

Effect of supercritical CO₂ treatment and kiln drying on collapse in *Eucalyptus nitens* wood

Bernard S. W. Dawson¹ · Hamish Pearson¹ · Mark O. Kimberley¹ · Bruce Davy¹ · Alan R. Dickson¹

Received: 30 April 2019 / Published online: 30 January 2020 © The Author(s) 2020

Abstract

Collapse-prone timbers such as species of *Eucalyptus* are poorly utilised due to low conversion rates that necessitate long pre-drying times. A supercritical CO_2 lumen water expulsion pre-treatment prior to kiln drying is proposed to bypass lengthy pre-drying. After drying (air, kiln or oven drying), shrinkage, collapse, washboard depression and checking of *Eucalyptus nitens* were determined using image analysis of 0.8 mm thick wafers and 5 mm thick biscuits. Lumen water expulsion-kiln drying reduced collapse by 75% and washboard depression by 71%, compared to drying from green. As water is removed from the water conductive tissue (vessels, rays, and fibre-tracheids) by lumen water expulsion, the water column is broken throughout the specimen, thereby disrupting the development of meniscus-induced water tension as subsequent drying occurs. Remaining water is proposed to reside in the non-water-conductive fibre tissue. If the process can be applied on large scale to *Eucalyptus nitens*, there is the opportunity for higher conversion rates to increase the commercial viability of solid wood products.

1 Introduction

The moisture content in living trees can exceed 200% (*m/m*) with large in-tree variation (Simpson and TenWolde 1999). Removing the bulk of this water (sap) after felling is a key step in wood processing. The drying of hardwoods can involve air- or pre-drying prior to kiln drying (Denig et al. 2000).

Yang and Liu (2018) reviewed the history of collapse in the genus *Eucalyptus*. They considered the morphological characteristics of collapse and gave an overview of the liquid tension theory of collapse, which involves a differential pressure across the menisci of saturated cell lumens (a lumen is the inside space of a cell). They listed treatments designed to reduce collapse as well as variants of drying processes. Drying of collapse-prone *Eucalyptus* species can result in low conversion into product using existing kiln drying methods. Collapse is thought to be the result of negative water tension that develops in boards during the early stages of drying while still at very high moisture contents (Kauman 1964; Chafe et al. 1992). The mechanics and thermodynamics

Bernard S. W. Dawson bernard.dawson@scionresearch.com described by the Laplace and Kelvin equations respectively are the basis of the water tension theory, which remains the best explanation for collapse. This theory of collapse requires (1) saturated lumens; (2) negative water tension arising from surface tension developed at menisci interfacing air or water-vapour filled cells bordering water-filled lumens and (3) average cell wall strength in compression, perpendicular to the cell walls, that is less than the negative water tension.

Drying methods for refractory Eucalyptus species often require a lengthy pre-drying phase to lessen the prevalence of collapse, washboard depression and internal checking. High pressure supercritical CO₂ treatment is a faster water removal technique from green timber that also reduces collapse (Dawson et al. 2015; Franich et al. 2010, 2013) in a variety of softwoods and hardwoods (Dawson and Pearson 2017; Dawson et al. 2015). The water removal efficiency of the supercritical CO₂ treatment varies according to species (Dawson and Pearson 2017). To better understand the process, a computational fluid dynamics model has been developed to predict the behaviour of the supercritical CO₂ (Pearson et al. 2019a, b). This model of the supercritical CO₂ treatment process is consistent with a lumen water expulsion (LWE) process in which CO₂ is cycled between atmospheric pressure and 20 MPa and results in the expulsion of free lumen water only. Following lumen water expulsion, kiln

¹ Scion, Te Papa Tipu Innovation Park, 49 Sala Street, Rotorua 3046, New Zealand

drying is required to reduce moisture contents down to service levels.

The hypothesis to be tested is that the lumen water expulsion pre-treatment will prevent the development of negative water tension and in doing so reduce cell collapse, washboard depression and internal checking on drying to service moisture contents.

