Increased Dimensional Stability of *Eucalyptus grandis* × *Eucalyptus urophylla* 'GLGU9' Wood through Palm Oil Thermal Treatment

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Eucalyptus grandis × *Eucalyptus urophylla* 'GLGU9' is one of the most commonly planted tree species in South China. It is a new variety created by Guangxi Zhuang Autonomous Region Forestry Research Institute. As a fast-growing species, the poor dimensional stability is one of its main drawbacks, which restricts its applications. Thermal treatment is one of the effective methods to improve the dimensional stability of wood. GLGU9 wood was treated using thermal modification with palm oil. The oil was used as a heating medium and a shielding material at temperatures of 150, 170, 190, 200, and 210 °C, at various treatment durations of 1.5, 3, 4.5 and 6 h. To investigate the effect of palm oil thermal treatment on dimensional stability, the anti-shrink efficiency (ASE_1) and anti-swelling efficiency (ASE_2) were examined. The results indicated that the ASE_1 and ASE_2 were increased by 62.8% and 56.6% at 210 °C for 6 h treatment, respectively.

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Keywords: Eucalyptus grandis × E. urophylla 'GLGU9' wood; Thermal treatment; Palm oil; Anti-shrink efficiency; Anti-swelling efficiency

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INTRODUCTION

The oil heat treatment of wood, which is one of five industrialized wood modification technologies – Thermowood process in Finland (Jämsä and Viitaniemi 2001; Syrjänen and Oy 2001; Aytin *et al.* 2022), Oil-heat treatment in Germany (Rapp and Sailer 2001), Plato process in Netherlands (Militz and Tjeerdsma 2001), and Le Bois Perdure and Retification process in France (Vernois 2001) – was started by the Menz Holz company from Germany. The heating medium usually is crude vegetable oil, such as rape seed, linseed oil, or sunflower oil (Rapp and Sailer 2001; Wang and Cooper 2005), where the oils offer excellent heat transfer characteristics and separates the oxygen from the wood. The boiling points of many natural oils are higher than the temperature required for the thermal modification of wood. Generally speaking, the efficiency of the oil treatment is dependent on the type of oil that is used as a heating medium (Okon *et al*. 2017; Lee *et al.* 2018; He *et al.* 2022; Ruwoldt and Toven 2022). The oil heat treatment has significant effect on wood properties, especially for dimensional stability, water uptakes capacity, and chemical components (Dubey *et al*. 2012; Humar and Lesar 2013; Aytin *et al.* 2022; Kaya 2023).

It was reported that palm oil is more effective than soybean oil in improving the radial and tangential dimensional stabilities of oil heat-treated white spruce at 220 ℃ for 2 h (Wang and Copper 2005). Furthermore, they also found that the treatment seemed to be more efficient in improving the radial rather than the tangential dimensional stability, though this result is opposite to the findings by Tjeerdsma *et al.* (1998a). Palm oil heat treatment is also more effective method for improving the properties of *Paulownia tomentosa* and *Pinus koraiensis* wood; the palm oil heat-treated wood exhibited lower volume shrinkage and weight loss at temperature range of 180 to 220 °C for 1 to 3 hours compared to air-heat-treated wood (Suri *et al*. 2022). Wood samples were treated by plant oil to create a hydrophobic layer on the wood cells indicating less water uptake; the dimensional stability of oil treated wood was improved significantly (Dubey *et al.* 2012; Tomak *et al.* 2011; Humar and Lesar 2013; Ruwoldt and Toven 2022).

As an environmentally friendly wood modification method, oil thermal treatment has been considered by far one of the most efficient methods to modify wood properties. Therefore, this paper focused on using palm oil as heat transfer medium to improve the dimensional stability of GLGU9 wood.

EXPERIMENTAL

Materials

Fifteen *E. grandis* × *E. urophylla* 'GLGU9' trees were selected from a planted forest in Hainan Province, China, which was planted in 2009. The wood samples used in this study with dimensions of 20 mm (radial) × 20 mm (tangential) × 300 mm (longitudinal) were cut from those trees. The average moisture content was 10% before palm oil thermal treatment at 150, 170, 190, 200, and 210 ℃ for 1.5, 3, 4.5 and 6 h in an oil bath. All the treated samples were submersed in the palm oil to isolate oxygen. The untreated specimens from the same trees were used as control samples.

