DENSIFICATION OF NEW ZEALAND-GROWN EUCALYPTUS SPECIES: EFFECT OF GRAIN ORIENTATION AND DENSIFICATION PROCESS ON WOOD PROPERTIES

R. Sargent*

Senior Technologist Scion, Titokirangi Drive, Rotorua 3010, New Zealand Email: rosie.sargent@scionresearch.com

M. Mikuljan

Assistant Researcher Email: marica.mikuljan@innorenew.eu

A. Kutnar[†]

Director InnoRenew CoE, Livade 6a, Izola 6310, Slovenia and University of Primorska, Andrej Marušič Institute Muzejski trg 2, Koper 6000, Slovenia Email: andreja.kutnar@innorenew.eu

(Received May 2023)

Abstract. *Eucalyptus fastigata* and *Eucalyptus nitens* were densified using a thermo-hydro-mechanical (THM) densification process. The THM treatment was applied either as a surface densification of one wood surface or as a bulk densification of the entire wood thickness. To understand the effect of grain orientation on final wood properties, both quarter-sawn and flat-sawn boards were densified. The *E. nitens* boards were able to be compressed to a greater degree without being damaged compared with the *E. fastigata* boards. This led to substantial increases in surface hardness and surface density in *E. nitens*. Additionally, levels of set-recovery (irreversible swelling from contact with water) for bulk densified *E. nitens* were substantially lower than *E. fastigata* and lower than literature values for other species with a similar density. The reason for this unusually low set-recovery is unacceptable and would need to be eliminated. Density profiles showed that the peak density was generally at, or very close to, the wood surface, giving the maximum increase in surface hardness for a given degree of densification. The properties following densification were not substantially different between the quarter-sawn and flat-sawn boards, suggesting that densification was effective irrespective of grain orientation.

Keywords: Eucalyptus, wood densification, density profile, Brinell hardness, hardwood, grain orientation.

INTRODUCTION

Increased demand worldwide for high-value wood products, plus concerns about deforestation and unsustainable logging, have increased interest in plantation-grown timbers that will perform well in demanding situations. There is also increasing interest in utilizing a wider range of wood species for sawn timber and using wood modification as a method of improving wood properties. *Eucalyptus* nitens (H.Deane & Maiden) Maiden (*E. nitens*) and *Eucalyptus fastigata* H.Deane & Maiden (*E. fastigata*) are grown in plantations in New Zealand but are not currently well utilized for high-value sawn timber. Both species have an attractive hardwood grain, similar to other species of Australian-grown eucalyptus that are commonly used for interior applications such as hardwood flooring. Neither species is as hard as oak, or Australian-grown hardwoods (Janka hardness of ~5 kN for *E. fastigata*, and *E. nitens*, compared with ~7 kN for American Oak and

^{*} Corresponding author

[†] Society of Wood Science & Technology member

 \sim 6 kN for Victorian Ash) and are thus not sufficiently hard for flooring applications. Many mechanical properties of wood, eg, MOE and hardness, are proportional to wood density (Rautkari et al 2011; Navi and Sandberg 2012a), so increasing the wood density via densification offers a potential route to improving the surface hardness of these species. Densification of wood has been studied for a long time, having been initially developed in the early 20th century, but there has been increased scientific interest in the last 30 yr (Kutnar et al 2015).

Wood densification is a process where wood cells are mechanically compressed so they deform, reduce the void spaces, and flatten, preferably without the cell wall fracturing. If the cell deformation can be retained after the compression force is removed, this will increase the wood density and, consequently, the wood hardness. To ensure that the wood retains its densified shape after the compression force is released, it is important that the cells are softened and the stresses that form during compression are released. This is done by ensuring the wood components (mainly lignin) are above the glass transition temperature (T_{g}) during compression, and the wood is cooled to below the glass transition temperature before the compressive load is released (Navi and Sandberg 2012a). Because the glass transition temperature of lignin and hemicellulose are a function of temperature and moisture content (MC) (Lenth and Kamke 2001a; Kutnar and Sernek 2007), the correct combination of press temperature and wood MC is required to ensure the cells are densified without being damaged, and stresses are adequately relieved to retain the compressed shape. To ensure the wood remains in a softened state, the wood either needs to be at a high temperature or maintain a consistently high MC during the densification process. For example, Lenth and Kamke (2001a) observed softening at 200°C in Pinus taeda at 0% MC, but this reduced to <100°C at 10-15% MC. To maintain high moisture contents at temperatures $>100^{\circ}$ C, elevated pressures are required (Lenth and Kamke 2001b). For this reason, densification is often performed in a "closed system" where mechanical force can be applied in a pressurized steam atmosphere (Navi and Sandberg 2012a).

