EFFECT OF VACUUM HEAT TREATMENT TEMPERATURE ON PHYSICAL AND MECHANICAL PROPERTIES OF EUCALYPTUS PELLITA WOOD

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(Received January 2014)

Abstract. This study investigated how vacuum heat treatment influenced the physical and mechanical properties of Eucalyptus pellita wood. The investigated properties included mass loss, oven-dry density, dimensional stability, modulus of elasticity (MOE), and modulus of rupture (MOR). For the study, wood samples were heated under vacuum atmosphere at temperatures ranging from 80 to 280°C for 4 h. The results showed that although the mass loss of wood showed only a slight change below 200°C, there was a sharp increase in loss after 240°C. Oven-dry density of wood decreased slowly with an increase of treatment temperature with decreases of 4.4 and 10.4% compared with the control sample observed at 200 and 240°C, respectively. Conversely, the dimensional stability of samples increased by about 30% at 200°C. As temperatures increased, MOE and MOR initially showed gradual enhancements before declining rapidly. Compared with the untreated sample, MOE increased by 25.2% at 200°C, whereas MOR augmented by 6.5% at 160°C. Vacuum heat treatment temperature between 160 and 200°C would be available for improving dimensional stability and keeping mechanical strength of Eucalyptus pellita wood.

Keywords: Eucalyptus pellita, vacuum heat treatment, physical property, modulus of elasticity, modulus of rupture.

INTRODUCTION

As one of the world’s fastest growing tree species, Eucalyptus pellita has been widely planted in the southern area of China for use in the furniture industry. Eucalyptus pellita wood has a number of desirable traits, including its attractive color, high density, strength, and hardness. However, poor dimensional stability limits its wider application in industry. Therefore, there is an urgent need to determine appropriate methods for improving the dimensional stability of Eucalyptus pellita wood and also maintain its mechanical properties as much as possible.

Heat treatment modification of wood properties began as a discipline in the early 1920s, when Tiemann showed that drying at high temperatures decreased the equilibrium moisture and the consequent swelling of wood. Lots of heat-treated wood products have been developed since that time (Kollmann 1936; Seborg et al 1945). However, because of the availability of
high-quality alternative woods, none of the previous products had much commercial success. Recently, with declining production of durable timber and increasing demand for sustainable building materials as well as tightening government restrictions on the use of toxic chemicals (Boonstra 2008), there has been a renewal of interest in heat treatment research. Previous studies have shown that heat treatment is one of the most effective methods for improving dimensional stability, because it leads to permanent changes in the chemical components and physical structure of wood (Burmester 1973; Esteves et al 2008). However, previous research has also shown that results are highly dependent on the treatment process and parameters adopted (Bourgois and Guyonnet 1988; Gosselink et al 2004). Parameters that play an important role in determining final wood properties include the treatment temperature, duration, and thermal media (Militz and Tjeerdsma 2001). Nitrogen (Hofmann et al 2008), steam (Ding et al 2010), or hot oils (Manoj et al 2012) are usually used as heat transfer and protection mediums during heat treatment. Although heat treatment improves dimensional stability, it also decreases the mechanical strength of wood (Shi et al 2007; Esteves and Pereira 2009).

Compared with heat treatment, which requires a thermal media, vacuum heat treatment works by transferring thermal energy by thermal radiation. Because of the absence of a thermal media, vacuum heat treatment is more environmentally friendly and has lower energy requirements. Furthermore, previous research has demonstrated that vacuum heat treatment causes less color darkening (Wang et al 2001; Srinivas and Pandey 2012) as well as lower declines in mechanical property values (Wang et al 2011) compared with air. However, there is still little available research on vacuum heat treatment across a range of treatment applications. Therefore, more studies are needed to fully determine the effect of vacuum heat treatment on physical, mechanical, and chemical properties of heat-treated wood. The aim of this study was to research the effect of vacuum heat treatment on properties of Eucalyptus pellita wood, with the specific objective of identifying optimal parameters for vacuum heat treatment based on testing modulus of rupture (MOR), modulus of elasticity (MOE), weight loss ratio, oven-dry density, and dimensional stability of treated wood.

