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Wood Properties Characterisation of Thermo-Hydro Mechanical Treated Plantation and Native Tasmanian Timber Species

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Abstract: Thermo-hydro mechanical (THM) treatments and thermo-treatments are used to improve the properties of wood species and enhance their uses without the application of chemicals. This work investigates and compares the effects of THM treatments on three timber species from Tasmania, Australia; plantation fibre-grown shining gum (*Eucalyptus nitens* H. Deane and Maiden), plantation saw-log radiata pine (*Pinus radiata* D. Don) and native-grown saw-log timber of the common name Tasmanian oak (which can be any of *E. regnans* F. Muell, *E. obliqua* L'Hér and *E. delegatensis* L'Hér). Thin lamellae were compressed by means of THM treatment from 8 mm to a target final thickness of 5 mm to investigate the suitability for using THM-treated lamellas in engineered wood products. The springback, mass loss, set-recovery after soaking, dimensional changes, mechanical properties, and Brinell hardness were used to evaluate the effects of the treatment on the properties of the species. The results show a marked increase in density for all three species, with the largest increase presented by *E. nitens* (+53%) and the smallest by Tasmanian oak (+41%). *E. nitens* displayed improvements both in stiffness and strength, while stiffness decreased in *P. radiata* samples and strength in Tasmanian oak samples. *E. nitens* also displayed the largest improvement in hardness (+94%) with respect to untreated samples. *P. radiata* presented the largest springback whilst having the least mass loss. *E. nitens* and Tasmanian oak showed similar dimensional changes, whilst *P. radiata* timber had the largest thickness swelling and set-recovery due to the high water absorption (99%). This study reported the effects of THM treatments in less-known and commercially important timber species, demonstrating that the wood properties of a fibre-grown timber can be improved through the treatments, potentially increasing the utilisation of *E. nitens* for structural and higher quality timber applications.

Keywords: *Eucalyptus*; plantation; wood modification; thermo-hydro mechanical treatments; structural properties

1. Introduction

Increasing demand for wood and wood-based products for construction is stimulating the use of fast-grown species to supplement and replace the more classical use of native forest species [1]. The area of planted forests has considerably increased in recent years, with plantations covering approximately 291 million ha in 2015 [2], allowing increased availability of fast-grown timbers to supply market demand. The species most commonly found in plantations are pines and eucalypts, covering 42% and 26% of the global planted areas, respectively [3]. In Tasmania, the most commonly planted species are shining gum (*Eucalyptus nitens* H. Deane and Maiden), native to Australia, and radiata pine (*Pinus radiata* D. Don), native to California, but extensively planted in temperate areas of Australia. Planted eucalypt species, which are grown for short rotations mainly to produce fibre for the pulp and paper sector, present lower density and different wood properties than their native counterparts—characteristics that might hinder their full deployment as structural products or engineered wood products (EWP) [4]. Concerns regarding important mechanical properties such as strength, hardness, dimensional stability, and durability of timbers have encouraged the adoption of wood modification treatments and processes [5–8]. Ecologically sustainable wood treatment options, avoiding the use of chemicals to preserve the timber, can benefit from the combined effects of temperature, moisture, and pressure to compress the timber and improve its properties and stability [9]. These thermo-hydro mechanical (THM) treatments evolved from the combination of heat treatments and densification of wood through compression [10]. Densification treatments have been a focus for decades as wood density is considered one of the most important properties for structural timber and EWP. High-density species are sought for construction purposes and treatments that contribute to its enhancement are favored.

There are several advantages of the use of THM treatments as wood treatment processes. For example, resistance to decay is improved by application of high temperatures [11], which has been largely employed for thermally treated wood [12,13]. The treated wood presents enhanced hygroscopicity due to the degradation of hemicellulose; a process that reduces the water absorption of wood [14] where effects are species-dependent according to the chemical composition of the timber [15]. However, mechanical properties tend to be reduced under heat treatments alone [16]. The addition of wood compression is an excellent combination to preserve and enhance important structural properties; the use of steaming and heating reduces the set-recovery of the modified timber, a phenomenon that occurs when timber is re-exposed to moisture and tends to recover the deformation created during the compression treatment. Hence, through reduction of the shape recovery of the treated wood, the effects of densification are stabilised [17–19]. The superior mechanical properties and increased durability are desirable for applications such as engineered wood floorings [20] and have generated interest for industrialisation of THM-treated material and improved processing options [21,22].

The modification of timber species originating from plantations has received considerable interest in Europe for species such as poplar [19,23,24], softwoods [25,26], and some eucalypt species [15,27,28]. Given the major representation of *E. nitens* and *P. radiata* in timber plantations, an understanding of the consequences of THM treatments on these species is warranted. To our knowledge, little to no research has examined the use of THM treatments to densify fibre-grown *E. nitens*, nor have the effects of the treatments been studied in comparison to other species such as *P. radiata*, or native-grown eucalypts. Given the positive effects of TMH treatments on other fast-grown species, there is much interest in understanding their suitability to modify eucalypt and pine plantation timbers. Such information would support the exploration of densification to enhance the properties of those fast-grown species, providing opportunities to increase their use in structural products.