Once the wood is set in its compressed state, contact with liquid water or changes in air humidity often lead to the wood swelling and regaining some of its original dimensions, a process known as "set-recovery" (Navi and Sandberg 2012a). Preventing set-recovery has been the subject of numerous studies (Kutnar and Kamke 2012; Laine et al 2013). Postdensification heat treatment, such as pressure steaming or thermal modification, is a very promising method of preventing set-recovery (Laine et al 2016). Kutnar and Kamke (2012) compared levels of set-recovery after densifying poplar in either saturated or atmospheric pressure steam, finding dramatically lower levels of set-recovery following water soaking for the samples compressed in pressurized (saturated) steam. Set-recovery is typically measured by comparing the initial specimen thickness with the thickness following water soaking and oven drying. This is often repeated over several water soaking steps (Fu et al 2017). Laine et al (2013) compared several different methods of quantifying set-recovery, including water soaking in hot or cold water and through changes in ambient humidity. They noted that set-recovery through changes in RH was more likely to replicate the conditions that many target products for densified wood would encounter in service, eg, wooden flooring.

Process conditions such as press temperature, initial wood temperature and MC, the degree of compression, and the rate of compression all impact the density profile and peak density (PD) of the compressed wood (Rautkari et al 2011; Zhou et al 2019). This gives the possibility of increasing the density throughout the entire wood thickness or restricting the densification to the wood surface, leaving the rest of the wood unchanged (Navi and Sandberg 2012b). Surface densification has the advantage of retaining more of the original wood thickness during densification and gives a product with a high surface hardness but with only a small increase in the overall density and weight of the wood.

Densification is typically performed in a radial orientation (compression force perpendicular to the growth ring orientation). This preferentially densifies the larger thinner-walled earlywood fibers (Kutnar et al 2015), and corresponds to the grain orientation typical of sawn timber of many softwoods (flat-sawing or back-sawing). Many eucalypt species are typically milled into quartersawn boards where the growth rings are perpendicular to the long face of the board. Densifying these boards would compress the wood in a tangential direction, meaning that both the earlywood and latewood bands are compressed at the same time. Wang and Cooper (2005) densified black spruce and balsam fir with three different grain orientations (growth rings oriented 0° , 45° , and 90° from the wide face of the board). For balsam fir, the density profile before densification showed distinct peaks for the late wood bands in the 0° (flat-sawn boards). These differences were retained after densification, with both the earlywood and latewood density increasing. For the 45° and 90° (quarter-sawn) boards, the vertical density profile was very uniform prior to modification, and following modification, there were definite density peaks near each surface of the board with a lower density in the center. For surface densification, where these density peaks are being sought, grain orientation may have a significant impact on the density profile, and hence on the success of the process (Wang and Cooper 2005).

Despite densification being a well-known process, it has not yet been investigated for New Zealandgrown eucalypts. Koumba et al (2014) densified Chilean-grown *E. nitens* and *Pinus radiata* in a steam environment but did not report any mechanical properties or dimensional stability data. Balasso et al (2020) densified a thin lamella of *E. nitens*, *P. radiata*, and Tasmanian Oak (a mix of *Eucalyptus regnans* and *Eucalyptus obliqua*). In this study, timber from *E. nitens* and *E. fastigata* has been densified using two densification processes (surface- and bulk densification), and with two grain orientations (flat-sawn and quarter-sawn), and then the properties of the densified wood were investigated.

MATERIALS AND METHODS

Densification

E. fastigata boards were sourced from a previous sawing study (Jones et al 2010). The trees were 25 yr old, and boards were cut from either the butt log or the 1st log of each tree. The boards were cut from 10 different trees. The E. nitens boards were also sourced from a sawing study using 18-yr-old trees grown in the southern South Island of New Zealand. These boards were all from the 1st log and were also cut from 10 different trees. For each species, equal numbers of flatand quarter-sawn boards were cut to 500 mm long and were planed to 20 mm thick. Most boards were 100 mm wide, but some E. nitens boards were 90-95 mm wide. Each 500-mm-long board was cut into two matched 250-mm-long boards. The 250-mm-long boards were assigned to the following treatments:

- Undensified control
- Surface densification
- Bulk densification

The optimum press force and press closing gap (target densification thickness) were determined for each species prior to starting the experiments, to ensure a high degree of densification without cracking or splitting in the wood. The quarter-sawn *E. fastigata* boards had significant issues with bulk densification, where densifying to a final thickness below 16 mm caused substantial cracking and darkening of the wood. This meant that the *E. fastigata* was densified to a greater target thickness (ie, less densification) than the *E. nitens*.

For the experiment, for each combination of species, grain orientation, and densification process, 10 replicates were used, giving a total of 120 boards for property testing.

The thermo-hydro-mechanical (THM) densification treatment was applied as surface densification and as bulk densification. Table 1 presents the parameters used in the study.

The final thickness of the densified samples was determined by placing metal bars of an appropriate

	Surface	Bulk
Top platen initial temperature (°C)	170	170
Bottom platen initial temperature (°C)	20	170
Press force (kPa)	3000	3000
Hold time at the initial temperature (minutes)	3	3
Heating rate to a high temperature (°C/min)	20	20
Top platen high temperature (°C)	200	200
Bottom platen high temperature (°C)	20	200
Top platen cooling temperature (°C)	60	60
Bottom platen cooling temperature (°C)	60	60
Target densified thickness ^a (mm)	17/16 (E. fastigata/E. nitens)	16/10 (E. fastigata/E. nitens)
Densification ratio ^b	0.15/0.2	0.2/0.5

Table 1. Parameters of the THM surface and bulk densification processes used in this study.