Equations

Firstly, the volumetric shrinkage rate and volumetric swelling rate of untreated and palm oil thermal treated wood sample were calculated according to Eqs. 1 through 4 as follows, which are quoted from the Chinese standard. Then the anti-shrink efficiency (ASE_1) and anti-swelling efficiency (ASE_2) were determined based on variations of the volume of samples before and after palm oil thermal treatment. The ASE_1 was divided into $ASE_{1\text{-}1}$ and $ASE_{1\text{-}2}$ due to the difference of volume situation. Identically, the ASE_2 was also divided into $ASE_{2\text{-}1}$ and $ASE_{2\text{-}2}$. The different *ASE*s were calculated according to Eqs. 5 through 8 as follows,

$$\alpha_{V\,w} = \frac{V_{W} - V_0}{V_0} \times 100\% \tag{1}$$

$$\alpha_{V\,max} = \frac{V_{max} - V_0}{V_0} \times 100\% \tag{2}$$

$$\beta_{V\,w} = \frac{V_{max} - V_w}{V_{max}} \times 100\% \tag{3}$$

$$\beta_{V\,max} = \frac{V_{max} - V_0}{V_{max}} \times 100\% \tag{4}$$

where $\alpha_{V\,w}$ is volumetric swelling rate of wood sample from absolute dry to air dry, V_w is air dry volume of wood sample, and V_0 is absolute dry volume of wood sample. The parameter $\alpha_{V\,max}$ is the volumetric swelling rate of wood sample from absolute dry to saturation, and V_{max} is the saturated volume of wood sample. The quantity $\beta_{V\ w}$ is the volumetric shrinkage rate of wood sample from saturation to air dry condition, and $\beta_{V\ max}$ is the volumetric shrinkage rate of wood sample from saturation to absolute dry condition.

$$ASE_{1-1} = \frac{\beta_{Vw0} - \beta_{V\ wt}}{\beta_{V\ w0}} \times 100\% \tag{5}$$

$$ASE_{1-2} = \frac{\beta_{V\ max0} - \beta_{V\ maxt}}{\beta_{V\ max0}} \times 100\% \tag{6}$$

$$ASE_{2-1} = \frac{\alpha_{Vw0} - \alpha_{V\ wt}}{\alpha_{V\ w0}} \times 100\% \tag{7}$$

$$ASE_{2-2} = \frac{\alpha_{V\ max0} - \alpha_{V\ maxt}}{\alpha_{V\ max0}} \times 100\% \tag{8}$$

where ASE_{1-1} is the increase of anti-shrink efficiency on air-dry volume (mm^3), $\beta_{V\ w0}$ is the volumetric shrinkage of untreated wood sample, and $\beta_{V\ wt}$ is the volumetric shrinkage of palm oil thermal treated wood sample. ASE_{1-2} is the increase of anti-shrink efficiency on absolute dry volume (mm^3), $\beta_{V\ max0}$ is the volumetric shrinkage of untreated wood sample, $\beta_{V\ maxt}$ is the volumetric shrinkage of palm oil thermal treated wood sample. ASE_{2-1} is the increase of anti-swelling efficiency on air-dry volume (mm^3), $\alpha_{V\ w0}$ is volumetric swelling of untreated wood sample, and $\alpha_{V\ wt}$ is volumetric swelling of palm oil thermal treated wood sample. ASE_{2-2} is increase of anti-swelling efficiency on absolute dry volume (mm^3), $\alpha_{V\ max0}$ is volumetric swelling of untreated wood sample, and $\alpha_{V\ maxt}$ is volumetric swelling of palm oil thermal treated wood sample.

Test Standards

The Random Complete Block Design (RCBD) method was used to arrange each experimental unit in this study to minimize the variations from different trees. The anti-shrink efficiency (ASE_1) and anti-swelling efficiency (ASE_2) of control and treated samples with a dimension of 20 mm (radial) × 20 mm (tangential) × 20 mm (longitudinal) were tested according to Chinese standards GB/T 1932 (2009) and GB/T 1932.4 (2009), respectively. Fifteen replications were tested for each treatment.

The analysis of variance (ANOVA) was performed according to Microsoft® Excel® 2016 MSO software (Version 2210, Redmond, WA, USA). The mean values were accepted to be considerably important when $p \leq 0.01$.

RESULTS AND DISCUSSION

The dimensional stability of palm oil thermal treated GLGU9 wood was improved remarkably compared with the control samples. The anti-shrink and anti-swelling efficiencies are shown in Figs. 1 and 2, respectively. Repeatable two-factor ANOVA indicated that not only temperature had a significant effect on dimensional stability of thermal treated wood at the level of 0.01, but also treatment duration, and the interaction between temperature and treatment duration had also a significant effect on the dimensional stability of treated wood at the same condition.