The aim of this study was to investigate the effects of wood modification treatments (THM treatment and THM and preheating treatment) on timbers from the two plantation species (*E. nitens* and *P. radiata*), grown under different regimes, and on a mixture of native forest-grown eucalypt species commercially known as Tasmanian oak. We were interested in understanding the changes in density and mechanical properties due to the wood modification treatments on the fast-grown, short-rotation plantation hardwood, on the planted softwood, and on the slow-grown, long-rotation native hardwood, and in comparing the effects of the treatments across the wood types. The objectives of the study were to (i) quantify the change in basic density and mechanical properties (stiffness, strength, and Brinell hardness) within each species due to the treatments, (ii) compare that change across the species, and (iii) examine and compare across species the characteristics of the wood species after the treatments in terms of compression degree, immediate springback, mass loss, set-recovery after soaking, dimensional changes, mechanical properties, and Brinell hardness. Finally, (iv) we were interested in exploring the correlation between the mechanical properties and final density for each wood species. The characterisation of the treated timbers will inform the feasibility of using fast-grown plantation species as a substrate for high-quality engineered wooden floorings of other EWP.

2. Materials and Methods

2.1. Material

The wood material used in the study were kiln-dried and planed lamellae of three Australian commercially important species: plantation fibre-grown shining gum (*Eucalyptus nitens* H. Deane and Maiden) managed for fibre production, plantation-grown radiata pine (*Pinus radiata* D. Don) managed for saw-log production, and native-grown hardwood timber of the commercial brand Tasmanian oak (which comprises wood from any of three closely related native eucalypt species: *E. regnans* F. Muell, *E. obliqua* L'Hér, and *E. delegatensis* L'Hér). We chose samples harvested and milled according to conventional practices that represent timber commercially grown for production of structural products. Forty samples per species were used, of which ten per species were used as control samples. The samples were a mixture between quartersawn and transitional/backsawn cuts, with growth-ring orientation ranging from perpendicular to parallel to the wide face of the lamella. The samples were kept in a conditioning chamber at relative humidity (RH) 65% and 20 °C before transportation to the laboratory.

2.2. Thermo-Hydro Mechanical (THM) Treatment and Preheating Treatment

The THM treatments and the tests were carried out at the InnoRenew CoE laboratories in Izola and Koper, Slovenia. Thirty 325 mm (L) × 45 mm (T) × 8 mm (R) samples per species were densified under THM treatment in an open-system process using a hydraulic, 30-ton capacity Langzauner "Perfect" LZT-UK-30-L model hot press (Langzauner Gesellschaft m.b.H., Lambrechten, Austria) equipped with a water-cooling system. The press plates were preheated to 170 °C, samples were inserted between the two plates and enclosed for a cycle of 30 min, during which a mechanical pressure of 300 kN was applied in the press. At reaching target thickness, the specimen was held at 170 °C for three min and the temperature was increased to 200 °C. When the temperature of 200 °C was reached, the plates were cooled under compression to 60 °C before opening the press. For the Tasmanian oak samples, a preheating treatment at 100 °C for 30 min was applied before the THM treatment to reduce the moisture content of samples to avoid bursting the samples during the THM treatment.

2.3. Physical Tests

The thickness of the samples was measured before treatment and again immediately after. Samples were compressed to the target thickness of 5 mm, and the compression ratio (CR), also known as target densification ratio, was calculated expressing the percentage of the target decrease in thickness compared to the initial thickness of the samples (Equation (1)):

$$CR\ (\%) = \frac{T_o - T_t}{T_o} \times 100 \tag{1}$$

where T_o is the original thickness (mm) and T_t is the target compressed thickness (mm).

The actual densification was evaluated with the final achieved thickness T_c, which calculates the actual densification ratio (DR) on the entire densified lamellae, expressing the percentage decrease in thickness compared to the initial thickness of the samples (Equation (2)):

$$DR\ (\%) = \frac{T_o - T_c}{T_o} \times 100 \tag{2}$$

The difference between compression ratio and actual densification ratio is due to the springback of the samples at the opening of the press, which measures the immediate recovery of thickness in the samples due to the phenomenon of shape memory effect of wood (Equation (3)):

$$Springback\ (\%) = \frac{T_c - T_t}{T_o - T_t} \times 100 \tag{3}$$

The compression degree (CD) (Equation (4)) to which the samples were densified measures the deformation caused by the treatment and serves as a measure of the increase in the density of the samples:

$$CD\ (\%) = \frac{T_o - T_c}{T_c} \times 100 \tag{4}$$

The mass loss (ML) was calculated (Equation (5)) to account for the variation in sample mass due to the treatment:

$$ML\ (\%) = \frac{M_o - M_c}{M_o} \times 100 \tag{5}$$

where M_o is the sample dry mass (g) of the conditioned samples (entire lamellae) before the treatment, and M_c is the sample dry mass (g) measured after the treatment. The M_o of Tasmanian oak samples was determined before the preheating treatment, as the samples were placed immediately into the press after the preheating, to avoid intake of moisture from the environment.

