 to 5%
	Increase to 120 °C at a rate of 20 °C/h	Wet-bulb depression remained 15 to 20 °C	
Preheating stage	120 °C	Increase quickly to 100 °C and maintain for 30 min	
Heating stage	Increase to 120, 130, 140, 150, 160, 170, 180, 190, or 200 °C at a rate of 15 to 20 °C/h	100 °C	
Heat preservation	120, 130, 140, 150, 160, 170, 180, 190, or 200 °C	100 °C	Moisturizing treatment was conducted for 3 h
Cooling stage	110 °C	100 °C	Heating stopped and a fan was employed
Refrigeration stage	110 °C	100 °C	Treated wood was naturally cooled to 60 °C
Dry bulb temperature	Reading on the dry bulb thermometer exposed to the air but not the direct rays of the sun		
Wet bulb temperature	Thermometer temperature sensor was surrounded by wet gauze, and the lower portion of gauze was in water to maintain air humidity of temperature sensor at saturation. Maintained air circulation around the gauze to get ambient air close to enthalpy. Stable reading on the thermometer was wet bulb temperature		

Determination of absolutely dry wood density

After the heat treatment, the samples (20 mm x 20 mm x 20 mm) were dried by a drying oven. Then the samples were weighed and measured by a micrometer (accuracy ± 0.001 mm) to determine the volume. The density of the samples was calculated.

Mechanical testing

According to the method of testing, the size of the samples in the impact toughness test (GB/T 1940 2009, nail-holding ability test (GB/T 14018 2009), surface hardness test (GB/T 1941 2009), bending strength test (GB/T 1936.1 2009), and bending modulus of elasticity test (GB/T 1936.2 2009) was required to be 300 mm×20 mm×20 mm, 150 mm×50 mm×50 mm, 70 mm×50 mm×50 mm, and 300 mm×20 mm×20 mm, respectively. Test devices are diagrammed in Fig. 1. Samples too thin for testing were glued together, and the glued could be neglected. All the samples were placed in a BPS-100CL constant temperature humidity chamber (Shanghai Yi-Heng Scientific Instrument Co., Ltd.; Shanghai, China) at 20 °C and 65% humidity for one week. Then all the samples were determined by a universal mechanical testing machine (Shimadzu Corp., Japan). After the test, the moisture content of testing sample was measured.



1 2 3 4

1--- Universal mechanical testing machine (Shimadzu Corp., Japan)
2--- The device for nail-holding ability testing
3--- The device for surface toughness testing
4--- The device for bending strength and bending modulus of elasticity

Fig. 1. Devices employed in the physical test procedures

Statistical analysis

Data from eight control samples data and eight treated samples from each mechanical test were then analyzed by Microsoft® Excel 2007 (Microsoft Corp.; Redmond, Washington). The mean data was illustrated in the figures and individual data for variance analysis.

RESULTS AND DISCUSSION

Whole Wood Density

Density is an important factor affecting the mechanical properties of wood. From Fig. 2, the whole wood density of treated wood decreased relative to the control with increasing temperature. Kamdem *et al.* (2002) found that after heat treatment there was an increase trend in lignin and a decreasing trend in carboxylic groups, which were abundant in the hemicellulose component. The presence of hemicellulose or fragile pentoses and hexoses became evident during the heat treatment. Therefore, the mass of the treated wood samples decreased, but not too sharply. The mass affected the density with a positive correlation.

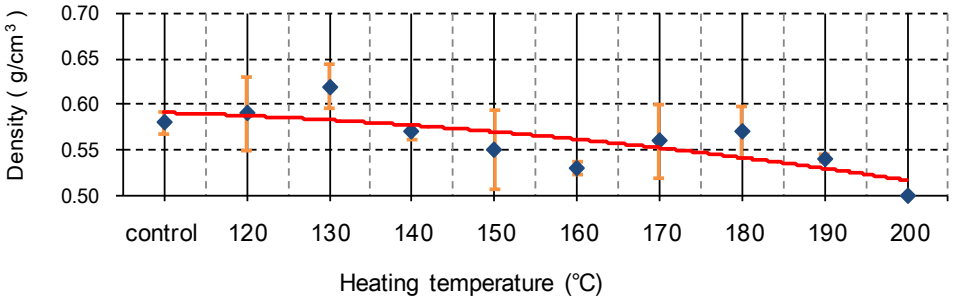


Fig. 2. The density of control sample and treated sample. Data provided as the mean ± standard deviation

Mechanical Properties

The variance analysis in Table 2 was calculated with mechanical property data of the treated samples from 120 °C to 200 °C. As Table 2 shows, when the value of F was larger than the F_{crit}, the heat treatment temperature had a remarkable influence on the test stability. The F values for impact toughness, nail-holding ability, surface hardness, bending strength, and bending modulus of elasticity were bigger than the F_{crit}. Therefore, in Table 1, heat treatment had a significant influence on the impact toughness, nail-holding ability, surface hardness, bending strength, and bending modulus of elasticity. Wood structure is anisotropic, so the physical and mechanical properties changed inconsistently as the heat treatment conditions changed.