^a Target densified thickness is the thickness of the metal stops used to control the final press gap.

^b Densification ratio is a theoretical degree of compression based on the nominal initial thickness and the press gap.

thickness (Table 1) into the press to control the final press gap.

The press was heated to the target temperature (Table 1). Then, the samples were loaded into the press, and the densification process was implemented according to the parameters in Table 1.

Prior to densification, the weight and dimensions of each board were measured. Following densification, once the boards were cool enough to handle (usually after 10-15 min), the width and thickness of the boards were measured again. The spring-back (recovery of board thickness immediately after pressing) and the width expansion of each board can be calculated:

Spring-back =
$$\left(\frac{t_d - t_t}{t_o - t_t}\right) \times 100 \ [\%]$$
 (1)

Where:

 t_o is the initial (uncompressed) thickness of the sample,

 t_d is the thickness after densification

 t_t is the target thickness (press gap)

Width expansion is defined by Eq 2.

$$WE = \left(\frac{w_o - w_d}{w_o}\right) \times 100 \ [\%] \tag{2}$$

Where:

 W_o is the original width of the sample W_d is the width after densification

Following densification, the boards were conditioned at 20°C, 65% RH for 4 wk. The board dimensions were measured again following conditioning.

Property Testing

Set-recovery is the extent to which the densified sample resists returning to its original dimensions. This was assessed in two ways, 1) by soaking in water, and 2) by exposure to high-humidity air (RH cycling).

From each board, two $20 \times 20 \text{ mm}$ blocks were cut side by side, each at least 5 mm from the board edges and 60 mm from the board end (Fig 1). One block from each pair was assessed via water soaking and one via RH cycling.

For the water-soaking test, the thickness of the blocks was measured in three places. Then, the blocks were oven-dried overnight at 103°C, and their thickness was measured again.

In the next step, the blocks were submerged in water at 20° C for 24 h, then oven-dried at 103° C for 24 h, and their thickness was measured again. The water soaking, oven drying, and subsequent thickness measurement were repeated for a further 4 cycles.

Set-recovery after water soaking is calculated as follows:

$$SR_{WS} = \left(\frac{t_{wOD} - t_{OD}}{t_o - t_{OD}}\right) \times 100 \,[\%]$$
(3)



Figure 1. A cutting plan showing the location of the property test specimens within each board. The view is of the upper face of the board.

Where:

 t_o is the initial uncompressed thickness

 $t_{\rm OD}$ is the oven-dried thickness following densification

 t_{wOD} is the oven-dried thickness following water soaking

The RH cycling test was similar to the water soaking test, but instead of oven drying, the boards were conditioned at 25°C, 65% RH for 2 wk, and then, conditioned at 25°C, 85% RH for 2 wk, and their thickness was measured at the end of each conditioning period. After five conditioning cycles, the blocks were oven-dried, and their thickness was measured again.

Set-recovery after humidity cycling is calculated as follows:

$$SR_{RS} = \left(\frac{t_{85} - t_c}{t_o - t_c}\right) \times 100 \left[\%\right]$$
(4)

Where:

 t_o is the initial uncompressed thickness

 t_c is the thickness following densification and conditioning to 65% RH

 t_{85} is the thickness after conditioning to 85% RH.

For hardness testing, $100 \times 50 \,\mathrm{mm}$ blocks were cut across the entire width of each board, starting around 5 mm from the board end (Fig 1). Because surface densification is primarily intended to change the properties of the wood surface and not necessarily the bulk of the wood sample, it is important to use a hardness test that does not penetrate too deeply into the sample (Scharf et al 2022). Here, a modified Brinell hardness test was used where a steel ball 11.28 mm in diameter was indented 4 mm into the surface of the sample and the applied load was recorded (Fig 2). Each tested sample was placed on a second board of the same material to minimize the effect of board thickness on the measured hardness. Some quarter-sawn bulk-densified boards split before the hardness testing was complete. For these boards, the applied load and indentation depth at the time of the break were recorded.

Brinell hardness (BHN) is calculated according to Eq 5.

$$BHN = \frac{2F}{\pi D \left(D - \sqrt{D^2 - d^2} \right)} \left[kN/mm^2 \right] \quad (5)$$

Where:

F is the applied force (kN)



Figure 2. Brinell hardness testing set up, showing the sample to be tested sitting on top of a second sample of the same species, grain orientation, and densification process (ie, surface- or bulk-densified).

D is the diameter of the ball (mm)

d is the maximum diameter of the indentation (mm)

The diameter of the indentation (*d*) was calculated as follows:

$$d = \sqrt{8h(D/2 - h/2)} \,[\text{mm}]$$
 (6)

Where:

h is the depth of the indentation (mm)

And D and d are as defined in Eq 5.

