Effect of a thermo-mechanical densification process on selected properties of densified wood of Tasmanian oak and Eucalyptus nitens

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Title: Effect of a thermo-mechanical densification process on selected properties of densified wood of Tasmanian oak and *Eucalyptus nitens*

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Abstract

Regrowth Tasmanian oak and *Eucalyptus nitens* are fast-growing Australian plantation hardwoods widely cultivated in Tasmania. Despite their favourable physical and aesthetic properties, their relatively low density limits use to wood-chip production and minor interior applications. This study aimed to enhance the material properties of these species through thermo-mechanical densification. The effects of compression ratio, pressing time, and pressing temperature were evaluated on color change, set recovery (wet and dry), pull-off strength, and delamination. *Eucalyptus nitens* showed excellent color stability under all conditions ($\Delta E^*ab < 5$), while Tasmanian oak exhibited significant darkening at 175 °C (ΔE^*ab 6.17 - 9.06). At 175 °C and 37 % compression, *E. nitens* responded more strongly, achieving a mean wet-use set recovery of 0.0 %, compared with 2.7 % for Tasmanian oak. Both species showed significantly improved dimensional stability at 37% compression ratio compared to 25%. The highest *F*-values observed for *Eucalyptus nitens* and Tasmanian oak were 25.36 and 17.91, respectively. Increased compression also improved pull-off strength in both species, but in Tasmanian oak, extractive migration at 175 °C likely reduced coating adhesion. The densification process had minimal impact on bondability overall. However, a higher pressing temperature significantly reduced delamination in Tasmanian oak (*P*-value 0.031), while showing negligible effect in *Eucalyptus nitens*. The results suggest that both *Eucalyptus nitens* and Tasmanian oak species were stable following the densification process and would make the densified wood serviceable under conditions in which the EMC does not exceed 17% if unprotected.

Statements and Declarations

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Benoit Belleville. The first draft of the manuscript was written by Benoit Belleville and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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1 Introduction

Density is one of the most important characteristics of wood as it is strongly correlated to its mechanical properties (Sandberg et al., 2013).Wood modification techniques have been used to valorise underutilised wood materials and increase their performance with respect to durability, mechanical characteristics, and new forms and functions desired by consumers and designers alike (Sandberg et al., 2017). Several densification processes have been presented and developed to improve the strength and surface properties of lower-value wood species (Pelit and Emiroglu, 2021).

During the densification process, wood is softened and compressed, resulting in densification, ideally without fracturing the cell walls. Compression typically takes place in a press between 120 to 180 °C by carefully controlling the pressing conditions (Rautkari et al., 2011). The moisture in cell walls induces a mechano-sorptive effect and further softens the wood, enabling mechanical compression of wood without cell wall fracture (Bao et al., 2017). Densification of wood is typically carried out in the radial direction (Kutnar et al., 2015). Densification in the tangential direction is typically avoided as tangential compression can lead to damage and buckling of the latewood annual rings resulting in a zig-zag pattern forming through the cross-section of the densified wood material (Sandberg et al., 2013; Kutnar et al., 2015; Sotayo et al., 2020). The densification profile in compressed solid wood depends on factors such as the initial wood moisture content, wood grain orientation, press temperature and time of compression (Rautkari et al., 2011). Other factors such as species, pre/post-treatment conditions, compression ratio, pressure applied, and pressing speed can also influence the process, quality, and mechanical properties of the densified material (Sotayo et al., 2020).

The degree of cell wall plasticization during compression is a key factor. If adequately plasticized, wood cells can be compressed without fractures as they deform instead of breaking when buckled. During the heating of the wood, the lignin, hemicellulose, and cellulose display different softening behaviour depending upon the temperature and moisture content (Salmén, 1990). For this reason, the compression step is mainly performed at temperatures exceeding the glass transition temperature (Tg) of wood constituents and reaching temperatures at which these constituents decompose (Navi and Sandberg, 2011). The heat treatment can also improve resistance to decay (Huang et al., 2012), decrease hygroscopicity (Metsä-Kortelainen et al., 2006; Kariz et al., 2017), and improve dimensional stability (Esteves et al., 2007; Kariz et al., 2017). If the deformations during the densification process are large, the result is the viscous buckling of cell walls without major fracture taking place and the strength and stiffness of the wood material are increased approximately in proportion to the increase in density (Kutnar et al., 2008).

The morphology of densified wood depends largely on the percentage of densification (Kutnar et al., 2009). Higher temperature enables relaxation of the inner stress and even minor thermal degradation of the cell wall components takes place, leading to a more stable state after compression (Laine et al., 2014). By contrast, with lower temperatures, there is very little stress relaxation, and thus, the deformation is mainly elastic. The elastic energy is stored in the cell walls, and as the load is removed, the stress is released, causing immediate spring back deformation (Navi and Heger, 2004). An increase in holding time or temperature results in an increase in peak distance, whilst an increase in initial moisture content increases peak density (Rautkari et al., 2011; Laine et al., 2013).

For samples to remain stable, without spring back or blow after thermal compression, the moisture content must reach close to the oven-dry state by the time the press is opened. Some species are greatly superior to others for producing compressed wood. The low permeability of some species may also inhibit steam movement and dissipation during hot pressing, causing spring back, blows and cracks (Wang and Cooper, 2005). Uniform and regular density profiles in densified wood are more likely to be achieved at high temperatures and with species featuring small diameter vessels (Tenorio et al., 2020).

Most research regarding densification have been conducted on wood from coniferous species and in close systems, and scarcely on wood from hardwood species, with the exception of beech wood, whose anatomical structures are more complex and have a greater influence on the result of the process (Navi and Heger, 2004). Jakob et al. (2022) compared the suitability of partially delignified sliced veneer sheets of domestic beech wood and sawn poplar wood for densification. The authors observed excessive thickness swelling in the presence of water, potentially as a result of a low pressing temperature (120°C).

Despite extensive investigation into wood modification techniques as outlined above, a striking gap persists regarding how densification protocols affect commercially cultivated plantation Eucalyptus species. This leaves a critical knowledge gap in the optimization of these protocols for commercial application.