MATERIALS AND METHODS

Material

Six-yr-old Eucalyptus pellita wood was obtained from the Eucalyptus Research Center in Guangdong Province, China. Eucalyptus pellita wood samples were oven-dried below 80°C before vacuum heat treatment. Three hundred testing specimens with dimensions of $330 \times 40 \times 40$ mm (longitudinal $\times$ tangential $\times$ radial [L $\times$ T $\times$ R]) were prepared from the oven-dried wood. Specimens were divided into two groups. One group was heat-treated at 80, 120, 160, 200, 240, and 280°C for 4 h (time for temperature increasing not included) under $-0.08$ to $-0.09$ MPa, and the other group was the control.

The vacuum heat treatment equipment, HJ-ZK60, produced by Guangzhou Hengjun Company, which has a minimum pressure of $-0.09$ MPa and a maximum temperature of 350°C, was used to treat the wood. The equipment consists of two parts, a processing box and a vacuum pump. During treatment, the vacuum pump was used to achieve vacuum conditions and a heating cord in the processing box helped to achieve a high-temperature environment. There were two layers of walls in the processing box with the heating cords located between the two layers. Therefore, when the cords were heating, wood in the vacuum pump section was also being heated.

Testing Methods

Mass loss. Specimens with dimensions of $20 \times 20 \times 20$ mm (L $\times$ T $\times$ R) were cut from wood samples. The weight of all specimens was recorded before and after treatment. All the weight data were measured in dry condition. Thirty replications were tested for each
temperature or control with the average value and standard deviation calculated.

**Oven-dry density.** Specimens with dimensions of $20 \times 20 \times 20$ mm ($L \times T \times R$) were cut from wood samples. Oven-dry density of untreated and treated wood was tested according to Chinese standard (2009b). Besides weight, specimen volume data were also recorded in dry condition.

Thirty replications were tested for each temperature range as well as for the control, and the average value and standard deviation were also calculated.

**Dimensional stability.** Specimens were cut from treated and untreated blocks with dimensions of $20 \times 20 \times 20$ mm ($L \times T \times R$). To evaluate dimensional stability, antishrinking efficiency ($A_{SE_1}$, moisture state from maximum to oven-dry) and antiswelling efficiency ($A_{SE_2}$, moisture state from oven-dry to maximum) of wood was measured according to Chinese standard (2009a, 2009c). Formulas used to calculate $A_{SE_1}$ and $A_{SE_2}$ follow:

$$A_{SE_1} = \frac{V_{c,shrinking} - V_{t,shrinking}}{V_{c,shrinking}} \times 100 \quad (1)$$

$$A_{SE_2} = \frac{V_{c,swelling} - V_{t,swelling}}{V_{c,swelling}} \times 100 \quad (2)$$

where $V_{c,shrinking}$ is volume shrinkage ratio of control sample, %; $V_{t,shrinking}$ is volume shrinkage ratio of heat-treated sample, %; $V_{c,swelling}$ is volume swelling ratio of control sample, %; and $V_{t,swelling}$ is volume swelling ratio of heat-treated sample, %.

As before, 30 replications were tested for each temperature range as well as for the control with average value and standard deviation then calculated.

**Mechanical properties.** MOE and MOR were measured according to Chinese standard (2009d, 2009e). A three-point bending load method was used for specimens with dimensions of $300 \times 20 \times 20$ mm ($L \times T \times R$). During MOE testing, measurements were made using a constant velocity of 2 mm/min with the upper and lower load being 300 and 700 N, respectively. Tests were repeated three times. During MOR testing, a constant velocity of 5 mm/min was used until the specimens ruptured after 1-2 min. MOE and MOR were determined according to the following equations:

$$MOE = \frac{23PL^3}{108bh^3f}$$

$$MOR = \frac{3P_{\text{max}}L}{2bh^2}$$

where $P_{\text{max}}$ is the load at failure, N; $P$ and $f$ are any load and their corresponding displacement below the proportional limit, N and mm, respectively; $L$ is the span length, mm; and $b$ and $h$ are width and height of the specimen, respectively, mm.

The moisture content of testing samples was 12%. Again 30 replications were tested for each temperature as well as for the control with average value and standard deviation then calculated.