2 Materials and methods

Log sections, 1200 mm long, were cut 2.5 m from the base of ten 14 years old *Eucalyptus nitens* (H. Deane and Maiden) logs from a Southwood Exports Ltd forest (46.5° S, 168.4° E), end-sealed with wax and wrapped in plastic for refrigerated transport to Scion. Within 10 days of felling, each log section was reduced to eight 37 mm \times 37 mm \times 600 mm boards (Fig. 1). Each board was cut into two 200 mm specimens for treatment and one 150 mm specimen for determination of physical properties. Boards were double-plasticbagged and refrigerated at 4 °C until treated.

Redman et al. (2016) found that wafers less than 1 mm in thickness were necessary to avoid collapse in Eucalyptus obliqua. Pure shrinkage and pure collapse were therefore determined using 0.8 mm thick cross-section wafers and 5 mm cross-section biscuits, after respective drying schedules and conditioning to 12% moisture content. A 0.8 mm cross-section wafer for image processing was carefully cut from an end of each green 150 mm specimen as soon as it was produced (Fig. 2). In the setup, the base plate B prevents the wafer from being lost, the pusher block P moves the wafer across the saw blade and the wafer deflector moves the wafer safely away from the saw blade. Micro-adjustment of the fence on the table holding the specimen afforded control of wafer thickness, which was determined using callipers. Once the minimum thickness at which the integrity of the wafer was determined, the table position was locked and wafers were cut. An AKE saw blade, model # 27 303 30 with 60 teeth, for precision cutting of fine veneers, acrylics, melamines and laminates without fibre pull-out, was used for cutting the wafers.

Cut wafers were placed on a filter paper between two glass slides (75×50 mm), held together loosely by rubber bands, and kept under water until photographed.

Eucalyptus nitens was chosen as a collapse-prone species known to have a medium lumen water expulsion efficiency (Dawson and Pearson 2017).

The two pre-treatments used were either the green state or after lumen water expulsion using a high pressure supercritical CO_2 treatment. The specimens were then air dried, kiln dried or oven dried. The specimens from each tree were randomised before being assigned to one of six treatment



Fig. 1 Sampling and drying regime of *Eucalyptus nitens* heartwood to produce 0.8 mm green wafers and 5 mm 12% MC biscuits for image processing

sets (Table 1). Each treatment set had one specimen from each of the ten trees.

The supercritical CO_2 treatment of wooden specimens involves raising the CO_2 pressure around the specimens to 20 MPa and holding for an incubation period so that the CO_2 can diffuse into the specimen sap (water), while maintaining the treatment chamber at 50 °C. Depressurisation down to atmospheric pressure then results in conversion from supercritical fluid to gas phase CO_2 inside the specimen, which forces water out of specimen wood cells (Dawson et al. 2015; Dawson and Pearson 2017). The pressurisationdepressurisation cycle is repeated multiple times to continue water expulsion from the specimens. The criteria for determining the number of cycles are dependent on the species being treated; for *Eucalyptus nitens*, ten cycles were chosen



Fig.2 Set-up for cutting 0.8 mm green *Eucalyptus nitens* wafers. **a** Specimen S before the saw blade; **b** Specimen still attached to board, being pushed by block edge P. Base plate B moves with the table towards saw blade preventing loss of the sample. Wafer deflector D

Table 1 Trial factorial design (n=10 for each treatment) for treatment of *Eucalyptus nitens* specimens

Pre-treatment	Drying treatment	Full treatment	Temperature (dry bulb; °C)	Relative humidity (%)
Green	Air dry	Green-Air	25	65
Green	Kiln dry	Green-Kiln	70	80
Green	Oven dry	Green-Oven	103	0
LWE	Air dry	LWE-Air	25	65
LWE	Kiln dry	LWE-Kiln	70	80
LWE	Oven dry	LWE-Oven	103	0

LWE lumen water expulsion

based on scoping run data and practicality in terms of treatment time.