In research by Esteves et al. (2007), steam treatment of *Pinus pinaster* and *Eucalyptus globulus* at 190–210 °C for 2–12 h reduced moisture content by 46% and 61%, and improved anti-shrinking efficiency by up to 57% and 90%, respectively. For *Eucalyptus globulus,* modulus of elasticity decreased by <15% and bending strength fell by 50% at 9% mass loss. The contact increased until 8% mass loss and then stabilized at 75–80° until 13% mass loss. *Eucalyptus globulus* showed higher mass loss (3.7–14.5%) but greater dimensional stability gains at moderate degradation. Cloutier et al. (2014) investigated steam-assisted thermo-hydro-mechanical densification of *Pinus radiata* and *Eucalyptus nitens* at 160–200 °C, 550 kPa steam pressure, and 4.5–9 MPa pressing. *Eucalyptus nitens* achieved greater densification and lower moisture uptake than *Pinus radiata*, as reflected by lignin-to-carbohydrate ratios of 0.78 versus 0.87 at 160 °C. FT-IR revealed distinct patterns of lignin condensation between the two species, while Py-GC/MS and XPS demonstrated marked carbohydrate breakdown and enhanced sugar accessibility, indicating 160 °C as the critical threshold for effective chemical modification. Balasso et al. (2020) investigated the effect of thermo-hydro mechanical treatments on *Eucalyptus nitens* and Tasmanian oak. The authors obtained promising results pressing thin lamellae but suggested further investigation to explain the different degrees of densification on the physical and mechanical properties, especially dimensional stability, spring back and variation in compression degree. Sargent et al. (2022) compared surface and bulk thermo-hydro-mechanical densification of 20 mm *Eucalyptus nitens* and *Eucalyptus fastigata* boards (compressed 15–50% under 170–200 °C steam at 3 MPa for 3 min), finding that *Eucalyptus nitens* tolerated higher compression, doubled surface hardness, and achieved only 30–40% set-recovery versus >40% for *Eucalyptus fastigata*, with density peaks within 1 mm of the surface. Subsequently, Sargent et al. (2023) demonstrated that grain orientation (flat vs. quarter-sawn) had negligible effect on densification outcomes when boards were treated at 200 °C under identical steam and pressure conditions, as *Eucalyptus nitens* again outperformed *Eucalyptus fastigata* in achievable densification ratios and exhibited lower set-recovery (7–32% under RH cycling), with densification profiles consistently concentrated at the compressed surface. The effects of heat-treatment temperature (165, 185, 205 °C) and duration (2–4 h) on the surface color (CIELAB L*, a*, b*, ΔE*) and gloss (20°, 60°, 85°) of young *Eucalyptus urophylla* × *grandis* were studied by Lu et al. (2022). The results showed that higher temperatures and longer treatments progressively darkened the wood (decreased L* and b*), shifted a* values, and highlighted temperature as the primary influence (*P*-value < 0.001). A desirable teak-like hue was achieved at 185 °C. Gloss measurements declined at all angles, especially at 85°, indicating smoother surfaces as treatment severity increased. Zhang et al. (2023) investigated the discoloration mechanisms of *Eucalyptus grandis* × *urophylla* wood subjected to conventional thermal treatment in hot air (180 °C, 12 h) versus saturated steam (130 °C, 12 h). Effects on chromophore structure were examined via CIELAB colorimetry, UV–Vis, FTIR, NMR, and XPS. Steam markedly enhanced hemicellulose depolymerization, lignin demethoxylation, and chromophore synthesis. These reactions diminished lightness and produced a pronounced red–dark hue similar to that of precious Burma padauk wood (*Pterocarpus macrocarpus*). Hot-air treatment produced comparatively minor modifications. Consequently, saturated-steam proved more effective for precise padauk-like coloration.

According to Schwarzkopf (2020), only a few wood densification applications have been industrialized to some extent. The reasons for this include inadequate consideration of plasticization or stability of the products. The main challenge associated with this type of densification is the fixation of the compressive deformation when the densified wood is exposed to moisture. Studies found that wood with the highest degree of compression shows the highest potential for compression deformation recovery or set-recovery (Blomberg et al., 2006; Kutnar et al., 2009). The set-recovery effect occurs because internal stresses introduced during compression are relieved when the wood is exposed to moisture. Several approaches to fixing set-recovery of densified wood are viable, including impregnation with a synthetic resin, mechanical fixation, or thermo-hydro-mechanical (THM) treatments at high temperature and moisture (Navi and Heger, 2004). The fixation of compressive deformation can be achieved with a sufficiently long treatment time and temperature (Heger et al., 2004). According to Wolcott and Shutler (2003), compression at high temperatures and low moisture content appears to result in kinetic changes in the cell wall polymers that may produce a stable compressed structure. A method to determine the set recovery of densified solid wood has been developed and proposed by (Laine et al., 2013; 2016).

A negative aspect of compressing bulk wood is the loss of wood volume. An alternative approach is to densify the surface only to a depth of 1 to 5 mm or only a few cell layers (Sandberg et al., 2013). Rautkari et al. (2011) developed a surface densification process, where only the first few millimetres beneath the wood surface are compressed. Increasing the density of the surface only to improve surface hardness rather than modifying the whole material could be an advantage in applications where it is desirable to maintain the low bulk density of wood but improve the surface properties (Rautkari et al., 2010). Furthermore, densifying the surface instead of throughout the whole thickness of the wood sample might require less energy in the densification process (Laine et al., 2013). However, further machining of the surface is limited because it could remove the densified layer.

Where mechanical properties and dimensional stability of compressed wood have been well documented, the literature is scarce on the impact of the densification process on the bond and finishing performance of densified materials. The present study aims to research and develop a sustainable and economically and logistically feasible modification techniques for Australian-grown Eucalyptus species. More specifically the objective was to systematically examine how key thermo-mechanical densification parameters such as pressing temperature, pressing time, and compression ratio, affect the functional and aesthetic performance of Tasmanian oak and *Eucalyptus nitens*. By subjecting the samples to controlled densification regimes, the research aimed to generate a comprehensive understanding of how these variables influence critical properties including the degree of color change, dimensional stability after soaking in water and under moisture cycling, surface adhesion of coatings, and bondability with adhesives. These insights are intended to inform the development of value-added applications for fast-growing hardwoods in interior and semi-structural contexts.

2 Materials and methods

2.1 Material preparation and Densification

Seasoned wood of thinned and pruned plantation *Eucalyptus nitens* (26-year-old, seasoned density at 12% MC: 554 kg m^{-3}) and regrowth Tasmanian oak species (i.e., *Eucalyptus regnans*, *Eucalyptus obliqua*, and *Eucalyptus delegatensis*, approx. 60 to 80 years, seasoned density at 12% MC: 683 kg m^{-3}) from Tasmania, Australia, was used in the present trial. On reception, the wood material has been conditioned at 23°C and 65% relative humidity (RH) until constant mass was reached (12% MC). Selected rift sawn lamellas of 275 mm x 90 mm x 16 mm (length x width x thickness) were prepared and then compressed, perpendicular to the grain, without steaming pretreatment. The densification process consisted of three stages adapted from Tenorio and Moya (2021): 1) preheating at 150°C or 175°C for 10 min; 2) compression perpendicular to the grain until reaching the target thickness of 12 mm (compression ratio: 25% or 37%) for 10 or 20 min, at the temperature maintained in stage 1; 3) cooling where the lamellas were kept compressed but without heat (platens temperature < 60°C) for an additional 10 min. Ten replicates per combination of parameters have been prepared for *Eucalyptus nitens* and Tasmanian oak for a total of 160 densified lamellas (Table 1). A minimum of 3 replicates for each of the species composing Tasmanian oak have been densified per combination of parameters. An example of a densification schedule for a 25% compression ratio using a 150°C pressing temperature for 10 min and showing the three (3) steps involved in the densification treatment (preheating, pressing, and cooling) is presented in Figure 1.

Table 1: Material and densification parameters.