Density Profiles

Density measurements were made using the Scion DiscBot measurement system (Scion 2016). This consists of a range of measurement tools connected to an X-Y table to enable automated two-dimensional measurements of discs, cores, or small sections of boards.

A 25-mm-long sample was cut from near the center of each board (avoiding the end-most 50 mm of each end of the board, as shown in Fig 1) and these were equilibrated under standard conditions (25°C, 65% RH) until their weight stabilized. Prior to testing, the weight and dimensions of each sample were recorded. These values were used to calculate a nominal gravimetric density for each sample, ie, the density of the wood plus associated moisture.

Prior to being measured in the DiscBot, each sample was fixed into a frame to ensure it was oriented correctly for the X-ray density measurement. Samples were oriented relative to their orientation in the hot press, namely, with the face compressed by the top platen facing in the same direction for every sample.

X-ray density measurements were taken using a polychromatic X-ray source (Spellman RB150 PN600X4009) with an output of 70 kV and 3 mA. The X-rays pass through the sample and are detected with a Hamamatsu Photonics model C9750-10F line camera. This measurement was repeated on a 0.4-mm grid over the entire surface of the sample. Density was calculated at the measurement pixel level from the X-ray intensity, plus an empirically derived mass attenuation coefficient (Eq 7). This was used to generate a two-dimensional map of nominal density values (converted to kg/m³) on a 0.33 mm grid over the entire sample width and thickness.

$$\rho = \left(\frac{1}{\mu_m t}\right) \cdot \left(-\ln\left(\frac{I}{I_0}\right)\right) \left[g/cm^3\right]$$
(7)

Where:

 ρ is the specimen density (g/cm³)

 μ_m is the X-ray mass attenuation coefficient (0.2946 cm²/g)

t is the specimen thickness (cm)

I is the X-ray intensity through the sample, minus the source-off signal

 I_0 is the X-ray intensity through the air, minus the source-off signal

The nominal density values were adjusted using R software (R Core Team 2021). The samples were not always perfectly oriented to the X- and Y-axes of the DiscBot, so the samples were rotated when required, and a new coordinate system was applied so the board edges were parallel

to the X- and Y-axes. Linear interpolation was used to produce a new set of density values on a 0.5-mm grid using the new coordinate system. One-dimensional density profiles were produced by averaging the density values over the entire width (X-axis) for each point on the Y-axis, ie, at 0.5 mm spacing through the thickness of the original board.

For the densified samples, the one-dimensional density profiles were further characterized according to the metrics described by Zhou et al (2019), as reproduced in Fig 3. Briefly, the PD is the maximum density in the profile, and the peak density depth (PD_i) is the distance from the surface to the PD. For the surface densified samples, the thickness of the densified zone (DTh) was also quantified. This is defined as the thickness over which the density is greater than 80% of the PD.

For each combination of species, grain orientation, and densification process, an average (onedimensional) density profile was produced by aligning all the samples with the equivalent of the top platen face and averaging the density values at each point through the wood thickness. Because the wood thickness does vary slightly between specimens, the thickness of each sample



Figure 3. Metrics calculated to characterize the onedimensional density profiles.

was normalized to the corresponding press gap (compressed thickness) for each species and densification process. This ensured consistent alignment of both the top and bottom surfaces across all the samples. For each densified sample, there is an equivalent undensified sample which was cut from the same board. The average density of each of these undensified samples was used to normalize the density of their equivalent densified boards, ie, each individual density value was divided by the average density of the equivalent undensified control sample.

Statistical Analysis

Because of the differences in target thickness, ie, the press gap between the two species, it was decided to analyze each species separately. Within each species, the data were initially assessed for normality and homogenous variances using Shapiro-Wilk and Levene's tests. Hardness, final density, and final MC were normally distributed. So a two-way ANOVA analysis was used to compare the means of the different densification processes and sawing orientations. Linear modeling was used to determine the significance of relationships between variables. All other variables were found to have significant variation from normality and homoscedasticity. Therefore, nonparametric test methods were used to assess the data. The Kruskal–Wallis test with Holm adjustment was used to determine significant differences between the densification processes and with different grain orientations.

RESULTS AND DISCUSSION

A summary of the board properties following THM densification is shown in Table 2. For both densification processes and for both grain orientations, the final thickness of the *E. fastigata* boards was, on average, significantly thicker than the press gap. The surface densified boards had a high percentage of spring-back (\sim 9-18% on average) which would contribute to the difference between the press gap, and the final board thickness. For *E. nitens*, the average final board thickness was not significantly different to the press

Species	Grain orientation	Densification type	Spring-back (%)	Width expansion (mm)	Final thickness (mm)
E. fastigata	Flat-sawn	Surface	17.7 ^a	1.2^{ab}	17.91 ^g
E. fastigata	Quarter-sawn	Surface	9.1 ^{ab}	1.0^{b}	17.42 ^g
E. fastigata	Flat-sawn	Bulk	3.5 ^b	1.9 ^a	16.34 ^g
E. fastigata	Quarter-sawn	Bulk	6.4 ^{ab}	1.5^{ab}	16.36 ^g
E. nitens	Flat-sawn	Surface	1.5 ^e	$1.6^{\rm e}$	16.03
E. nitens	Quarter-sawn	Surface	2.9 ^e	1.4 ^e	16.13
E. nitens	Flat-sawn	Bulk	2.3 ^e	4.9^{f}	9.93
E. nitens	Quarter-sawn	Bulk	10.8 ^e	3.6 ^f	10.33

Table 2. Spring-back, width expansion, and final thickness for each species and sawing orientation. Prior to densification, the samples had an average width of 97 mm and an average thickness of 21 mm.