Basic density and moisture content (MC) were measured following the oven-dry method according to AS 1080.1 and AS 1080.3 [29,30]. Measurements were made on a 25 × 45 mm strip (Section A, Figure 1) cut from the sample prior to the treatment and from a second 25 × 45 mm strip (Section B, Figure 1) cut from the sample after the treatment. From the same end, another 25 × 45 mm strip (Section D, Figure 1) was cut to perform the set-recovery and water absorption tests. The compression set-recovery (SR) determines the irreversible recovery in thickness of the THM-treated samples after soaking in water. Samples were first dried to 0% moisture content, then soaked for 24 h in water at 20 °C and then oven-dried at 103 °C for 24 h following the procedure described in Laine et al. (2013) [31]. Dimensions and weights of the samples were recorded after soaking and at oven-dried conditions. SR was calculated using Equation (6):

$$SR\ (\%) = \frac{T_r - T_c}{T_0 - T_c} \times 100 \tag{6}$$

where T_r is the oven-dried thickness (mm) after soaking. Water absorption (WA) was calculated using Equation (7):

$$WA\ (\%) = \frac{w_1 - w_e}{w_e} \times 100 \tag{7}$$

where w_1 is the weight (g) after soaking for 24 h and w_e is the oven-dried weight (g) before immersion in water.

The change in dimensions was accounted through the measurement of thickness swelling (TS), according to Equation (8):

$$TS\ (\%) = \frac{T_s - T_c}{T_c} \times 100 \tag{8}$$

where T_s is the thickness (mm) of the saturated sample after soaking.

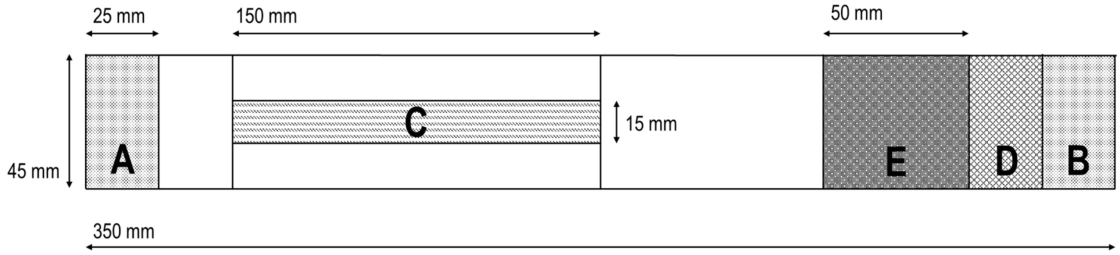

Figure 1. Sample preparation and tests from cut lamellae: (**A**) pre-thermo-hydro mechanical (THM) treatment density and moisture content (MC) tests, (**B**) post-THM treatment density and MC test, (**C**) modulus of elasticity (MoE) and modulus of rupture (MoR) tests, (**D**) set-recovery after soaking tests, and (**E**) Brinell hardness test.

2.4. Mechanical Tests

Stiffness of the control and THM-treated samples was measured through Young's modulus in compression (MoE—modulus of elasticity) and strength through modulus of rupture (MoR), which were tested under a three-point bending arrangement. From the conditioned samples, a 150 × 15 mm strip was cut (Section C, Figure 1) and loaded in the Zwick (10 kN) universal testing machine (ZwickRoell, Germany) with the application of a load at a displacement rate of 7 mm/min. Tests were performed according to DIN 52–192 1979 [32] on a loading span of 85 mm. MoE and MoR were obtained through the recorded strain–stress curves provided by the precalibrated Zwick software. Two *E. nitens* and three Tasmanian oak samples were damaged during the treatment; hence the final sample size for the mechanical tests of stiffness and strength on these timbers was lower than initially planned. MoE and MoR were tested on control samples under a similar arrangement, on a loading span of 128 mm.

Brinell hardness (HB) was measured on 45 × 50 mm control and treated samples (Section E, Figure 1) adapting EN 1534:2000 [33] and following the methodology developed by Rautkari et al. (2011) [24]. The test was performed keeping the indentation depth constant, given the low thickness of the samples, and the applied force was recorded. The test was performed with the Zwick universal testing machine using a steel sphere 11.28 mm in diameter and HB (N/mm^2) was calculated using Equation (9):

$$HB = \frac{F}{\pi \times D \times h} \tag{9}$$

where F is the force (N) exerted into the samples, D is the radius (mm) of the indenter, and h is the depth (mm) of the indentation.

2.5. Statistical Analysis

Statistical analyses were performed with the RStudio software (RStudio Team 2016). The normality and homogeneity of variance in the data were verified with Shapiro–Wilk and Levene's tests, respectively. Datasets were inspected for outliers using the Tukey's fences method [34], as well as visual inspection of plots and outliers that could significantly bias the testing. Four samples of Tasmanian oak that presented exceptionally high basic density values prior to the treatment were removed from the dataset.