Table 2. Variance Analysis of Treated Wood Samples in Different Temperature

Item		Impact toughness	Nail-holding ability	Surface hardness	Bending strength	Bending modulus of elasticity
Variance analysis	F	10.683940	10.988938	13.872728	20.451954	7.560010
	F crit	3.4248348	3.4248348	3.4248348	3.4248348	3.4248348
	P-value	1.541E-07	1.103E-07	6.066E-09	3.089E-11	7.083E-06
	α	0.005	0.005	0.005	0.005	0.005
Significance level		**	**	**	**	**

** denotes the relative significance at 99.5% confidence level between the property and heat treatment.

Impact Toughness

Variance analysis in Table 2 showed, at a level of $\alpha = 0.005$, that heat treatment had a significant (**) impact on the impact toughness of *E. regnans*. Figure 3 shows that the impact toughness of the treated wood was less than that of the control wood. The impact toughness of wood treated at 120 to 160 °C showed that as temperature increased, there was a decrease in a large range of impact toughness, about 51%; at 160 to 200 °C, the impact toughness tended to decrease slowly.

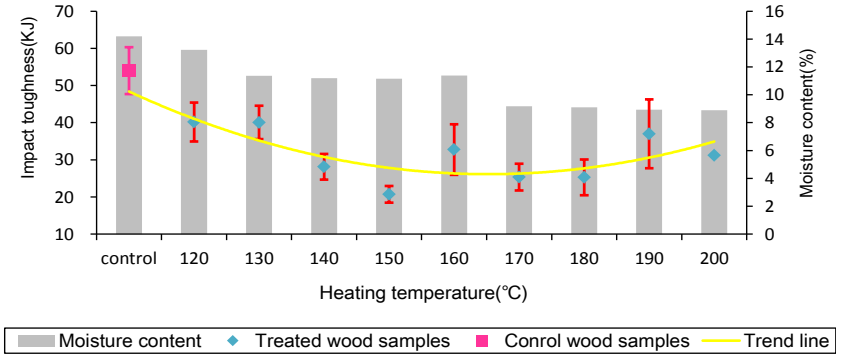


Fig. 3. Impact toughness of control samples and treated samples. Data provided as the mean ± standard deviation

Hillis (1984) pointed out that the decomposition of hemicellulose during high-temperature processing of wood causes the mechanical performance to decline. Yin (1996) suggested that the decomposition of hemicellulose caused wood to become

fragile, decreasing the impact toughness. The heat treatment temperature in this study was lower than that required for the degradation of cellulose molecules, but hemicelluloses, with poor thermal stability, may have become partially restructured or degraded. Also, lignin may have softened. After the degenerating hemicellulose cooled, the reorganized structure may have reinforced the mechanical properties. Therefore, the impact toughness decreased slowly in the range 160 to 200 °C.

Nail-holding Ability

Variance analysis in Table 2 showed, at a level of α = 0.005, that the heat treatment had a significant (**) influence on the nail-holding ability of treated wood. Figure 4 shows the nail-holding ability of treated wood at 120 to 130 °C was stronger than that of the control wood, but it was weaker at 140 to 200 °C. The nail-holding ability of *E. regnans* decreased with increasing temperature, but it slowed down at 150 to 200 °C.

The density positively affected the nail-holding ability. Results from a heat treatment study performed by the Institute of Environment Technology in 1999 showed that the major impact on screw holding strength was due more to variations in wood density than to heat treatment (Möller and Otranen 1999). But at 120 to 130 °C it is possible that during the loss of moisture in the cellulose amorphous region, the surfaces of the adjacent cellulose moved closer, thus cellulose molecular chains became more closely aligned and samples got harder. The extent of density reduction was weak.