^{a-f} Values followed by the same letters in superscript do not differ significantly from one another at alpha = 0.05.

^g Final thickness is significantly different to press gap, ie, target final thickness.

gap for any of the grain orientations or densification processes. Spring-back can occur when elastic deformation in the wood cells is not adequately relieved before the press is opened. Low levels of spring-back are preferred, as this reduces thickness variation in the densified boards, and means that the energy put into compressing the boards is not lost when the press force is released. For E. fastigata, there were no substantial differences in width expansion between the different densification processes or sawing orientations. For E. nitens, the width expansion was significantly higher in the bulk densified boards compared with the surface densified, but there was no difference between sawing orientations. Lower width expansion is preferable, to avoid width variation in the densified boards.

Set-Recovery by Water Soaking

Set-recovery is a measure of how much the densified wood resists returning to its original undensified dimensions when the wood MC increases. A set-recovery of 0% means the board retains its densified dimensions, and a set-recovery of 100% means the board has reverted to its original undensified dimensions. The set-recovery resulting from repeated water soaking is shown in Fig 4. For both species, the surface-densified boards had a high set-recovery (average 75-85%), indicating the boards had regained over threequarters of the reduction in thickness from densification. For *E. fastigata*, the bulk densified boards did not have a significantly different set-recovery to the flat-sawn surface densified boards. For E. nitens, the bulk densified boards had significantly lower set-recovery, and there was no significant difference in set-recovery between the two grain orientations. The setrecovery of bulk densified E. nitens (average 40%) is somewhat higher than that found by Balasso et al (2020), who measured a setrecovery of 27.5% in densified E. nitens after a single water soaking cycle. However, the set-recovery values in this study are still much lower than those seen in other species. For example, Laine et al (2013) prepared bulk densified Scots pine which showed a set-recovery of 75% after 3 cycles of water soaking. In another attempt, Darwis et al (2017) bulk densified Gmelina arborea to various ratios (densification ratios from 0.125 to 0.375) and found that the set-recovery increased proportional to the densification ratio (from ~ 60 to 80% set-recovery). The bulk-densified E. nitens had a higher densification ratio again (0.5), but much lower setrecovery. Despite the unusually low set-recovery for the bulk densified E. nitens, a 30-40% increase in thickness on contact with water is unlikely to be acceptable in service, since set-recovery would need to be eliminated for a commercially viable product. As shown by Darwis et al (2017), thermal modification can reduce the set-recovery. In their work, heat treatment at 180°C for 5 h reduced the set-recovery by more than half. Thermal modification could be considered here as a way to reduce the set-recovery of either bulk



Figure 4. Set-recovery by densification type, species, and grain orientation. Superscript letters indicate treatments within each species that are not significantly different from each other (95% confidence level, Kruskal-Wallis test with Holm adjustment).

densified or surface densified wood, potentially to an acceptably low level.

Set-Recovery by RH Cycling

As noted by Laine et al (2013), the water soaking test is a harsh test, especially for products such as flooring that are used indoors, which would generally be coated before use and are unlikely to become water saturated. As an alternative method of assessing set-recovery, the set-recovery after five cycles of alternating high and low RH was assessed. The obtained results are shown in Fig 5. The values of set-recovery were much lower than those obtained for the water soaking test but show similar trends. For E. nitens, the set-recovery was lower for the bulk densification compared with that of the surface densification, and there were no significant differences between the different grain orientations. The trend for E. fastigata was similar, with higher set-recovery for the surface densification compared with the bulk densification, but with significant differences between the two grain orientations for each densification process. These results also showed that the RH cycling set-recovery of densified *E. nitens* is less than that of *E. fastigata* for all tested samples (Fig 5). The causes of this difference in set recovery between species are not known.

The EMC at 25°C, 65% RH following five humidity cycles is shown in Table 3. For both species, the EMC (EMC) reduced significantly with increasing degree of densification. Lower EMC can correspond to increased dimensional stability (Navi and Sandberg 2012c), implying that a significant reduction in EMC is a positive result. These results also show that grain orientation did not have a significant effect on EMC.