Two-way mixed measures analysis of variance (ANOVA) was used to compare the means of the basic density of the three species, using species and treatment as factor variables. Two-way analysis of variance (ANOVA) was used to compare the means of the mechanical properties (MoE, MoR, and HB) of the three species on control and treated samples. To identify differences between species, multiple pairwise comparisons were used as *post hoc* tests.

The differences across species in the properties after the treatment (CD, springback, ML, TS, WA, set-recovery) were evaluated with the Kruskal–Wallis test and the paired-samples Wilcoxon test, as the assumption of normality or homoscedasticity were not met for any of these variables.

Linear regression was used to model the relationships between structural properties (MoE and MoR) with the densities of THM-treated samples. All test results were compared with a significance threshold set at 0.01.

3. Results and Discussion

3.1. Densification of the Material

Initial densities were 0.52 g/cm^3 for *E. nitens*, 0.46 g/cm^3 for *P. radiata*, and 0.61 g/cm^3 for Tasmanian oak. The initial density before the treatment was significantly different between the three species ($F(2,82) = 22.6$, $p < 0.01$). While the THM treatment significantly increased the density in all three species (Table 1, Figure 2) ($F(1,82) = 1143.1$, $p < 0.01$), species response was different, showing a significant effect of treatment on the species ($F(1,82) = 11.3$, $p < 0.01$). *E. nitens* samples had the larger percentage increase in density (53%), compared to both the *P. radiata* and Tasmanian oak samples (respectively, 43% and 42%), and its final density of 0.80 g/cm^3 was not significantly different to that of the Tasmanian oak samples ($p = 0.57$), which reached 0.89 g/cm^3 on average. The density of *P. radiata* samples after the treatment was 0.66 g/cm^3 on average, significantly lower than both *E. nitens* and Tasmanian oak ($p < 0.01$).

Table 1. Actual and target compressed thickness (mm), densification ratio (%), average values of oven-dry initial density, and final density after the THM (Thermo-Hydro Mechanical Treatment) (g/cm^3). Standard deviation presented in parentheses ($n = 30$).

Species	Initial Thickness (mm)		Compressed Thickness (mm)		Densification Ratio (%)		Density (g/cm^3)	
	Target	Actual	Target	Actual	Target	Actual	Before Treatment	After Treatment
E. nitens	8.0	8.17 (0.14)	5.0	4.86 (0.03)	38.8	40.5 (1.19)	0.52 (0.07) a	0.80 (0.09) d *
P. radiata	8.0	7.99 (0.25)	5.0	5.29 (0.44)	37.3	33.8 (5.84)	0.46 (0.07) b	0.66 (0.09) e *
Tasmanian oak	8.0	8.44 (0.65)	5.0	5.37 (0.53)	37.3	36.2 (6.81)	0.63 (0.12) c	0.89 (0.19) d *

a, b, c letters denote significant differences within the group of species before the treatment; d and e denote significant differences within the group of species after the treatment. The asterisk indicates significant differences between the density prior to the treatment and the density after, per each species considered. All significant levels had $p < 0.01$.

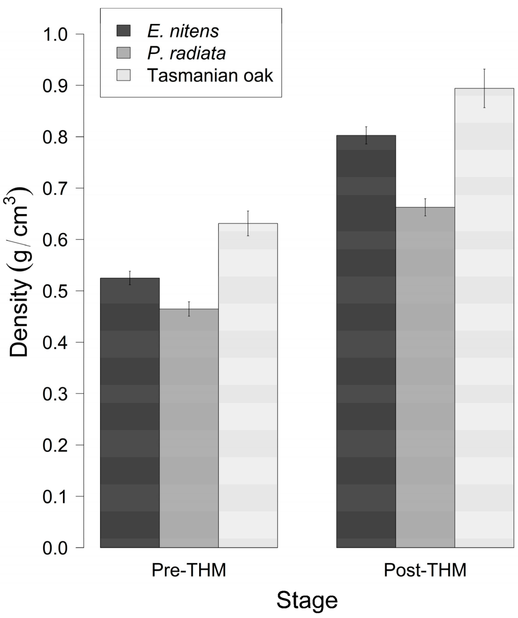

Figure 2. Density before (Pre-THM, left) and after the treatment (Post-THM, right) of the three species (black, *E. nitens*; dark grey, *P. radiata*; and light grey Tasmanian oak). The line on the bar represents the standard deviation.

After the THM treatment, the basic density of *E. nitens* was close to that of the native Tasmanian oak samples and surpassed the density of the other plantation species, *P. radiata*. These results suggest selection of timber for densification might be better based on the species, rather than on the initial density, which is usually low for fast-grown species. It is worth noting that the variance in density after the treatment of Tasmanian oak samples was higher than both that of *E. nitens* and *P. radiata*, suggesting that the response of the slow-growing species to densification was less consistent than that for the two plantation species.