Factor	
Species (2)	*Eucalyptus nitens* and Tasmanian oak
Pressing Time (2)	10 min & 20 min
Pressing temperature (2)	150°C and 175°C
Compression ratio (2)	25% (from 16 mm to 12 mm) & 37% (from 19 mm to 12 mm)

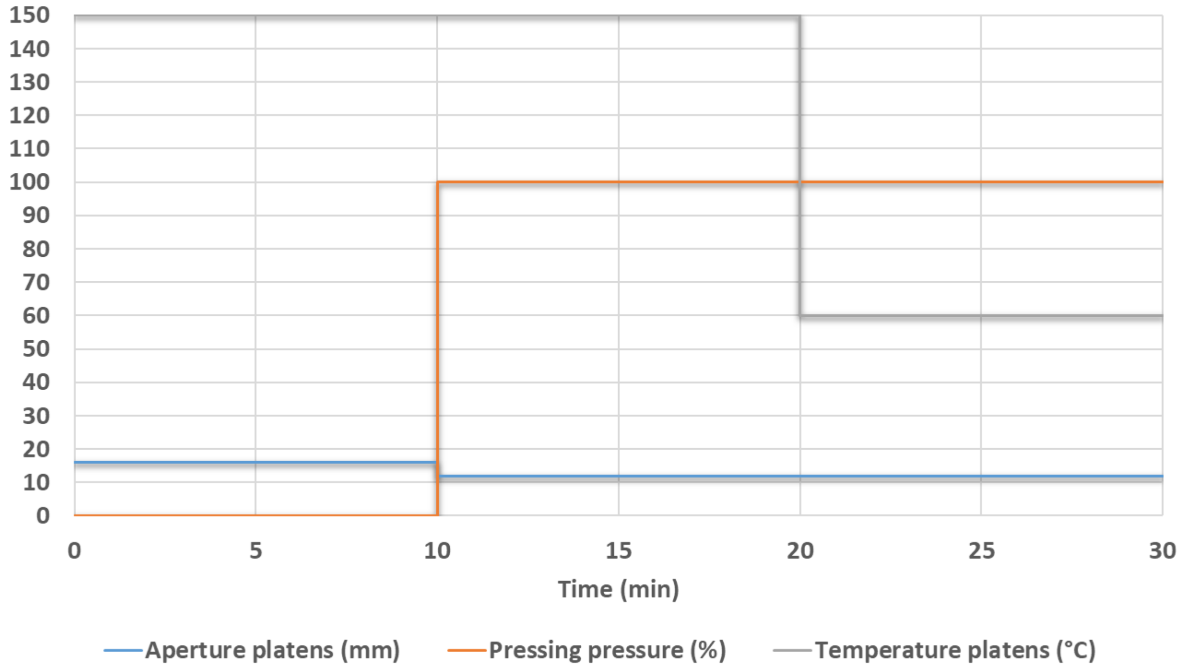

Figure 1: Example of densification schedule: 25% compression ratio at 150°C pressing temperature for 10 min. Time 0 to 10 min: preheating period; 10 to 20 min: pressing or densification of samples; 20 to 30 min: cooling period.

2.2 Post-densification assessment

The post-densification assessment of densified lamellas for the scale up trial included spring back after opening the press (immediate recovery) and color change or discoloration upon exposure to heated press platens. Thickness measurements have been taken at three (3) different points on each lamella before and after opening the press (S1, S2 and S3, Figure 2a) to determine spring back or immediate recovery (Eq. 1):

$$Spring\ back\ (\%) = \frac{T_d - T_t}{T_o - T_t} \times 100 \qquad \text{[Eq. 1]}$$

where T_d is the post-densification thickness after opening the press (mm), T_t the target thickness (mm), and T_o is the original thickness before densification (mm).

Color measurements were taken in four specific points (C1, C2, C3 and C4, Fig. 2a) on the surface of each lamella ($4 \times 4\ mm^2$) before and after the densification process using a BYK-Gardner digital color apparatus (Light source type D65, observation angle of 10°). CIELAB coordinates (a*, b*) and color difference (ΔE^*_{ab}) have been determined in accordance with ISO (2019).The evaluation of color differences has been performed using a chart proposed by Bieske in (Buchelt and Wagenführ, 2012).

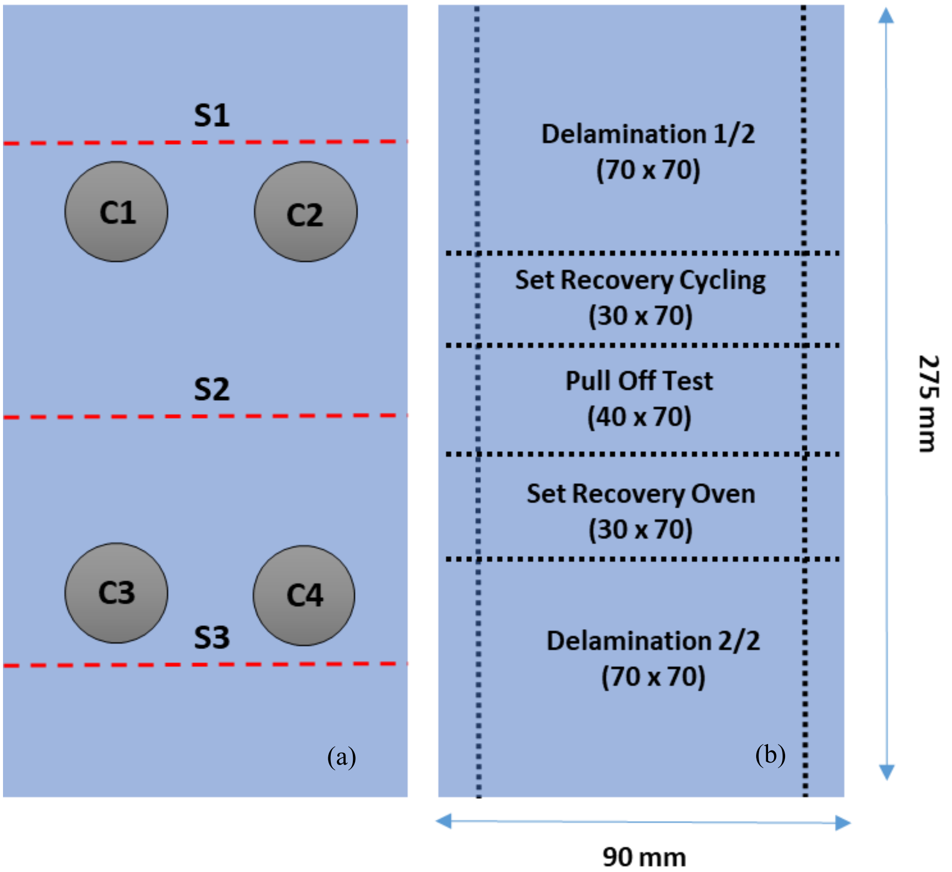

Figure 2: Schematic view of spring back (S) and color change (C) measurements on a lamella before and immediately after opening the press following densification (a) and machining of a densified lamella for further testing (b).

Two weeks after being returned at 23°C and 65% RH, the densified lamellas were machined to prepare samples for set recovery, adhesion of coating, and delamination tests (Figure 2b). Ten mm from both sides and ends of the lamella were removed. The set recovery consisted of two tests to assess the suitability of densified wood for specific use in defined environmental conditions i.e., dry-use or capable of producing sufficient dimensional stability to make the densified wood serviceable under conditions in which the equilibrium moisture content (EMC) does not exceed 17% and wet-use or capable of producing sufficient dimensional stability to make the densified wood serviceable under conditions in which the EMC may be 17% or higher.

The set recovery was first measured by soaking 30 mm x 70 mm samples in water for 24 h and measuring the oven-dried sample (40°C, 24 h) thickness and weight before and after soaking following the procedure described in Laine et al. (2016). Thickness was measured from each specimen and set-recovery calculated according to Eq. 2.

$$Set\ Recovery\ (wet-use, \%) = \frac{T_s - T_d}{T_o - T_d} \times 100 \qquad [\text{Eq. 2}]$$

where T_s is the recovered thickness after soaking and oven drying (mm) and T_d the post-densification thickness after 2 weeks at 23°C and 65% RH (mm).