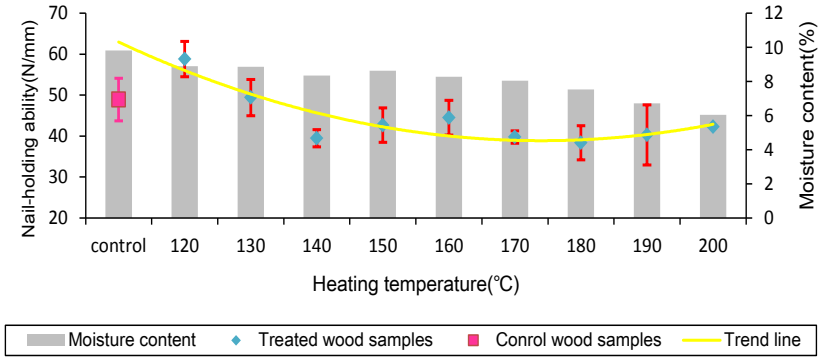


Fig. 4. Nail-holding ability of control samples and treated samples. Data provided as the mean ± standard deviation

Surface Hardness

Variance analysis in Table 2 showed, at a level of α = 0.005, that the heat treatment had a significant (**) influence on the surface hardness of treated wood. Figure 5 shows that surface hardness values of the treated wood were higher than that of the control at 120 to 130 °C, but weaker at 140 to 200 °C. Moreover, surface hardness value decreased with an increase in temperature. Surface roughness of wood can be affected by various factors, such as annual ring variation, wood density, cell structure, and latewood/earlywood ratio (Kilic *et al*. 2006). After the heat treatment, the loss of density occurred in the range 130 to 200 °C, which might affect the surface hardness.

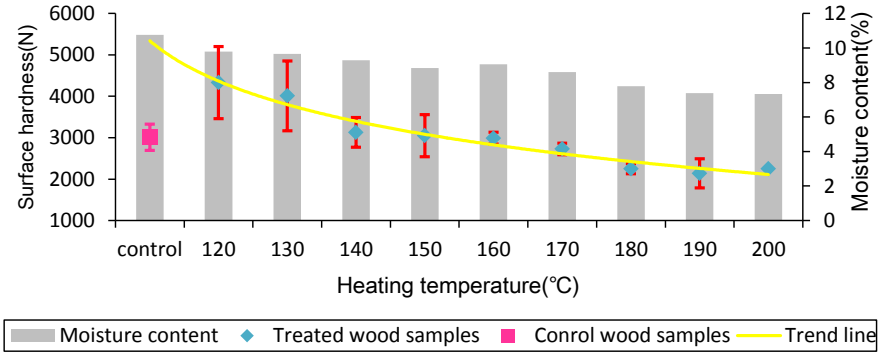


Fig. 5. Surface hardness of control samples and treated samples. Data provided as the mean ± standard deviation

Bending Strength

Variance analysis in Table 2, at a level of $\alpha = 0.005$, showed that heat treatment had a significant (**) influence on the bending strength of treated wood. Figure 6 shows that wood treated at 120 and 130 °C had stronger bending strength than that of the control wood, while wood treated at temperatures greater than 140 °C had decreased bending strength compared to the control wood. With increasing temperature, the bending strength of treated wood decreased. The decrease in bending strength of wood treated at 160 to 200 °C tended to level out.

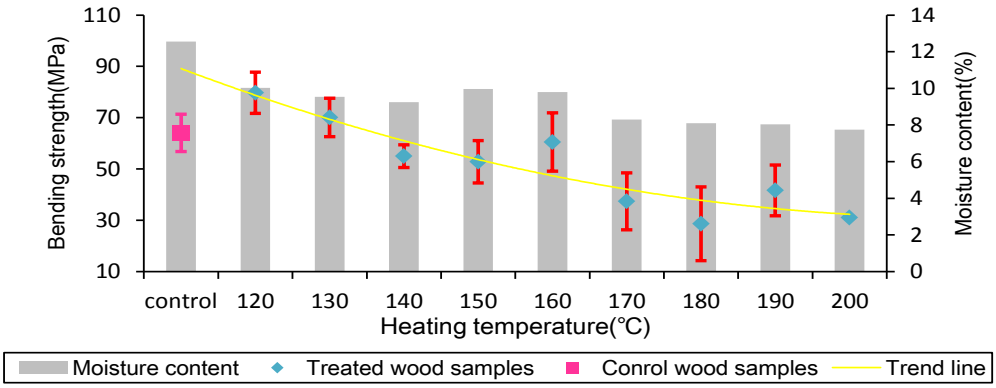


Fig. 6. Bending strength of control samples and treated samples. Data provided as the mean ± standard deviation