**RESULTS AND DISCUSSION**

**Mass Loss and Oven-dry Density**

Mass loss of *Eucalyptus pellita* wood treated under different temperatures for 4 h is shown in Fig 1. Mass loss ranged from 1.4 to 25.5% when

![Figure 1. Mass loss at different heat treatment temperatures.](image)
temperature increased from 80 to 280°C. Mass loss increased slowly as the temperature increased from 80 to 200°C but increased rapidly at temperatures greater than 200°C. Mass loss was less than 2.2% below 160°C before rising to 4.6% at 200°C, 13.3% at 240°C, and 25.5% at 280°C. This result is comparable with previous studies. For example, Esteves et al (2007b) found that mass loss of *Eucalyptus globules* heated in steam had a positive correlation with temperature. When treated for 6 h, mass loss of 7.6% was reported at 190°C, and it increased to 10.2% at 200°C and 13.9% at 210°C. Mass loss of *Eucalyptus globules* Labill wood also increased with temperature when heated in air with mass loss of 5.5% observed at 200°C (Esteves et al 2007a). This is a bit higher than the loss reported in our study at the same temperature. This was probably because of the fact that heat is mainly transferred by thermal radiation in a vacuum environment, whereas thermal conduction, convection, and radiation all exist when treated in air. Wood treated in vacuum treatment receives less thermal energy than that treated in air, which causes slightly less thermal decomposition, thus leading to decreased mass loss. Also, in a heat treatment, in which air is present, oxygen also reacts with the components of the cell wall, leading to greater loss of mass.

Figure 2 shows oven-dry density of samples treated at different vacuum heat treatment temperatures. It generally shows a downward trend as temperature increases. The decrease ratios of treated samples compared with the control were 0.2% (80°C), 0.8% (120°C), 2.1% (160°C), 4.4% (200°C), 10.4% (240°C), and 16.5% (280°C), respectively. At 160°C or lower, the change ratio was less than 2.2%, and at 240°C or higher, oven-dry density decreased by more than 10%. The phenomenon of wood density decline after heat treatment is very common. Unsal and Ayrimis (2005) found the air-dry density of eucalyptus wood decreased as temperature increased and treatment times were prolonged. Air- and oven-dry density of redbud maple wood also decreased by 0.16 and 1.4%, respectively, when treated at 120°C for 2 h (Korkut and Guller 2008). The density decrease ratios of beech and spruce were 2.25 and 1.73%, respectively, when heat-treated at 130°C for 2 h (Yildiz et al 2006).

There are two possible causes for the decrease of heat-treated wood density. Hemicellulose in wood could be easily affected by thermal radiation, even at low temperature. The degradation starts by deacetylation, and the released acetic acid as a depolymerization catalyst further increases polysaccharide decomposition (Nuopponen et al 2005). Conversely, most of the extractives disappeared during the heat treatment, especially the more volatile components (Yin 1996). Both of these two factors could cause the mass loss observed in heat-treated wood as well as explain the decrease in density.

**Dimensional Stability**

Testing results of ASE\(_1\) are shown in Fig 3. ASE\(_1\) went up as temperature rose and changed gradually from 80 to 160°C but varied obviously from 200 to 240°C. When heat treatment temperature rose from 240 to 280°C, ASE\(_1\) changed little from 65.72 to 70.00%.

Figure 4 shows the testing results of ASE\(_2\). The changing trend of ASE\(_2\) was similar to that of ASE\(_1\). ASE\(_2\) increased as temperature increased, showing a gradual change from 80 to 160°C followed by a significant rise from 200 to
280°C. However, when temperature rose from 240 to 280°C, ASE₂ changed little, increasing from 63.8 to 68.7%.

Studies show that heat treatment is an effective way to improve the dimensional stability of wood. ASE augmented by 50% at 170°C for *Pinus pinaster* and by 77% at 190°C for *Eucalyptus globulus* wood (Esteves et al. 2006). When treated by oil at 200°C, ASE of *Pinus sylvestris* wood increased by about 43% (Rapp 2001). There are two main reasons that explain why the dimensional stability of wood increases under heat treatment. First, hydroxyl groups, the main hygroscopic groups in wood, decrease because of a dehydration reaction of hemicellulose under the thermal effect (Weiland and Guyonnet 2003; Rowell et al. 2009). Second, cellulose crystallinity increases because of degradation of amorphous cellulose under the thermal effect with content of amorphous cellulose decreased. This results in a decreased accessibility of hydroxyl groups to water molecules (Wikberg and LiisaMaunu 2004; Boonstra and Tjeerdsma 2006).