Water absorption (WA) and thickness swelling (TS) of saturated samples after soaking for 2 h and 24 h were also calculated using Eq. 3 and 4.

$$TS\ (\%) = \frac{T_i - T_d}{T_d} \times 100 \qquad [\text{Eq. 3}]$$

where T_i is the recovered thickness after soaking in water for 2h or 24h and oven drying (mm).

$$WA\ (\%) = \frac{W_i - W_d}{W_d} \times 100 \qquad \text{[Eq. 4]}$$

where W_i is the weight after soaking in water for 2h or 24h and oven drying (g) and W_d the post-densification weight after 2 weeks at 23°C and 65% RH (g).

The set recovery in dry-use conditions (ΔT) has been measured by exposing densified 30 mm x 70 mm samples to different ambient conditions via two desorption/absorption cycles (Figure 3). The test simulates conditions which densified material may encounter in service i.e., humid climate (85% RH, 23˚C), moderate climate (65% RH, 23˚C), and dry climate (30% RH, 55˚C). Thickness measurements have been obtained every 7 days for a total 56 days and set-recovery calculated according to Eq. 5.

$$Set\ Recovery\ (\Delta T\ dry-use, \%) = \frac{T_j - T_d}{T_d} \times 100 \qquad \text{[Eq. 5]}$$

where T_j the recovered thickness after 7 days exposure to a specific climate (mm).

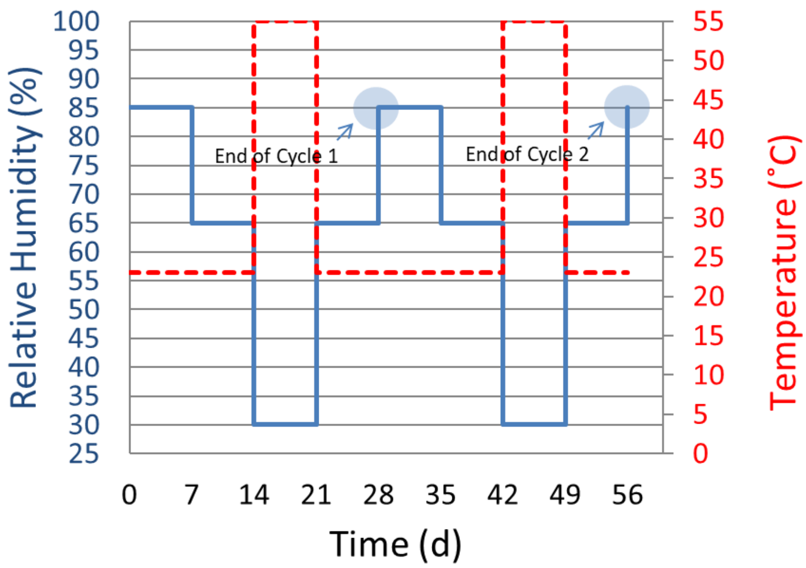

Figure 3: Wood moisture content cycling for the determination set recovery. Ambient conditions: humid climate (85% RH, 23˚C), moderate climate (65% RH, 23˚C), and dry climate (30% RH, 55˚C).

The effect of the densification process on the adhesion property of densified material has been evaluated using a pull-off strength test in accordance with ASTM (2022). An oil-based polyurethane coating system (Cabot's CFP Floor) has been applied (3 coats) with a brush on the surface of densified 40 mm x 70 mm samples. The bare densified surface was lightly sanded using 180 grit sandpaper prior to applying the first coat. The surface was again lightly sanded prior to application of the 2nd and 3rd coats using 240 grit sandpaper. One week after the application of the 3rd coat, a metallic dolly (20 mm in diameter) was glued to the surface of each sample with an epoxy adhesive and tested using a pull-off test apparatus (PosiTest AT, DeFelsko®, USA).

The performance of glued joints made from densified material has been assessed in various environmental conditions as part of a delamination test where a gradient was introduced in the moisture content of the wood to build up internal stresses, resulting in tensile stresses perpendicular to the glueline. Individual 70 mm x 70 mm densified samples were face laminated using a liquid one-component polyurethane adhesive for structural wood bonding (Jowapur 681.60). The specimens were then trimmed down to 65 mm x 65 mm to remove the glue excess. The specimens were first placed in a pressure vessel and completely submerged in water. A vacuum of 70 kPa (20 in. Hg) was first drawn and held for 5 min. Then, samples were pressurised at 500 kPa (72.5 psi) for 1 h. This vacuum-pressure cycle was repeated once more. The samples were finally dried for a period 22 h in air at 60°C and < 15% RH. After the cycle, the specimens have been evaluated for glueline delamination i.e., length of delamination by the total length of exposed bond line (%) as per ASTM (2023).

2.3 Statistical analysis

The data were analyzed in Minitab statistical software (Minitab Version 21.3) to evaluate the effect of the densification parameters on the properties of each selected species. A general linear model and a Fisher pairwise comparisons analysis (α = 0,05) were used to compare the different sets of densification parameters. Ad-hoc comparisons with respective control groups were also conducted were applicable (i.e., pull-off test, delamination). An increment was used, and a log transformation applied when there were negative values (i.e., wood moisture cycling).

3 Results and discussion

Both Tasmanian oak and *Eucalyptus nitens* have been densified successfully, showing no sign of spring back (i.e., 0% spring back immediately after opening the press), using both 25% and 37% compression ratios (Table 2).

Density of densified Tasmanian oak at 12% EMC using 25% and 37% compression ratios were 846 and 1029 kg m^{-3}, respectively. Density of densified *Eucalyptus nitens* at 12% EMC using 25% and 37% compression ratios were 713 and 847 kg m^{-3}, respectively.

Table 2: Average spring back (%) of densified *Eucalyptus nitens* and Tasmanian oak after opening the press as a function of pressing time, pressing temperature and compression ratio.

Pressing temperature (°C)	**150**				**175**			
Pressing time (min)	**10**		**20**		**10**		**20**	
Compression ratio (%)	**25**	**37**	**25**	**37**	**25**	**37**	**25**	**37**
Eucalyptus nitens	-1.3 (0.5)	1.3 (1.8)	-1.5 (0.7)	0.0 (1.5)	-1.5 (1.7)	0.3 (2.0)	-1.7 (1.6)	0.1 (3.7)
Tasmanian oak	-1.3 (0.7)	0.1 (2.6)	-1.5 (0.7)	0.4 (1.7)	0.0 (2.0)	-0.7 (1.8)	-2.1 (0.8)	0.2 (3.2)

Average of 10 lamellas. Standard deviation is presented in parentheses.

3.1 Color change

The color change or discoloration (ΔE*ab) of densified lamellas before and after the densification process has been completed, representing a total of 1,280 measures. A summary of ΔE*ab following densification is presented in Table 3.

Table 3: Average color change (ΔE*ab) of densified *Eucalyptus nitens* and Tasmanian oak as a function of pressing time, pressing temperature and compression ratio.