Wood bending strength is related to the intensity of the lateral coupling among wood fibers. High-temperature heat treatment softened lignin and degraded hemicellulose, which destroyed the combination and reduced the coupling strength of hemicellulose, lignin, and cellulose. The increase in broken coupling strength resulted in intercellular layer fracturing, which weakened the wood bending strength (Li *et al.* 2011; Shi *et al.* 2011a,b). At the same time, wood brittleness could also develop in response to the change in bending strength, *i.e.*, the brittleness increased as the bending strength decreased, and conversely, the brittleness decreased as the bending strength increased. Because in some sense, impact toughness test was a kind of special bending strength test that was under the condition of a high speed large instantaneous load. The high-temperature heat treatment, perhaps caused by the decomposition of cellulose, made

wood brittle; therefore, longitudinal tensile strength decreased, causing the loss of tangential bending strength (Li *et al*. 2009).

Bending Modulus of Elasticity

Analysis of variance in Table 2 shows that at α = 0.05, the impact of high-temperature heat treatment on the elastic modulus (MOE) of *E. regnans* was significant (**). The MOE of treated wood was greater than that of the control wood. Figure 7 shows that the MOE of treated wood increased slightly with increasing heat treatment temperature.

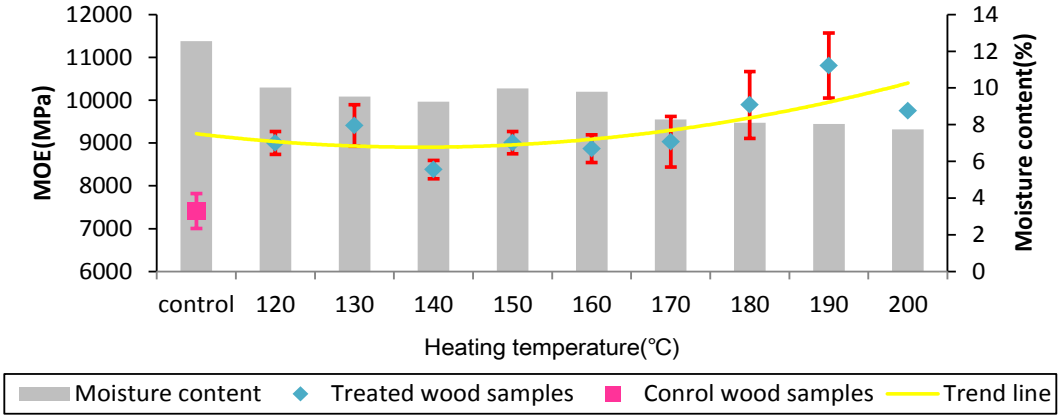


Fig. 7. MOE of control samples and treated samples. Data provided as the mean ± standard deviation

When the heat treatment temperature was low (less than 200 °C), the extractives and volatile components in the wood cell wall material might be lost. In addition, poor thermally stable hemicellulose could start partially restructuring or degradation, and the molecular chain of hemicellulose could have lost hydroxyl groups. It is possible that loss of moisture in the cellulose amorphous region and the surface of the adjacent cellulose moved closer, resulting in cellulose molecular chains being more closely aligned. Thus, the hydroxyl groups of cellulose molecular chains could have undergone a "bridging" reaction, leading to the formation of new hydrogen bonds. The mechanical strength of wood might increase as the crystallinity of cellulose molecules increased. Additionally, in this phase, the loss of cellulose and hemicellulose polysaccharide material could also cause the relative content of lignin to increase, leading to the improvement in the mechanical properties (Hill 2006; Liao *et al*. 2013).

Determination of the ideal treatment temperature relative to the dimensional stability of *E. regnans*, is a worthy goal for future research. The present results provide a basis upon which such a study can be designed. The goal would be to improve the dimension stability as much as possible, without changing or even enhancing the physical-mechanical properties of *E. regnans*.

CONCLUSIONS

1. The effects of heat treatment on the impact toughness, nail-holding ability, surface hardness, bending strength, and bending modulus of elasticity of *E. regnans* were significant (α = 0.005).

2. The nail holding ability, surface hardness, and bending strength of 120 and 130 °C heat-treated *E. regnans* increased in relation to the untreated wood, while processing temperatures of 140 to 200 °C caused these properties to be reduced. The bending modulus of elasticity value increased with the heat treatment from 120 to 200 °C, while the impact toughness was reduced. Therefore, heat treatment at 120 and 130 °C is more suitable to ensure sufficient strength.

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