Anti-shrink Efficiency

The anti-shrink efficiency on air-dry volume (ASE_{1-1}) and absolute-dry volume (ASE_{1-2}) of heat-treated GLGU9 wood in palm oil were increased with an elevation of thermal treatment temperature from 150 to 210 °C and a prolongation of treatment duration from 1.5 to 6 h, as shown in Fig. 1. It was observed that $ASE_{1\text{-}1}$ and $ASE_{1\text{-}2}$ increased slightly while temperature was under 190 °C. Once the temperature exceeded 190 °C, the increasing tendency of ASE_1 moved quickly. This phenomenon seems to be similar to the results by other researchers. It was reported that depolymerization of the hemicellulose increased when temperature was raised to 185 °C. The hemicellulose content of Scots pine decreased from original 39.3% to 33% at 165 °C and to 19.7% at 185 °C during the process of hydro-thermolysis treatment (Boonstra and Tjeerdsma 2006). In the present study, the holocellulose content was decreased from 68.1% in control sample to 49.62% in palm oil thermal-treated GLGU9 wood treated at 190 °C for 4.5 h.

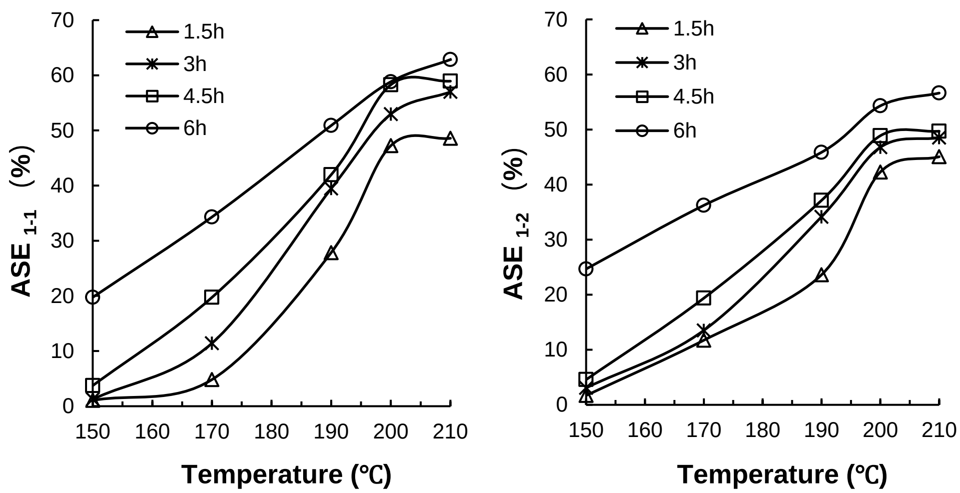

Fig. 1. Increased rate of ASE_1 of palm oil thermal-treated GLGU9 wood
($ASE_{1\text{-}1}$: increase rate of anti-shrink efficiency on air-dry volume (mm^3); $ASE_{1\text{-}2}$: increase rate of anti-shrink efficiency on absolute dry volume (mm^3))

Anti-swelling Efficiency

The anti-swelling efficiencies for air-dry volume ($ASE_{2\text{-}1}$) and water-absorbed volume ($ASE_{2\text{-}2}$) of thermal-treated GLGU9 wood in palm oil were increased with an elevation of treatment temperature and a prolongation of treatment, as shown in Fig. 2. The maximum increase observed was 56.24% for $ASE_{2\text{-}1}$ and 61.28% for $ASE_{2\text{-}2}$, both at 210 °C for 6 h. The ANOVA indicated that not only treatment temperature resulted in a significant difference ($\alpha = 0.01$) with respect to ASE_2 but also treatment duration and interaction of above two crucial factors made a similar significant difference to ASE_2 too, respectively. This trend is consistent with other findings. The linseed oil heat-treated *Pinus radiata* specimens achieved water repellent efficiencies of between 28 and 46%, indicating less water uptake and volumetric swelling was decreased by 29-31% at 180 °C for 3 h (Dubey *et al.* 2012). The water absorption of the maple wood treated by impregnated with linseed oil was decreased by 72% at 240 °C as compared to the control sample (Kaya 2023).