Pressing temperature (°C)	**150**				**175**			
Pressing time (min)	**10**		**20**		**10**		**20**	
Compression ratio (%)	**25**	**37**	**25**	**37**	**25**	**37**	**25**	**37**
Eucalypts nitens								
Δ E^*_{ab}	1.84 (0.73)	1.68 (0.86)	1.82 (0.41)	2.72 (1.56)	3.21 (0.87)	3.20 (1.03)	4.10 (0.89)	4.29 (1.58)
Δ L^*	-1.18 (0.76)	-1.19 (1.02)	-1.26 (0.37)	-2.20 (1.42)	-2.49 (0.68)	-2.64 (0.99)	-3.24 (0.74)	-3.71 (1.53)
Δ a^*	-0.06 (0.19)	-0.04 (0.12)	-0.12 (0.15)	0.06 (0.32)	0.05 (0.33)	0.02 (0.32)	0.19 (0.35)	0.27 (0.24)
Δ b^*	1.15 (0.52)	1.00 (0.30)	1.24 (0.38)	1.42 (0.90)	1.98 (0.54)	1.71 (0.60)	2.33 (0.85)	2.08 (0.64)
Δ Gloss	0.4 (0.3)	0.2 (0.3)	0.4 (0.1)	0.0 (0.5)	0.5 (0.2)	0.3 (0.1)	0.5 (0.2)	0.2 (0.3)
Tasmanian oak								
Δ E^*_{ab}	1.71 (0.30)	1.81 (0.74)	2.11 (0.54)	2.71 (0.46)	6.17 (1.94)	6.54 (1.30)	6.92 (1.75)	9.06 (2.49)

Δ *L**	-1.18 (0.26)	-0.95 (0.85)	-1.52 (0.49)	-2.05 (0.87)	-5.73 (2.11)	-6.23 (1.46)	-6.53 (1.88)	-8.85 (2.55)
Δ *a**	-0.64 (0.43)	-0.49 (0.35)	-0.74 (0.22)	-0.58 (0.17)	-1.09 (0.35)	-0.85 (0.23)	-1.22 (0.17)	-0.99 (0.24)
Δ *b**	0.76 (0.49)	1.27 (0.47)	1.05 (0.57)	1.32 (0.68)	1.49 (0.79)	1.62 (0.45)	1.44 (0.90)	1.30 (0.90)
Δ Gloss	0.4 (0.1)	0.5 (0.2)	0.3 (0.1)	0.2 (0.3)	0.4 (0.2)	0.3 (0.3)	0.2 (0.2)	0.0 (0.3)

Average of 10 lamellas (4 measures per lamella). Standard deviation is presented in parentheses.

Eucalyptus nitens showed a low color change ($\Delta E^*_{ab} < 5$) across all the pressing conditions (Table 3). Since only ΔE^*_{ab} values > 5 are generally visible to the naked eye, these changes remained effectively imperceptible (Buchelt and Wagenführ, 2012). Although longer pressing times and higher temperatures increased ΔE^*_{ab} slightly, the variation still fell below the threshold of visual detection. The overall trend observed with *Eucalyptus nitens* was a shift in ΔL in the negative direction, meaning that the samples have become slightly darker after the densification process. The second component of the overall color change was a positive Δb*, meaning that the samples have become slightly yellower after the densification process. The final component contributing to the overall ΔE color change was a negative or positive Δa* as the samples have become marginally greener or redder following the densification process. Gloss slightly increases when going from a pressing temperature of 150°C to 175°C.

Tasmanian oak followed the same directional trends but proved more heat-sensitive (Table 3). At 150 °C (10 or 20 min) ΔE*ab remained < 5 , yet at 175 °C perceptible changes emerged (ΔE*ab ≈ 6.2 and 6.9 and 6.54 and 9.06 for 10 and 20 min press times and 25 and 37% CR, respectively). Like Eucalyptus nitens, color change variability widened with temperature and samples darkened (negative ΔL* increase) and yellowed (Δb* > 0), with small negative Δa* values indicating a slight shift toward green. Gloss also increased after densification, though without a clear dependence on pressing time or temperature.

At 150°C, the temperature was too low to induce significant de-acetylation of hemicellulose or substantiative lignin modifications (Lu et al., 2022; Gašparík et al., 2024). Consequently, there was no perceptible impact on the color of neither Eucalyptus nitens nor Tasmanian oak. Likewise, Cloutier et al. (2014) found that densifying Eucalyptus nitens at 160–180 °C left its lignin-condensation index essentially unchanged (≈ 0.76) and produced only minor carbohydrate loss, confirming that such mild conditions generate few new chromophores. The markedly greater color change in Tasmanian oak at 175 °C likely stemmed from its higher content of phenolic- and tannin-rich extractives. During thermal treatment these extractives oxidised to red-brown pigments. Simultaneously, lignin condensed into quinonoid chromophores, and acids released from hemicellulose catalysed both reactions, collectively intensifying darkening of the wood surface (Zhang et al., 2023).

3.2 Set recovery – Soak in water and oven-dry (wet-use)

The average set recovery of densified *Eucalyptus nitens* and Tasmanian oak as a function of pressing time, pressing temperature and compression ratio are presented in Table 4. *WA* and *TS* results are presented in Figure 4 and Figure 5. An increment has been used and a Box-Cox transformation applied as part of the statistical analysis.

Table 4: Average set recovery (%) of densified *Eucalyptus nitens* and Tasmanian oak following immersion in water as a function of pressing time, pressing temperature and compression ratio.

Pressing temperature (°C)	**150**				**175**			
Pressing time (min)	**10**		**20**		**10**		**20**	
Compression ratio (%)	**25**	**37**	**25**	**37**	**25**	**37**	**25**	**37**
Eucalyptus nitens	9.4 (8.8)	10.5 (6.1)	8.4 (13.7)	8.5 (8.9)	8.5 (13.9)	2.0 (3.0)	8.7 (14.0)	0.0 (1.9)
Tasmanian oak	8.4 (15.9)	10.3 (4.2)	5.3 (14.4)	9.2 (4.7)	11.0 (17.7)	2.6 (3.7)	5.5 (11.6)	2.7 (4.3)

Average of 10 lamellas. Standard deviation is presented in parentheses.

The statistical analysis revealed that pressing temperature significantly affected the average set-recovery of densified *Eucalyptus nitens* (*P*-value 0.003). Specimens hot-pressed at 175 °C rebounded markedly less than those treated at 150 °C. Elevated temperatures accelerate viscoelastic relaxation of cell-wall polymers, as proposed by

Laine et al. (2014) and confirmed by Paul et al. (2024), whose meta-analysis (>100 studies) argues that heating above the species-specific *Tg* unlocks chain mobility, promotes new bond formation, and is essential for fixing deformation in thermally-densified wood. This outcome was corroborated by findings of Sargent et al. (2022), who reported that bulk-densified *Eucalyptus nitens* compressed slightly above its *Tg* regained only 30–40 % of its original thickness, whereas surface-densified panels along with all *Eucalyptus fastigata* equivalents rebound almost completely. In a subsequent investigation, Sargent et al. (2023) demonstrated again the overriding importance of temperature, achieving <1 % residual set-recovery after five soak–dry cycles when bulk-densifying at 200 °C, compared with ≈3 % at 175 °C in the present study. Collectively, these data indicate that higher pressing temperatures promote enhanced viscoelastic flow and stress relaxation within the lignocellulosic framework, thereby permanently stabilising the compressed geometry.

However, this was not the case for Tasmanian oak as none of the densification parameters had a significant effect on set recovery. Similarly, Balasso et al. (2020) observed that, although densification increased both density and hardness in *Eucalyptus nitens* and Tasmanian oak, only *Eucalyptus nitens* displayed a substantial reduction in post-soak swelling, while Tasmanian oak remained essentially temperature-insensitive. Collectively, the results underscore the pivotal role of starting density. The higher native density of Tasmanian oak (~650 kg m^{-3}) restricts further compression and restricts polymer mobility during pressing, whereas the lower-density *Eucalyptus nitens* (~500 kg m^{-3}) allows greater stress relaxation and permanent fixation.