The automated high pressure supercritical CO₂ treatment plant used was capable of supercritical CO₂ extraction/cosolvent extraction. All sensors and actuated valves were connected to a programmable logic controller (PLC) controlled via a custom user interface (RSLogix, Rockwell Automation). The plant consists of two 1 L cylindrical pressure vessels (75 mm internal diameter; 230 mm internal length) operating from 9 MPa vacuum to 33 MPa and with a temperature span of -10 to 120 °C. Internal vessel temperatures were maintained by an electric heating jacket controlled via the PLC to maintain the pressure vessels at 50 °C. Vessels were pressurised and depressurised sequentially. One vessel was in the pressurisation phase of the lumen water expulsion treatment cycle while the other vessel was in depressurisation. A Haskel[®] air-operated pressure pump (haskel.com; model DSF-B35; 22 MPa pressure rating) delivered CO₂ (food grade, >99.8%; www.boc.co.nz) at supercritical pressure. Internal plant pressures were continuously logged by Gems Sensors pressure transducers (P_{max} 30 MPa; https://

directs wafer across the saw blade; **c**, **d** Wafer deflector safely moves wafer away from saw blade. For the photography set-up, specimen S in images **b–d** is 1.2 mm thick and is at 12% MC

Table 2 Process parameter values for lumen water expulsion treatment

Value
20
0.1
3
3
5
5
10
50

uk.rs-online.com/web/). The vessels were depressurised via an actuated needle valve which was controlled to maintain the depressurisation at a set rate. The CO_2 was returned to a storage vessel using an air-operated gas booster (Haskell AG-15). Two specimens were treated simultaneously, one in each treatment vessel (Table 2).

Air dried specimens were stored at 25 °C/65% relative humidity, which is equivalent to 12% equilibrium moisture content (EMC). The kiln drying schedule used a 0.36 m³ research kiln controlled by proportional-integral-derivative control (PID) loops and set points. The continuous mild kiln schedule had dry and wet bulb temperature set points of 70 and 65.2 °C respectively giving 80% relative humidity. The reduction in specimen moisture content in kiln drying was monitored manually, with specimens removed from the kiln, weighed and returned until the moisture content was in the 14–20% range (referenced against matched off-cuts which had been oven dried). Oven-dried specimens were held at 103 °C until constant mass was reached. After the three drying treatments and then equilibration in a 25 °C/65% relative humidity climate (to achieve 12% MC in specimens), a 5 mm cross-section biscuit was cut from the centre of each 200 mm specimen (Fig. 1).

2.1 Determination of shrinkage, collapse, washboard depression and internal checking

All imaging to determine surface features and external edges was carried out using incident (top) illumination via a ring light through which a digital single lens reflex camera was fitted, while imaging for internal checking used back illumination (light box). Collapsed/checked regions of the 5 mm thick biscuits were defined as those regions where light was transmitted through the 5 mm thickness of the sample. For spatial calibration, an opaque black dot of known size was included in each image.

All image processing and analysis was performed using ImageJ 1.51e (Abramoff et al. 2004). Images were binarised by the automatic thresholding of grey scale images using the 'default' method. The parameters measured by image analysis are shown in Fig. 3.

Pure shrinkage (S) was determined from wafers only, while pure collapse (C) was determined using both wafer and 5 mm biscuit measurements. Both S and C were quantified by pixel count areas (Eqs. 1, 2):

$$S = 100 \frac{A_{gw} - A_{w}}{A_{gw}},$$
 (1)

where A_{gw} is the area of the green wafer and A_w is the area of the wafer at 12% MC.

$$C = 100 \frac{A_{gw} - A_{bisc}}{A_{gw}},\tag{2}$$

where A_{bisc} is the area of the 5 mm biscuit less any cracked area due to collapse, measured at 12% MC.

The smallest best-fit rectangle (Fig. 3) was used to define the radial and tangential lengths of the wafer and the biscuit. The ratios of these were calculated both before and after treatment. Washboard depression was defined as the sum of the two maximum radial depressions from the convex hull (Fig. 3). This is an alternate measure of collapse and equates to material that needs to be removed to give two parallel straight faces on the widest dimension (Standards Australia/ Standards New Zealand 4787:2001-Timber-Assessment of Drying Quality).