The target thickness was 5 mm for all the samples, hence the target densification ratios (CR) were 38.8% for *E. nitens* and 37.3% both for *P. radiata* and Tasmanian oak. The thickness was measured on the conditioned samples prior to the treatment and immediately after. The compressed thicknesses for the three species were on average 4.86, 5.29, and 5.37 mm for *E. nitens*, *P. radiata*, and Tasmanian oak, respectively, indicating final densification ratios of 40.5%, 33.8%, and 36.2%, as presented in Table 1.

3.2. Mechanical Properties

The mechanical properties of the samples are summarised in Table 2. MoE of the control samples were 13.41 GPa for *E. nitens*, 11.67 GPa for *P. radiata* samples, and 17.08 GPa for Tasmanian oak samples, significantly different among the wood of Tasmanian oak and the other two species ($p < 0.01$) but not significantly different among the wood of *E. nitens* and *P. radiata* samples ($p = 0.018$). After the treatments, MoE of the lamellae was significantly different among all three wood types ($F(2,82) = 25.2$, $p < 0.01$). The THM treatment significantly increased the MoE only of *E. nitens* samples ($p < 0.01$), which reached an average of 17 GPa after the treatment; conversely the MoE of *P. radiata* THM-treated samples was on average lower than that of control samples, indicating that this property was not improved due to the process. Tasmanian oak samples showed the largest improvement, from 17 GPa to almost 23 GPa on average, although the two values were not significantly different ($p = 0.73$).

Table 2. Modulus of elasticity (MoE, GPa), modulus of rupture (MoR, MPa) and Brinell hardness (HB, N/mm^2) of the three species compared with values of the control samples. Standard deviation presented in parentheses (THM-treated samples: *E. nitens* $n = 28$, *P. radiata* $n = 30$, Tasmanian oak $n = 27$).

Species	MoE (GPa)		MoR (MPa)		HB (N/mm^2)	
	Before Treatment	After Treatment	Before Treatment	After Treatment	Before Treatment	After Treatment
E. nitens	13.41 (1.46)	17.03 (3.56) *	135.36 (25.86)	151.9 (45.3)	11.98 (3.03)	23.22 (9.51) *
P. radiata	11.67 (1.15)	11.24 (3.04)	115.86 (13.56)	139.51 (27.1) *	12.27 (3.54)	18.39 (6.56) *
Tasmanian oak	17.08 (1.05)	22.87 (9.62)	165.94 (5.17)	144.07 (97.22)	12.92 (1.75)	21.18 (7.75) *

Significance levels shown for before and after treatments comparison: * $p < 0.01$.

MoR of control samples was also significantly different among the wood of Tasmanian oak and the other two species ($p < 0.01$) but not significantly different among the wood of *E. nitens* and *P. radiata* samples ($p = 0.068$). After the treatments, MoR was not significantly different among the three wood types ($p = 1$). *E. nitens* showed an increase in MoR, albeit not significant ($p = 0.29$), while for *P. radiata* there was a significant increase of this property ($p < 0.05$), reaching almost 140 MPa on average in THM-treated samples. MoR of THM-treated Tasmanian oak samples was lower than that of control samples, showing a reduction in strength after the treatment, results which might have been conducted by the preheating treatment on these wood samples. Similar results were obtained on *P. elliotti* and *E. grandis* by Pertuzzati et al. [15], with only slight increases in MoE for the pine and MoR for the eucalypt species. In other thermal modification studies, eucalypt species have shown either slight increases in their mechanical properties, as for *E. nitens* [35], or decreases, as for *E. grandis* and *E. saligna* [36] and *E. globulus* [37]. During wood treatment, the thermal degradation of the chemical components of wood cells can offset the improvement of mechanical properties [38,39], rendering the wood structure more fragile. During the mechanical test, failure of THM-treated samples occurred suddenly, indicating an increased brittleness of the wood, while control samples under loading deformed constantly before breakage occurred.

The modified Brinell hardness method was necessary to evaluate thin lamellae, as it has been noted before how the hardness values depend upon the force applied during the test and on the standard adopted [24]. The same method was used on control and THM-treated samples. The Brinell hardness was not significantly different among wood types either before or after the wood treatments. A significant increase in hardness was found for all three species ($p < 0.01$), with the largest increase displayed by *E. nitens*, reaching 23.22 N/mm^2 on average, an increase of almost 94% compared to control samples. The lowest increase was displayed on *P. radiata* THM-treated samples, reaching 18.39 N/mm^2 on average, while Tasmanian oak reached 21.18 N/mm^2, albeit showing the largest hardness values on control samples. Similar values of Brinell hardness were found in Wentzel et al. [28] for *E. nitens* samples grown in Spain, although the method employed for the test was different than the one used in the present study. Hardness is known to improve through densification, as it is influenced especially by the density of the surface layer [40,41]. In this work, the hardness of all three species was positively correlated with their basic densities after the THM treatment, although being only significant for Tasmanian oak samples ($p < 0.01$, $R^2 = 0.56$, data not shown).

Bending properties of the three species studied and their densities after the THM treatment are shown in Figure 3a (MoE) and Figure 3b (MoR). The correlations between MoE, MoR, and post-THM density were positive and significant for all three species (Table 3). Regression models developed to predict final MoE and MoR considering initial densities are also shown. The increase in density also led to an increase in the MoE and MoR of the samples, and we hypothesise that the differences in the increase are due to the anatomical structure of the wood and the orientation of the wood samples.