A longer pressing time appears to improve set recovery for both *Eucalyptus nitens* and Tasmanian oak species, although not significantly. This observation is especially noticeable when a lower pressing temperature was used, suggesting that sufficient time is required for the cell wall components to relax while the densification process is occurring. No interactions between the densification parameters and set recovery have been observed for the selected species. Interestingly, a higher compression ratio had a significant positive effect on water absorption for both Tasmanian oak (*P*-value 0.025) and *Eucalyptus nitens* (*P*-value 0.002). The findings are supported by Balasso et al. (2020), Pelit and Emiroglu (2021), and Sargent et al. (2022) all of whom identified mechanical compaction of porous wood structures as the key mechanism behind reduced water absorption. By collapsing lumens, vessels, and other capillary pathways, higher compression ratios effectively limit both free and bound water uptake, enhancing the wood's dimensional stability.

Thickness swelling and thickness swelling variability both appeared to reduce by extending pressing time and/or pressing temperature, suggesting a more consistent and uniform densification process. A higher pressing temperature had a significant positive effect on thickness swelling for *Eucalyptus nitens* (*P*-value 0.006). *Eucalyptus delegatensis* did not appear to respond as well as *Eucalyptus obliqua* and *Eucalyptus regnans* across all the densification conditions tested in the present study. Limited sampling prevented making any conclusive conclusions with confidence. Further analysis would be needed to validate the observed trends in cases where Tasmanian oak might be considered for densification applications. Set recovery for all studied species could be minimised significantly with the application of an appropriate protective sealant and potentially be used under conditions in which the EMC of densified wood may be 17% or higher.

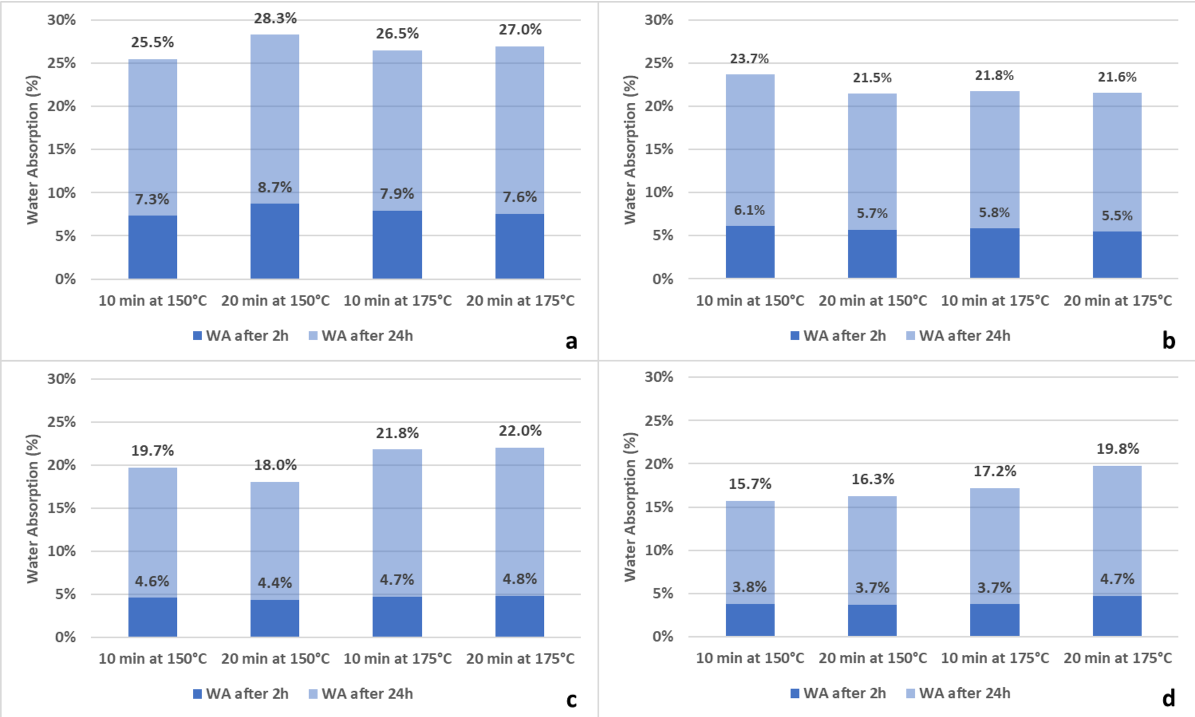

Figure 4: Water absorption (*WA*, %) of densified *Eucalyptus nitens* and Tasmanian oak after soaking in water for 2 h and 24 h as a function of pressing time, pressing temperature and compression ratio (*CR*). a) *Eucalyptus nitens* using a 25% CR; b) *Eucalyptus nitens* using a 37% CR; c) Tasmanian oak using a 25% CR; d) Tasmanian oak using a 37% CR.

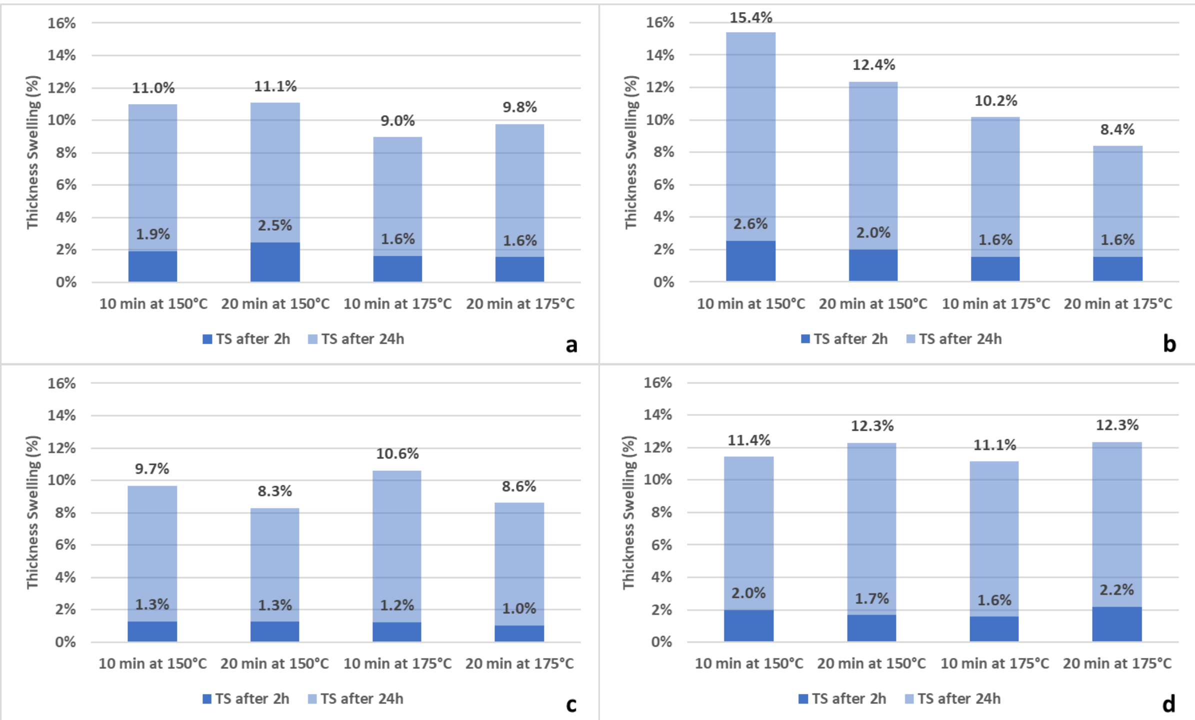

Figure 5: Thickness swelling (*TS*, %) of densified *Eucalyptus nitens* and Tasmanian oak after soaking in water for 2 h and 24 h as a function of pressing time, pressing temperature and compression ratio (CR). a) *Eucalyptus nitens* using a 25% CR; b) *Eucalyptus nitens* using a 37% CR; c) Tasmanian oak using a 25% CR; d) Tasmanian oak using a 37% CR.