Density Profiles

The average PD, and PD_i are shown in Table 4. For the surface densified boards, the DTh is also shown. For the *E. fastigata* boards, there is no significant difference in PD between the different densification processes and no significant differences in the PD_i values obtained for different samples. However, for *E. nitens*, the bulk densified boards have a higher PD than those of the



Figure 5. RH-cycling set-recovery by densification type, species, and grain orientation. Superscript letters indicate treatments within each species that are not significantly different from each other (95% confidence level, Kruskal-Wallis test with Holm adjustment).

surface densified boards, and the quarter-sawn surface densified boards have a higher PD than those of their flat-sawn equivalents. For the flatsawn surface densified boards, the PD is significantly deeper into the board compared with the equivalent quarter-sawn boards and compared with the bulk densified boards (both sawing orientations), as indicated by measured PD_i values (Table 4). These results also revealed that, the DTh value of flat-sawn boards is larger than that of quarter-sawn boards when a surface densification is implemented for *E. fastigata*. The difference between the two grain orientations was not significant for *E. nitens* (Table 4).

Table 3. Nominal density following densification and conditioning, hardness and EMC (EMC) for each densification process, and undensified controls.

Species	Grain orientation	Densification type	Nominal density (kg/m ³)	Brinell hardness (kN/mm ²)	EMC (%)
E. fastigata	Flat-sawn	Control	712 ^{cd}	26.6 ^{bc}	12.8 ^{ab}
E. fastigata	Quarter-sawn	Control	680^{d}	20.7°	13.2 ^a
E. fastigata	Flat-sawn	Surface	806 ^{ab}	37.5 ^a	12.4 ^{bc}
E. fastigata	Quarter-sawn	Surface	734 ^{bc}	31.1 ^{ab}	12.1 ^c
E. fastigata	Flat-sawn	Bulk	861 ^a	37.3 ^a	11.5 ^d
E. fastigata	Quarter-sawn	Bulk	779 ^{abc}	28.3 ^{abc}	10.9 ^d
E. nitens	Flat-sawn	Control	490 ^e	15.0 ^e	12.4 ^{ef}
E. nitens	Quarter-sawn	Control	510 ^e	15.7 ^e	12.6 ^e
E. nitens	Flat-sawn	Surface	$582^{\rm f}$	22.2^{f}	11.6 ^{fg}
E. nitens	Quarter-sawn	Surface	642^{f}	31.4^{fg}	11.5 ^g
E. nitens	Flat-sawn	Bulk	916 ^g	31.7 ^g	10.3 ^h
E. nitens	Quarter-sawn	Bulk	938 ^g	34.7 ^g	10.6 ^h

^{a-h} Superscript letters indicate treatment groups (within each species) that are not significantly different from each other (95% confidence level, Tukey's HSD test).

Species	Densification process	Grain orientation	PD (kg/m ³)	PD _i (mm)	DTh (mm)
E. fastigata	Surface	Flat-sawn	886 ^a	2.1 ^a	4.0 ^a
E. fastigata	Surface	Quarter-sawn	899 ^a	0.8^{a}	2.0 ^b
E. fastigata	Bulk	Flat-sawn	898 ^a	1.2^{a}	_
E. fastigata	Bulk	Quarter-sawn	844 ^a	0.5^{a}	_
E. nitens	Surface	Flat-sawn	770 ^g	1.4 ^e	2.4 ^e
E. nitens	Surface	Quarter-sawn	$870^{\rm f}$	0.3^{f}	1.7 ^e
E. nitens	Bulk	Flat-sawn	935 ^e	0.4^{f}	_
E. nitens	Bulk	Quarter-sawn	972 ^e	0.2^{f}	_

Table 4. Peak density (PD), Peak density depth (PD_i), and thickness of the densified region (DTH) for each species, densification process, and orientation.

 a^{-f} Values followed by the same letters in superscript do not differ significantly from one another at alpha = 0.05.

The average one-dimensional density profile for each species, densification process, and grain orientation are shown in Fig 6. For the *E. fastigata* boards, the overall increase in density is small, with peak densities being around 1.3 times higher than the density prior to densification (Fig 6[a]). The density at the densified surface of the surface densified boards is similar to that of the outer surfaces of the bulk densified boards, while the undensified surface has a similar density to the undensified boards. This is a good result, as the surface densification process aims to produce a hard densified surface on one face of the board, without unduly compressing the remainder of the board thickness. The flat-sawn bulk densified boards showed a flatter density profile across the board thickness compared with the quartersawn boards, which have a central area with a density similar to the density prior to densification (Fig 6[a]).

The *E. nitens* boards presented a considerably larger increase in density compared with the undensified controls. (Fig 6[b]), due to the higher densification ratio used on this species. The bulk densified samples showed similar density profiles to the bulk densified *E. fastigata*, but with the average density being around 1.8-1.9 times higher than the density prior to densification. The surfacedensified *E. nitens* has a slightly lower PD than the bulk-densified *E. nitens*. The quarter-sawn



Figure 6. Average density profiles for *E. Fastigata* (a) and *E. Nitens* (b). The normalized density is based on the pretest density of each board. The normalized thickness is based on the target thickness for each densification process.

surface-densified *E. nitens* has a density peak at the board surface, which provides the largest increase in surface density for a given densification ratio, which is the primary aim of the surface densification process. In contrast, the flat-sawn boards had a PD that was slightly below the wood surface (Fig 6[b]). As with *E. fastigata*, the undensified face of the surfacedensified *E. nitens* had a similar density to the undensified controls.