**Mechanical Properties**

Figure 5 shows MOE of *Eucalyptus pellita* wood treated at different temperatures. Compared with the control, MOE increased as temperature went up until 200°C. The increase ratios were 1.6% (80°C), 13.6% (120°C), 14.1% (160°C), and 25.2% (200°C), respectively. The MOE value at 240°C or greater was not calculated because the samples were broken. Our results show different trends for MOE compared with previous studies. Table 1 shows some results from various studies on MOE resulting from heat treatment. In some cases, MOE increased slightly when temperature was below 200°C but decreased evidently as temperature increased or treatment times were prolonged.

Figure 6 shows that MOR also demonstrated a positive trend with increasing temperature until 160°C. However, MOR only increased slightly before 160°C and decreased significantly after
As temperature increased, the changing ratios of heat-treated samples compared with the control were 2.5% (80°C), 4.8% (120°C), 6.6% (160°C), 31.2% (200°C), 66.8% (240°C), and 82.9% (280°C), respectively. In previous studies, MOR mainly decreased following heat treatment. Table 2 summarizes other data on strength changes in heat treatment of woods/bamboo. In most cases, there was a major decrease in MOR with rising temperature, especially above 200°C (Poncsák et al. 2006). Both MOE and MOR increased below 160°C, but the increased ratio of MOE was generally higher than that of MOR, except at 80°C. MOE reached its maximum value at 200°C, whereas MOR showed a downward trend for the first time at this temperature. When at 240°C or above, both MOE and MOR were significantly decreased. Regarding the different effect of heat treatment on MOE and MOR, Mitchell (1988) also found there was a greater loss in MOR than in MOE.

At 200°C or below, increasing MOE may be caused by the decrease of moisture content within the FSP, which enhanced rigidity and led to an increase of MOE (Zhang et al. 2013). Furthermore, part of the amorphous cellulose in wood partially crystallized, which increased the wood stiffness (Kubojima et al. 2000; Windeisen et al. 2007). The increasing MOR was probably caused by a decrease of moisture content within the FSP (Yin 1996). However, the decomposition of the chemical constituent under thermal effect leads to a relative decrease of MOR at 200°C or above.

**CONCLUSIONS**

Vacuum heat treatment had a significant effect on properties of *Eucalyptus pellita*. The weight and oven-dry density decreased, whereas dimensional stability increased; the MOE and MOR increased gradually at first and declined obviously at higher temperatures across a range from 200°C.
80 to 280°C for 4 h at a pressure of −0.08 to 0.09 MPa.

Mass loss augmented and oven-dry density of wood lessened as heat treatment temperature increased; change ratios of the two were small at first, becoming more marked after 200°C. Compared with untreated samples, mass loss of treated samples was 4.6% at 200°C and 25.5% at 280°C. Furthermore, compared with the control, oven-dry density of treated samples decreased by 4.5% at 200°C and 16.6% at 280°C.

Vacuum heat treatment was an effective way to improve dimensional stability of wood, especially at 200°C or above. ASE₁ and ASE₂ were 32.1 and 28.4%, respectively, at 200°C.

As temperature increased to 200°C, MOE of wood increased gradually and reached the maximum value of 18.67 GPa, which was 25.2% greater than for untreated samples. However, above 200°C, MOE decreased sharply. MOR grew slightly before 160°C and got to the maximum value of 118.97 MPa at 160°C. This represents an increase of 6.5% compared with the control. Above 160°C, MOR dropped significantly. Vacuum heat treatment at 160 and 200°C would be suitable for the general properties of Eucalyptus pellita wood.

ACKNOWLEDGMENTS
This work was funded by the Chinese National Natural Science Foundation “Thermal effect and mechanism of wood under vacuum heat treatment,” Grant No. 31370558, and China’s “12th Five-Year Plan” to support science and technology project “Design, Building and Evaluation of Bamboo/Wooden Demonstration Room,” Grant No. 2012BAD23B0105.

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