3.3 Set recovery – Ambient humidity cycling (dry-use)

A graphic summarising the overall set recovery results after exposure to different ambient conditions is presented in Figure 6.

An analysis of variance (ANOVA) performed on the set recovery data collected over successive 7-day intervals under controlled ambient conditions . A very significant difference was found between samples densified using a compression ratio of 37% and those densified using a compression ratio of 25% for both *Eucalyptus nitens* and Tasmanian oak. The highest *F*-values observed for *Eucalyptus nitens* and Tasmanian oak were 25.36 and 17.91, respectively. A compression ratio of 37% consistently outperformed 25%, suggesting that voids being totally closed, as opposed to being partially closed, allowed for physio-chemical bonding in the closed lumens. Fixing the deformation and giving greater stability to the densified *Eucalyptus nitens* boards was equally observed by Sargent et al. (2023). Navi and Heger (2004) demonstrated via X-ray micro-CT that at greater compression cell walls come into contact, allowing new inter-cellular hydrogen bonds to lock the collapsed structure once cooled below *Tg*.

Following the absorption stage from 23°C and 65% RH to 23°C and 85% RH, further analysis demonstrated that pressing temperature significantly influences set recovery (P-value = 0.004). Samples pressed at 175 °C exhibited notably lower set recovery compared to those pressed at 150 °C. This observation aligns with findings from the water immersion tests (Table 4), which showed that elevated pressing temperatures enhance the dimensional stability of densified wood, reinforcing the hypothesis of more effective relaxation of inner stresses and bonding within the wood cell wall components at higher temperatures (Murata et al., 2023; Cabral et al., 2024).

Consistent with Hill et al. (2012) and Fredriksson and Thybring (2019), the characteristic sorption hysteresis of wood remained evident in the densified samples. Under identical ambient conditions, equilibrium moisture content was consistently higher following desorption than after absorption.

However, the overall average set recovery was more important in *Eucalyptus nitens* than Tasmanian oak across all tested conditions, especially with a compression ratio of 37%. Comparative analysis of set recovery over two moisture content cycles revealed that both *E. nitens* and Tasmanian oak maintained dimensional stability post-densification, particularly under dry-use conditions where the equilibrium moisture content (EMC) does not exceed 17%. These findings suggest that thermo-mechanically densified wood is suitable for applications in environments with moderate humidity levels (<17 % EMC). Moreover, the application of appropriate protective sealants could further minimize set recovery, potentially extending the usability of densified wood products to conditions with higher EMC values.

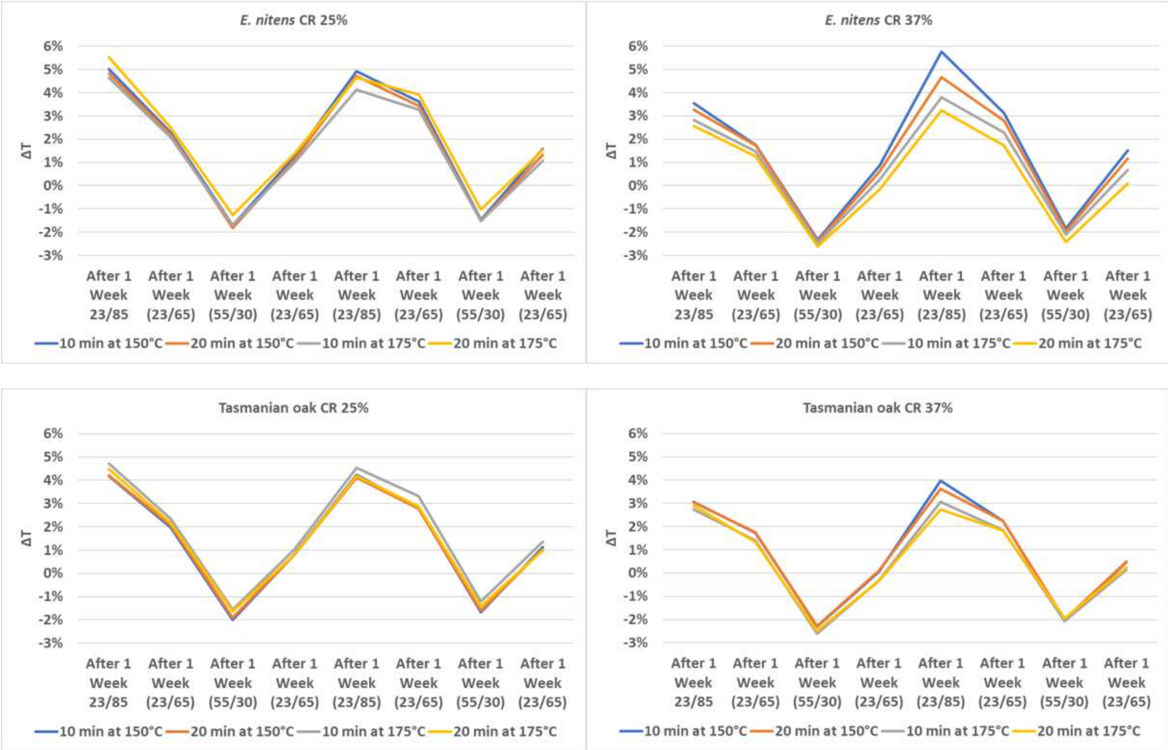

Figure 6: Set recovery (*ΔT*) of densified *Eucalyptus nitens* and Tasmanian oak after exposure to humid climate (85% RH, 23˚C), moderate climate (65% RH, 23˚C), and dry climate (30% RH, 55˚C) as a function of pressing time, pressing temperature and compression ratio (*CR*).

3.4 Adhesion of coating (Pull-off strength test)

The results of the pull-off strength test are presented in Table 5.

Table 5: Pull-off strength (MPa) test results for control and densified Tasmanian oak and *Eucalyptus nitens* as a function of pressing temperature, pressing time and compression ratio.

Pressing temperature (°C)	Control	150				175			
Pressing time (min)		10		20		10		20	
Compression ratio (%)		25	37	25	37	25	37	25	37
Eucalyptus nitens	4.07^{A} (0.37)	3.22^{CD} (0.70)	3.74^{AB} (0.56)	3.05^{D} (0.56)	3.78^{AB} (0.33)	3.33^{BCD} (0.45)	3.38^{BCD} (0.44)	3.15^{D} (0.48)	3.69^{ABC} (0.82)
Tasmanian Oak	4.32^{A} (0.30)	3.82^{BC} (0.48)	4.43^{A} (0.54)	3.57^{CD} (0.22)	4.10^{AB} (0.44)	3.76^{BCD} (0.33)	3.74^{BCD} (0.75)	3.27^{D} (0.47)	3.72^{BCD} (0.89)

Average of 10 lamellas. Standard deviation is presented in parentheses. Means that do not share a letter are significantly different.