Hardness and Bulk Density

The gravimetric (nominal) density of the densified samples and undensified controls are shown in Table 3. It should be noted that this is the density of the wood, plus associated moisture under standard conditions (25°C, 65% RH). Because the EMC of the wood is lower following the densification process (Table 3), this will alter the relationship between the nominal density and the oven-dry density for the densified samples. It is likely that the nominal density values reported here under-report the increase in oven-dry density of the bulk densified boards by a small amount (1-3%). For E. nitens, surface densification increased the density significantly compared with the undensified controls, and the bulk densification increased it further. No significant differences in final density were seen between sawing orientations. For E. fastigata, the differences in density were smaller, and there were not such clear-cut differences between the two densification processes. Additionally, for the bulk densified samples, the flat-sawn boards had a significantly higher density than the quarter-sawn boards. Balasso et al (2020) densified E. nitens to a target densification ratio of 0.39, which is somewhat less compression than the bulk densification in this study, however, their average final density $(800 \pm 9 \text{ kg/m}^3)$ was similar to the bulk densification in this study $(837 \pm 44 \text{ kg/m}^3)$. The undensified E. fastigata showed a higher density than the undensified E. nitens, which gives some explanation as to why the E. nitens boards could be densified to a greater degree without sustaining damage. For each species, the bulk densified boards were densified to the greatest extent possible

without damaging the wood, and for *E. nitens*, this resulted in a substantially higher final density than the *E. fastigata* boards, despite starting from a lower initial density. All things being equal, you would expect both species to reach a similar density before damage to the wood occurred. This suggests there are additional, unknown factors that make *E. nitens* more suitable for densification than *E. fastigata*, eg, wood structure or chemistry.

For the *E. nitens* boards, both surface and bulk densification significantly increased the wood hardness compared with the controls, and there were no significant differences in hardness between the two grain orientations. For *E. fastigata*, the flat-sawn surface- and bulk-densified samples had significantly higher hardness values than the undensified controls, but these differences were not significant for the quarter-sawn samples. For both species and for both densification processes, there were no significant differences in hardness between samples with different grain orientations.



Figure 7. Relationship between final density and Brinell hardness for each species and densification type. *E. Fastigata* is shown by triangles, *E. Nitens* by circles. The best fit lines show a correlation between density and hardness, taking the densification process into account. Adjusted $R^2 = 0.586$, p < 0.001.

Wood properties such as hardness are generally correlated with density. Linear regression was used to investigate the relationship between average cross-sectional density and Brinell hardness, after taking species, densification process, and grain orientation into account. The effects of species and grain orientation were not significant (p = 0.73 and p = 0.64, respectively). The relationship between hardness and density was significant (p < 0.001), and for a given density, surface-modified boards had on average, a Brinell hardness of 5.6 kN/m^2 , higher than both the undensified boards and the bulk-densified boards. Individual board values and fitted lines for each densification treatment are shown in Fig 7.

CONCLUSIONS

E. nitens was able to be densified to a greater degree without sustaining damage than was observed for *E. fastigata* (maximum densification ratios of 0.5 and 0.2, respectively). This resulted in larger increases in density and surface hardness in the *E. nitens* boards (100-120% increase in surface hardness for *E. nitens* compared with 40-50% in *E. fastigata*).

The set-recovery following water soaking was surprisingly low in the bulk densified *E. nitens* boards (average 40%), whereas the *E. fastigata* and the surface densified *E. nitens* had higher set-recoveries (average 55-85%) which is more in line with values from other species seen in the literature.

Despite the bulk densified *E. nitens* having an unusually low set-recovery, this level of irreversible swelling is still unlikely to be acceptable in service, so an additional treatment, such as thermal modification, would be required to reduce the set-recovery further. Set-recovery following humidity cycling was lower than that following water soaking (averages from 7 to 32%), but it followed a similar trend with bulk densified *E. nitens* showing the lowest set-recovery in this study. It would be worth investigating ways of further reducing the set-recovery in *E. nitens* to see if it can be eliminated. Density profiles showed that the PD for each sample was generally within 1-2 mm of the wood surface and was often right on the surface. Having a density peak close to the wood surface is beneficial because it results in the maximum increase in surface hardness for a given densification ratio, but allowances must be made for some of the surface material to be removed during final finishing, eg, sanding.

The relationship between surface hardness and average board density was independent of species and grain orientation for the tested samples. For a given density, surface densified boards had a higher surface hardness than undensified and bulk densified boards, which were not significantly different to each other.

In contrast with many softwood species, eucalypts are typically quarter-sawn, both for appearance and to reduce the incidence of drying degrade. Overall, there were minimal differences in performance between quarter-sawn and flat-sawn boards following densification in this study. In some cases, set-recovery and depth of PD, quartersawn boards performed slightly better than flatsawn boards. This is a positive result for eucalypts, as the densification process could be incorporated with existing sawing and processing methods that produce quarter-sawn timber, and consequently, new applications of their use could be applied.