The efficiency of lumen water expulsion, LWE_{eff} , of wood (Eq. 3) is defined as:

$$LWE_{eff}(\%) = 100 \frac{MC_g - MC_{LWE}}{MC_g - FSP},$$
(3)



Fig. 3 Parameters measured by image analysis. Left: photograph of a green *Eucalyptus nitens* specimen which had been oven dried prior to equilibration to 12% MC. Scale bar 10 mm. Right: the binarised image of the photograph. The convex hull (dotted black line) encloses the outer bounds of the sample area and is equivalent to a rubber band being wrapped around the area. The solid black line is the smallest best fit rectangle. The white dotted lines show the measurement of washboard depression

where MC_g and MC_{LWE} are moisture contents of green and lumen water expulsion treated specimens, respectively. The fibre saturation point (FSP) is 30% for species of similar density to *Eucalypts* (Redman et al. 2016; Budgen 1981; Haslett 1990).

Green moisture content (MC_g), basic density, maximum moisture content (MC_{max}) and % saturation were calculated following Kininmonth (1991). Basic wood density calculations used oven dry mass (103 °C until constant mass) and volume displacement.

2.2 Statistical analyses

Analysis of variance (ANOVA) was used to test the statistical significance of the pre-treatment, drying treatment and their interaction. Pairwise comparisons between treatment means and interaction means were performed using Tukey's test with α =0.05. These analyses were carried out using the SAS Version 9.4 GLM procedure. The response variables analysed were pure shrinkage, pure collapse, washboard depression, the number and area of internal checks and the radial:tangential shrinkage ratio. All variables were tested for normality using the SAS UNIVARIATE procedure prior to analysis and as a result, washboard depression and the number of internal checks were log-transformed, and the area of internal checks was square root transformed.

3 Results and discussion

The wood and moisture properties of the *Eucalyptus nitens* specimens are shown in Table 3. The mean basic density of 399 kg m^{-3} found for heartwood fits the low-density class

reported by Lausberg et al. (1995) for *Eucalyptus nitens*. Lower density *Eucalyptus nitens* heartwood is expected to show greater collapse on drying (Chafe et al. 1992).

3.1 Effect of supercritical CO₂ lumen water expulsion

The mean lumen water expulsion efficiency of the *Eucalyptus nitens* specimens was 44% (Table 3). The mean moisture content loss on the lumen water expulsion pre-treatment of *Eucalyptus nitens* was 54% ($MC_g - MC_{LWE}$). Previous work has shown that lumen water expulsion removes the majority of the cell lumen water in *Pinus radiata* (Dawson and Pearson 2017) resulting in moisture contents (MC_{LWE}) close to the fibre saturation point. For *Eucalyptus nitens*, the MC_{LWE} was well above the fibre saturation point indicating that considerable water (68%; MC_{LWE} – FSP assuming a 30% MC for the FSP) was not bound within the cell wall but could not be removed by the treatment used. By definition, water above the saturation point is not part of the cell wall and is unbound (Berry and Roderick 2005).

Difficult-to-dry or collapse-prone species are challenging to remove water from as there are impediments to permeability in their green saturated timber. For example, the durable softwood *Thuja plicata* collapses strongly due to high extractives content and high saturation levels (Cown and Bigwood 1979), some Eucalypt heartwoods suffer from strong water tension forces which cause them to collapse (Chafe et al. 1992) while the permeability of the heartwood of the hardwood Nothofagus fusca is decreased by extractives occluding pits (Kininmonth 1972). Applying the lumen water expulsion process to the heartwood of hardwoods can in some species (e.g. collapse-prone eucalypts) bypass these issues and reduce the moisture content from green to a level still well above the fibre saturation point. This can lessen pre-drying times, while reducing collapse of timber on subsequent drying down to 10-14% MC (Dawson and Pearson 2017; Dawson et al. 2015). For Thuja plicata and Nothofagus fusca, however, lumen water expulsion efficiency was very low and collapse was found to be high for Thuja plicata and moderate for Nothofagus fusca (Dawson and Pearson 2017).

Wood permeability and supercritical CO_2 diffusivity into lumen water both influence lumen water expulsion efficiency (Pearson et al. 2019a, b) and both factors are themselves influenced by wood anatomy (Wilkes 1988; Ziemińska et al. 2013). Ziemińska et al. (2013) reported the anatomical components of 24 Australian angiosperms in which they described three major wood cell tissue types: (1) vessels for transport water; (2) fibres for mechanical strength; and 3) parenchyma for nutrient storage and transport. Wilkes (1988) provided a range of percent volumes of cell types within the species Eucalyptus. The proportions of the various cell types were fibre (>60%); vessels (10-20%); axial parenchyma cells (<10\%); ray parenchyma cells (10-20%) and tracheids (2%). The combined parenchyma, vessel and tracheid tissue is about 40% if midpoint values of ranges are selected. Using comparable cell volume data for small branches of three Eucalypts representing variable situations (Ziemińska et al. 2013), the combined tissue volume was 42%.