The analysis showed that compression ratio had a highly significant effect on the pull-off strength of densified *Eucalyptus nitens* (*P*-value < 0.001) and a significant effect for densified Tasmanian oak (*P*-value 0.003). When assessed individually, a compression ratio of 37% consistently produced a significant or very significant positive effect in both species compared to 25%.

For Tasmanian oak, both pressing time (*P*-value 0.040) and pressing temperature (*P*-value 0.007) were also significant factors. Notably, samples subjected to shorter pressing times and lower pressing temperatures exhibited higher pull-off strength. This outcome may be linked to the increased diffusion coefficient at elevated temperatures. Given Tasmanian oak's relatively high extractive content compared to *Eucalyptus nitens*, higher temperatures may enhance extractive migration to the surface, potentially hindering adhesion of finishing systems (Jankowska et al., 2018). However, the delamination test using a polyurethane adhesive showed no significant effect of the densification process (Table 6). This suggests that while surface densification altered the wood surface, it did not impair the mechanical interlocking or chemical bonding required by the polyurethane adhesive.

In contrast, the oil-based polyurethane coating system, which relies more heavily on surface wetting and chemical compatibility with extractives, appeared to be more sensitive to changes in surface chemistry caused by extractive migration. This highlights that identical surface modifications may influence different bonding systems in distinct ways, depending on their mode of adhesion.

A comparison between densified samples and controls (Fisher LSD method, 95%) confirmed that the densification process has no or limited impact on the adhesion of a finishing system for both selected species. Interestingly and even though densification of wood samples has been carried out in the radial direction, the present study allowed observing a "banded" pattern which might be similar to the zig-zag pattern observed previously by Sandberg et al. (2013), Kutnar et al. (2015), and Sotayo et al. (2020) when densifying wood in the radial direction as a result of buckling of the latewood annual rings.

3.5 Delamination test

The performance of glued joints made from densified material has been assessed as part of a delamination test where a gradient was introduced in the moisture content of the wood to build up stresses to the glueline. The total delamination (%) results for each species per pressing conditions are presented in Table 6.

Table 6: Average total delamination (%) of control and densified samples per species and densification parameters.

Pressing temperature (°C)	**Control**	**150**				**175**			
Pressing time (min)		**10**		**20**		**10**		**20**	
Compression ratio (%)		**25**	**37**	**25**	**37**	**25**	**37**	**25**	**37**
Eucalyptus nitens	0.0^{A} (0.0 - 0.0)	1.6^{AB} (0.0 - 8.1)	1.5^{AB} (0.0 – 4.7)	0.2^{A} (0.0 - 0.9)	0.9^{A} (0.0 - 4.5)	3.0^{AB} (1.3 - 7.3)	3.8^{AB} (0.0 - 16.9)	5.8^{B} (0.0 - 13.3)	0.0^{A} (0.0 - 0.0)
Tasmanian Oak	22.6^{AB} (0.0 – 85.9)	33.9^{AB} (6.5 – 66.6)	44.9^{B} (1.7 - 94.1)	20.3^{AB} (1.0 - 48.4)	14.6^{AB} (2.0 – 34.0)	17.8^{AB} (0.0 - 53.1)	5.5^{A} (0.0 – 21.7)	14.4^{AB} (3.0 – 46.5)	7.3^{A} (0.0 – 26.9)

Average of 5 densified laminated lamellas. Length of delamination divided by the total length of exposed bond line (%). Delamination range in parentheses. Means that do not share a letter are significantly different.

The statistical analysis revealed a significant effect of pressing temperature on delamination for Tasmanian oak (*P*-value 0.031). A higher pressing temperature showed to have a positive effect on reducing delamination following a vacuum-pressure and oven-drying cycle. As mentioned previously, Tasmanian Oak has a higher density and unit shrinkage then *Eucalyptus nitens* and a higher temperature might have enabled relaxation of inner stresses inside cell wall components to take place. The pressing parameters had negligible effect on total delamination for *Eucalyptus nitens*.

A comparison between densified samples and controls (Fisher LSD method, 95%) confirmed that the densification process has no or limited impact on the bondability for both selected species. Where a higher temperature appears to improve total delamination of Tasmanian oak samples, splits and cracks occurring outside the glueline were not compiled in the total delamination results. The presence of such cracks suggest that buckling in Tasmanian oak species could not be totally prevented based on selected studied conditions.

4 Conclusion

This study investigated the influence of pressing temperature, pressing time, and compression ratio on the material properties of thermo-mechanically densified *Eucalyptus nitens* and regrowth Tasmanian oak. The results demonstrate that thermo-mechanical densification is an effective method for improving the qualitative and functional characteristics of low-density Australian hardwoods, although the outcomes are strongly dependent on species-specific responses and processing conditions.

Pressing temperature had the most substantial impact across all tested properties, with statistically significant or highly significant effects observed for set recovery, delamination resistance, and coating performance. A pressing temperature of 175 °C markedly improved dimensional stability following both water immersion and cyclic humidity exposure. However, these improvements came with notable trade-offs. Elevated temperatures resulted in reduced pull-off strength, particularly on Tasmanian oak, and led to visually perceptible discoloration, with

ΔE*ab values exceeding the commonly accepted visibility threshold. These effects were attributed to thermally induced changes in surface chemistry and the migration of extractives, which were more pronounced in Tasmanian oak than in *Eucalyptus nitens*. Compression ratio was the second most influential parameter, with a 37% compression ratio consistently outperforming the lower 25% ratio in terms of set recovery and pull-off strength. Notably, the benefits of higher compression were especially evident under moderate pressing temperatures, indicating a possible compensatory effect when thermal input is limited. Pressing time had a more moderate influence overall, although a shorter pressing duration was found to enhance the pull-off strength in Tasmanian oak. In contrast, *Eucalyptus nitens* maintained relatively consistent surface adhesion regardless of pressing time, reflecting its greater thermal stability and lower extractive content. Species-specific behaviour was observed throughout the study. While both timbers responded well to densification, *Eucalyptus nitens* demonstrated superior dimensional stability, color retention, and overall consistency across all pressing conditions. Tasmanian oak, by contrast, was more sensitive to changes in pressing temperature and time, particularly with regard to surface properties and discoloration. A comparison of set recovery results following wood moisture content cycling suggests that all studied species remained dimensionally stable after densification and are suitable for dry-use environments, particularly where the equilibrium moisture content (EMC) does not exceed 17%. With the application of a suitable protective sealant, the dimensional stability of densified wood may be further enhanced, enabling potential use in conditions exceeding 17% EMC.

All samples remained free of spring back after a standardised 10-minute cooling period, indicating effective fixation of deformation across all processing conditions. While this cooling time appears sufficient for dimensional stabilisation, future investigations should assess whether shorter cooling phases could deliver comparable results while increasing processing efficiency. The present study highlights the feasibility of tailoring thermo-mechanical densification parameters to improve specific performance attributes in Australian plantation and regrowth hardwoods. This includes optimising density, surface properties, and moisture-related dimensional behaviour in alignment with target end-use applications. However, other potentially influential factors such as initial moisture content, grain orientation, and post-treatment conditioning were not examined in the present study and warrant further investigation to support wider industrial adoption.

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