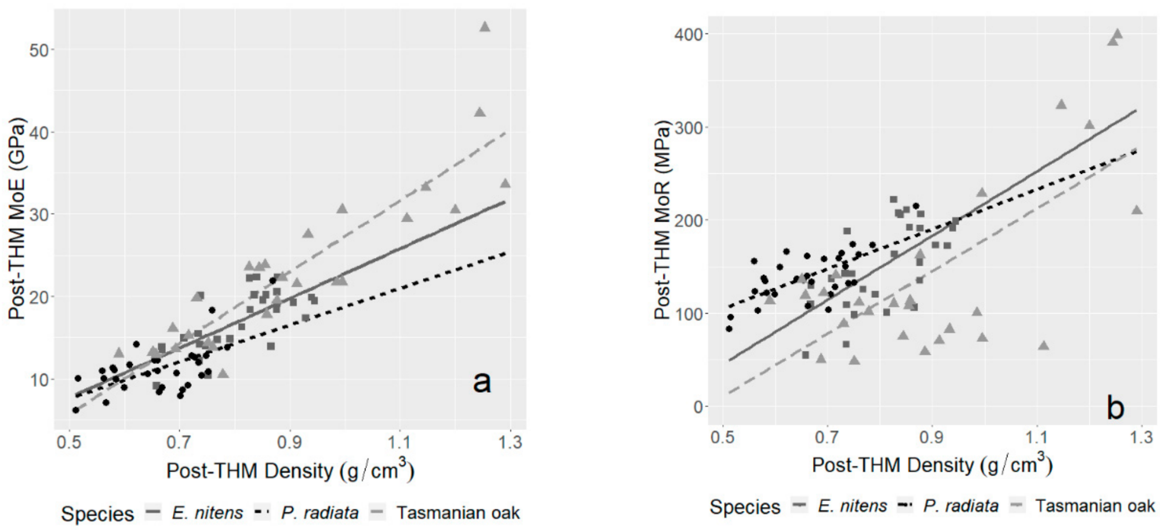

Figure 3. Correlation graphs between MoE (**a**), MoR (**b**), and density of the samples after THM treatment (*E. nitens* in grey solid line and square dots, *P. radiata* in black dotted line and black dots, and Tasmanian oak in light grey dashed line and triangular dots).

Table 3. Prediction of structural properties for the densified material (*E. nitens*, $n = 28$; *P. radiata*, $n = 30$; Tasmanian oak, $n = 27$).

Dependent Variable	Species	Linear Regression Model	Coefficient of Determination (R^2)	p Value
MoE (GPa)	*E. nitens*	$MoE = -7.35 + 30.13 \times \partial_{Post\ THM}$	0.47	***
	P. radiata	$MoE = -3.5 + 22.26 \times \partial_{Post\ THM}$	0.36	***
	Tasmanian oak	$MoE = -15.88 + 43.19 \times \partial_{Post\ THM}$	0.78	***
MoR (MPa)	*E. nitens*	$MoR = -127.29 + 345.17 \times \partial_{Post\ THM}$	0.37	***
	P. radiata	$MoR = -2.17 + 213.84 \times \partial_{Post\ THM}$	0.42	***
	Tasmanian oak	$MoR = -157.7 + 336.71 \times \partial_{Post\ THM}$	0.44	***

*** denote $p < 0.01$.

3.3. Compression Degree, Springback, and Mass Loss

In this study, higher compression degrees (CD) were achieved with the two eucalypt species, as also illustrated in Gong and Lamason [42] and Pertuzzati et al. [15]. The largest compression degree was for *E. nitens* samples, which compressed to 68.2% of their thickness, followed by Tasmanian oak and *P. radiata* samples, with 58.3% and 52.1%, respectively (Table 4). There was significant variation in compression degree among species ($\chi 2(2) = 42.31$, $p < 0.01$), while CD was statistically significant between *E. nitens* and both *P. radiata* and Tasmanian oak ($p < 0.01$), but no significant difference was detected between CD of *P. radiata* and Tasmanian oak ($p = 0.03$). These results were influenced by the final thickness of the samples, which for *E. nitens* was less than the target thickness controlled by the mechanical stops; an outcome most likely influenced by the shrinkage of the samples under the high temperatures applied during the treatments. This additional shrinkage would have to be investigated further with a different target densification ratio.

Table 4. Average springback (%), compression degree (%), and mass loss (%) for the three species tested ($n = 30$). Standard deviation presented in parentheses ($n = 30$).

Species	Compression Degree (%)	Average Springback (%)	Mass Loss (%)
E. nitens	68.2 a	−4.52 c	2.42 (1.18) e
P. radiata	52.1 b	9.57 d	3.60 (0.31) f
Tasmanian oak	58.3 b	2.49 cd	4.13 (1.72) gf

* Letters denote significant differences in the measurements within the group of species after the treatment. All significant levels had $p < 0.01$.