ACKNOWLEDGMENTS

The authors are grateful for the assistance of many people to complete this study. At InnoRenew CoE Josip Dijanić assisted with sample preparation, Václav Sebera assisted with hardness testing, and Črtomir Tavzes assisted with determining phytosanitary requirements for returning the samples to New Zealand. Lea Primožič assisted RS with travel arrangements to Slovenia to complete the experimental work. At Scion, Maxine Smith and Jamie Agnew assisted with sample preparation and RH set-recovery testing. John Lee and Mark Riddell x-rayed samples in the DiscBot and analyzed the density data.

This work was funded by the New Zealand Ministry for Business, Innovation, and Employment Strategic Science Investment Fund under Scion's Manufactured Products from Trees Science Platform (C04X1703), as well as the European Commission for funding the InnoRenew project (Grant Agreement #739574) under the Horizon 2020 Widespread-Teaming program and the Republic of Slovenia (investment funding of the Republic of Slovenia and the European Regional Development Fund), and the Slovenian Research Agency ARRS for funding the infrastructural program IO-0035.

REFERENCES

- Balasso M, Kutnar A, Niemelä EP, Mikuljan M, Nolan G, Kotlarewski N, Hunt M, Jacobs A, O'Reilly-Wapstra J (2020) Wood properties characterisation of thermohydro mechanical treated plantation and native Tasmanian timber species. Forests 11:1-15.
- Darwis A, Wahyudi I, Dwianto W, Cahyono TD (2017) Densified wood anatomical structure and the effect of heat treatment on the recovery of set. J Ind Acad Wood Sci 14:24-31.
- Fu Q, Cloutier A, Laghdir A (2017) Effects of heat and steam on the mechanical properties and dimensional stability of thermo-hygromechanically-densified sugar maple wood. BioResour 12:9212-9226.
- Jones TG, McConnochie RM, Shelbourne T, Low CB (2010) Sawing and grade recovery of 25-year-old *Eucalyptus fastigata, E. globoidea, E. muelleriana and E. pilularis.* N Z J For Sci 40:19-31.
- Koumba G, Chavez R, Bustos C, Cloutier A, Stevanovic T (2014) Chemical changes induced in *Pinus radiata* and *Eucalyptus nitens* following the densification process. J Sci Technol For Prod Process 4:20-22.
- Kutnar A, Kamke FA (2012) Influence of temperature and steam environment on set recovery of compressive deformation of wood. Wood Sci Technol 46:953-964.
- Kutnar A, Sandberg D, Haller P (2015) Compressed and moulded wood from processing to products: COST action FP0904 2010-2014: Thermo-hydro-mechanical wood behaviour and processing. Holzforschung 69: 885-897.

- Kutnar A, Sernek M (2007) Densification of wood. Zbornik gozdarstva in lesarstva 82:53-62.
- Laine K, Belt T, Rautkari L, Ramsay J, Hill CAS, Hughes M (2013) Measuring the thickness swelling and setrecovery of densified and thermally modified Scots pine solid wood. J Mater Sci 48:8530-8538.
- Laine K, Segerholm K, Wålinder M, Rautkari L, Hughes M (2016) Wood densification and thermal modification: Hardness, set-recovery and micromorphology. Wood Sci Technol 50:883-894.
- Lenth CA, Kamke FA (2001a) Moisture dependent softening behavior of wood. Wood Fiber Sci 33:492-507.
- Lenth CA, Kamke FA (2001b) Equilibrium moisture content of wood in high-temperature pressurized environments. Wood Fiber Sci 33:104-118.
- Navi P, Sandberg D (2012a) Wood densification and fixation of the compression-set by THM treatment. Pages 193-224 *in* Thermo-hydro-mechanical processing of wood. EPFL Press, Lausanne, Switzerland.
- Navi P, Sandberg D (2012b) Surface densification. Pages 241-248 *in* Thermo-hydro-mechanical processing of wood. EPFL Press, Lausanne, Switzerland.
- Navi P, Sandberg D (2012c) Heat treatment. Pages 249-286 *in* Thermo-hydro-mechanical processing of wood. EPFL Press, Lausanne, Switzerland.
- R Core Team (2021) R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria.
- Rautkari L, Laine K, Laflin N, Hughes M (2011) Surface modification of Scots pine: The effect of process parameters on the through thickness density profile. J Mater Sci 46:4780-4786.
- Scharf A, Neyses B, Sandberg D (2022) Hardness of surface-densified wood. Part 1: Material or product property? Holzforschung 76:503-514.
- Scion (2016) DiscBot—automated wood properties scanning. https://www.youtube.com/watch?v=KVv8_GVXQbU (24 April 2023).
- Wang JY, Cooper PA (2005) Effect of grain orientation and surface wetting on vertical density profiles of thermally compressed fir and spruce. Holz Roh Werkst 63: 397-402.
- Zhou Q, Chen C, Tu D, Zhu Z, Li K (2019) Surface densification of poplar solid wood: Effects of the process parameters on the density profile and hardness. BioResour 14: 4814-4831.