The quantity of accessible hydroxyl groups has a significant effect on dimensional stability of wood. The equilibrium moisture content (*EMC*) in the thermally modified wood would be reduced compared with control sample, which could be attributed to diminishing amount of accessible hydroxyl groups due to depolymerization of hemicellulose, thus increasing the proportion of relatively inaccessible crystalline cellulose and crosslinking of the lignin network that might inhibit the accessibility of free hydroxyl groups to water

(Tjeerdsma *et al.* 1998b; Lee *et al.* 2017). Correspondingly, the cellulose crystallinity of oil treated wood increased due to the decrease of huge amounts of affinity hydroxyl groups (Bhuiyan *et al.* 2000; Bhuiyan and Hirai 2005; Boonstra and Tjeerdsma 2006), and further crosslinking caused by the polycondensation reactions in lignin (Esteves *et al.* 2008; Ahmed *et al.* 2017). Linseed oil and tung oil were used to treat Norway spruce wood, it was found that oil-treated wood exhibited better water exclusion efficacy than that of untreated wood, oil treatment can reduce water uptake capacity of wood by half (Humar and Lesar 2013). This conclusion is consistent with this study. The EMC in the palm oil treated wood sample was decreased to in the range 12.84% to 7.81% with an increase of thermal treatment temperature and duration of treatment, compared to 14.66% of EMC in control sample. The lignin content was increased from 34.4% in control samples to 38.9% in palm oil thermal treated GLGU9 wood treated at 210 °C for 6 h.

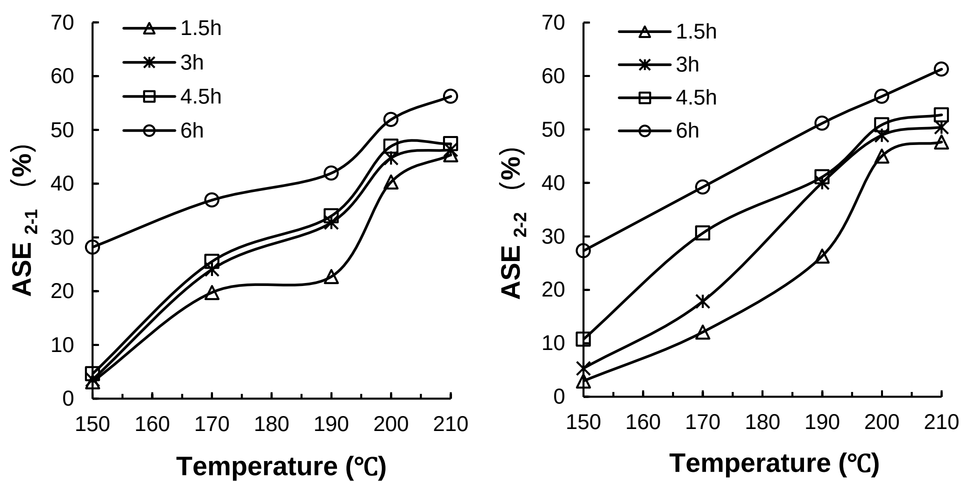

Fig. 2. Increased rate of ASE_2 of palm oil thermal treated GLGU9 wood.
(ASE_{2-1}: increase rate of anti-swelling efficiency on air-dry volume (mm^3) ; ASE_{2-2}: increase rate of anti-swelling efficiency on saturation volume (mm^3))

Concerning the oil uptake, Wang and Cooper (2005) reported that oil absorbed by heat-treated wood in palm and soya oil mainly affected the water absorption and was much less important to the swelling of wood, which is more affected by chemical changes as a result of high temperature. A similar result was observed by Dubey (2012). They used raw linseed oil to heat treat *Pinus radiata* wood and found that oil uptake by wood reduced the water absorption by an amount ranging from 10% to 85%. However, oil uptake in wood mainly affected the water hydrophobicity of the treated wood and was less important to the dimensional stability. It is considered that the linseed oil is a non-swelling chemical, and its molecule is too large to penetrate the cell wall of wood (Rosenqvist 2000; Olsson *et al.* 2001; Hill 2006). Therefore, the linseed oil was deposited in the cell lumens to block some water-accessible sites. As it is chemically bound with the cell wall so it would not reduce the swelling to any significant extent, although some dimensional stability (reduction in swelling) may be achieved after the impregnation due to oil in the cell wall and formation of protection layer on the wood (Dubey *et al.* 2012).

CONCLUSIONS

1. Treatment temperature and duration are crucial factors to improve the dimensional stability of wood in the process of palm oil thermal treatment of the examined *Eucalyptus* cross wood.
2. There was a significant difference at the level of 0.01 between the ASE and the treatment temperature, treatment duration, and interaction of above two factors, respectively.
3. Additionally, temperature was found to play a more important role than duration.

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