The differences between the target and the actual densification ratio are due to the immediate springback occurring at the opening of the press, due to the stresses built up during the THM treatment. The shrinkage of the *E. nitens* samples led to a negative value on the calculated springback (−4.52%), while different values were found on *P. radiata* (9.57%) and Tasmanian oak samples (2.49%). The results for *P. radiata* and Tasmanian oak samples are in agreement with those found in previous studies [43,44] where higher CRs lead to a greater springback. Earlywood and latewood compress differently, and different amounts of latewood present in the native slow-grown Tasmanian oak samples in comparison to the plantation-grown *P. radiata* samples might have impacted their rate of compression and the structural changes of the timber. This would lead to further considerations in the treatment of plantation timbers.

The springback was significantly different among the three species ($\chi 2(2) = 36.87$, $p < 0.01$), while being statistically significant only for *E. nitens* and *P. radiata* ($p < 0.01$), finding most likely due to the negative values of the springback in the *E. nitens* compared to the large springback in the *P. radiata* samples.

The *P. radiata* samples had the lowest compression degree and densification ratio but achieved the highest springback (Table 4). From the investigation of the densification ratio it appeared that the samples of this species had densified at different rates, an effect that might be due to the differences in initial densities of the samples. Furthermore, differences in springback of the samples are related to the amount of latewood present, as *P. radiata* is known to have significant differences in early and latewood density [45]. Detailed analysis was not made; however, it was noticed that samples with higher percentages of latewood undergo higher springback.

The mass loss on the entire lamellae was significantly different among the samples ($\chi2(2) = 25.81$, $p < 0.01$). *E. nitens* samples had significantly lower mass loss than the other two species ($p < 0.01$), with an average of 2.42%, in comparison to *P. radiata* (3.6%) and Tasmanian oak (4.13%) (Table 4). The larger mass loss on Tasmanian oak samples is in line with previous research, in which it was found that eucalypt species (*Eucalyptus grandis* and *Eucalyptus globulus*) had larger mass losses in comparison to pine species (*Pinus elliottii* and *Pinus pinaster*) [15,46]. Moreover, the higher mass loss of the Tasmanian oak samples might be related to the preheating treatment applied on the wood, which had affected the moisture content of the samples. This should be considered for the development of future treatment protocols for this species.

The variable level of mass loss in the samples might be explained by the differences in chemical composition of the wood, in which the substrates degrade in different ways; hemicellulose tends to be more subjected to degradation rather than lignin [47]. During densification, water vaporisation contributes to the mass loss, which is followed usually at higher temperatures by the degradation of extractives, hemicellulose, and at the latest stages, lignin [48]. Eucalypt wood has less cellulose and lignin, and more hemicellulose than pine wood, which is also more susceptible to thermal degradation due to the higher presence of xylans [49]. Moreover, there are differences in the amount of extractives that may have impacted the response of the species to the densification treatment. Growth rate and extractive concentrations are negatively correlated [50] and previous investigations have shown how younger eucalypt trees presented higher extractive contents than older ones [51]—findings that support the hypothesis of a different amount of extractives in the planted *E. nitens* with respect to the slower-grown native Tasmanian oak samples.

3.4. Dimensional Changes

The absorption of water during the soaking treatment caused a dimensional change in the samples. The dimensional change and shape recovery after soaking and drying were significantly different among the three species studied ($\chi2(2) = 50.27$, $p < 0.01$) (Figure 4). While the two eucalypt species had similar amounts of swelling in thickness (almost 30% for *E. nitens* and 24% for Tasmanian oak samples), pine samples exhibited significantly larger thickness swelling ($p < 0.01$), reaching more than 50%. The final thickness after soaking and drying of pine samples was 22% higher than that of the eucalypts.

The major swelling of pine samples was due to their high rate of water absorption (99.2%), which was significantly different ($p < 0.01$) compared to the other two timbers (Table 5), relating to previous findings on the same species where similar rates of water absorption were found [25]. Samples of *E. nitens* and Tasmanian oak had moderate water absorption rates of 53.4% and 45.2%, respectively. In our study, the water absorption rate of *P. radiata* samples was extremely high for samples that achieved a densification ratio over 35%, suggesting that more compression (hence large densification) would induce a greater swelling ratio and water absorption volume for this species.

Set-recovery values were different among species ($\chi2(2) = 55$, $p < 0.01$), while being significant only for the pine ($p < 0.01$; Table 5). No samples achieved a full recovery, indicating that some of the structural changes induced by the THM treatment were permanent. The treatment combination of pressure, high temperature, and increased moisture is known to ameliorate the build-up of internal stresses that are the principal cause of the springback and set-recovery of the samples. This was confirmed in our study, also highlighting the differences in the recovery among eucalypt and pine species.

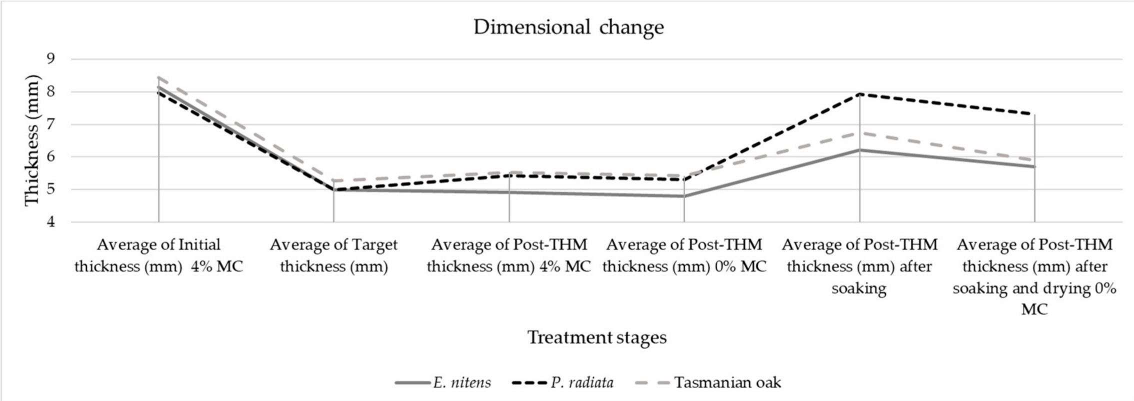

Figure 4. Dimensional change of the samples in each state, before the treatment, at the target thickness, after the densification (both at initial 4% MC and at oven-dried 0% MC), after soaking and at the final soaked and dried thickness (mm).

Table 5. Average values of thickness swelling (%), water absorption (%), and set-recovery (%) of the three species. Standard deviation presented in parentheses (n = 30).

Species	Thickness Swelling (%)	Water Absorption (%)	Set-Recovery (%)
E. nitens	30.0 (17.7) a	53.4 (10.9) a	27.5 (7.75) a
P. radiata	50.1 (6.55) b	99.2 (16.5) b	75.5 (9.74) b
Tasmanian oak	24.1 (14.8) a	45.2 (15.5) a	17.4 (5.83) a

a, and b letters denote significant differences in the measurements within the group of species after the treatment. All significant levels had $p < 0.01$.

4. Conclusions

In this study, samples of fibre-grown *E. nitens*, saw-log managed *P. radiata*, and native eucalypt species from Tasmania were treated under short-period thermo-hydro mechanical treatment and densified to a final thickness of 5 mm. The treatments successfully increased the basic density in all three species, with the major increment present on *E. nitens* samples (53%). The initial density of *E. nitens* prior to the THM treatment was significantly lower than that for Tasmanian oak. After the treatment there was no significant difference between the two species. Furthermore, the final density of the two eucalypt species was significantly higher than that of pine samples. This highlights the potential use of THM-treated fibre-grown *E. nitens* as a substitute for native eucalypt species (e.g., Tasmanian oak) in applications requiring high density materials, such as laminated floors. This is further enhanced by the higher availability and faster growth rate of *E. nitens* compared to native eucalypt species [1]. The relatively lower density of the THM-treated pine samples makes this alternative less suitable to applications requiring high density materials when compared to the other two eucalypt species.

The mechanical properties of stiffness and strength had different responses to the treatments, with an increase in stiffness only for the eucalypt timbers (*E. nitens* and Tasmanian oak samples) and a slight decrease for the *P. radiata* samples. There was no significant difference in the strength of THM-treated *E. nitens* and treated Tasmanian oak, which highlights the possibility of treating and deploying *E. nitens* timber for structural applications requiring stiff material. Additional testing will be required to further explore these opportunities. Strength was reduced after treatment only on Tasmanian oak samples. This will require further investigation as the current study highlights a possible impact due to the necessary preheating of this timber to avoid the bursting of the wood. The adapted Brinell hardness test revealed an improvement in hardness for all three wood types, with higher values for the eucalypt species compared to pine, and *E. nitens* obtaining the highest increment in hardness (+94%). For flooring applications, hardness is of large importance, and the high values obtained for the *E. nitens* samples support their suitability in such applications. Engineered wood products such as laminated floors require high-dimensional stability and elevated structural properties, and our study found that *E. nitens* THM-treated samples had higher performance than pine after the soaking and drying cycle, as well as presenting increases in all the tested mechanical properties, showing that the treatment was most effective for this species.

This study demonstrated the effectiveness of THM treatments in increasing the density of plantation-grown timbers (*E. nitens* and *P. radiata*) and native eucalypt timbers, as well as affecting their mechanical properties. Appreciable differences between the species were shown and the application of the treatment on fibre-grown *E. nitens* wood showed promising results in wood properties. This finding might direct further investigations in the suitability of densification of low-density fibre-grown *E. nitens* for the production of engineered structural products. Future investigation might focus on examining different degrees of densification and initial different preheating treatments, especially for the denser native timbers forming the Tasmanian oak samples. This initial study demonstrated the applicability of THM treatments on Australian timbers with satisfactory final results, which might be transferred for potential industrial processes and engineered timber applications.

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