It is proposed that this combined tissue volume, which represents the water conductive system in the living tree, is the source of the water removed by lumen water expulsion. *Eucalyptus nitens* has a LWE_{eff} of 44% (Table 3). The residual water after LWE therefore potentially resides in the cell tissue and fibre lumens. In general, fibres in hardwoods are not part of the water conductive system (Carlquist 2001).

The data in Table 3 can be used to calculate the distribution of wood and water masses and volumes before and after LWE (Table 4). The 98% *m/m* water remaining after LWE occupies 39% v/v of the specimen volume. This is proposed to reside in the cell walls and fibre lumens. The 54% *m/m* of water removed on LWE occupied 22% v/v of the specimen volume and it is proposed that this water was removed from the vessels and some of the elements associated with the vessels (ray and axial parenchyma and vasicentric tracheids). These conclusions are indicative only requiring further experimentation for confirmation.

This proposal offers a possible insight into the free water behaviour in the capillary fibre network of wood which has been modelled by Salin (2008, 2010). Salin developed a model to simulate the drying of sapwood in softwoods. However, since liquid tension is intrinsic to the model, it may also be applicable to the development for hardwoods. Salin applied a stochastic three-dimensional percolation model to groups of interconnected saturated fibres where the

Table 3 Means and 95% confidence intervals (in brackets) of basic density, green moisture content (MC_g), maximum MC (MC_{max}), % saturation, MC_{LWE} and lumen water expulsion treatment efficiency for *Eucalyptus nitens* specimens

	Basic density (kg m ⁻³)	MC _g (%)	MC _{max} (%)	Saturation (%)	MC ^a _{LWE} (%)	LWE ^b _{eff} (%)
E. nitens	399 (5.3)	152 (3.7)	185 (3.3)	82 (1.2)	98 (3.3)	44 (2.0)

^aMC_{LWE} is moisture content after LWE treatment

^bLWE_{eff} is the efficiency of lumen water expulsion

	Green			After LWE	LWE			
	Oven dry wood specimen	Cell wall only	Water	Total	Cell wall only	Water remaining	Water removed	Total
Weight (kg)	100	100	152 ^a	252	100	98 ^a	54	252
Density (kg m ⁻³)	399 ^a	1500 ^b	1000	_	1500	1000	_	_
Volume (m ³)	0.251	0.067	0.152	0.219 ^c	0.067	0.098	0.054	0.219 ^c
Volume (%)	100	27	61	87	27	39	22	87

Table 4 Masses and volumes of wood cell wall and water before and after LWE based on an oven dry wood mass of 100 kg

^aValues from Table 3

^b1500 kg m⁻³ is the density of oven-dry wood substance (Kininmonth 1991)

^cSaturated volume (cell+water) (82% saturation)

largest menisci in the network retreat into their associated fibres as water tension forces overcome meniscus forces. Salin showed that separated groups of fibres were being drained of their free water with no clear drying fronts. In the current work, the proposal is that the parenchyma, vessel and tracheid tissue constitute the group of cells that have their free lumen water removed. However, the fibres respond more slowly, in an analogous manner to that described using percolation modelling.

3.2 Shrinkage and collapse

There was no significant difference in the mean values for pure shrinkage, calculated using Eq. 1, over all treatments for *Eucalyptus nitens* (Table 5).

In contrast, the pure collapse of *Eucalyptus nitens*, calculated using Eq. 2, was significantly influenced by pretreatment and drying treatment (Table 5; Fig. 4). The lumen water expulsion pre-treatment significantly reduced collapse in both the pre-treatment and full treatment factors. The effect of the LWE on the kiln drying treatment was to reduce collapse by 75%, washboard depression values by 71% and the radial-tangential shrinkage ratios by 4%.

The reduction in collapse and washboard depression in *Eucalyptus nitens* on LWE is the result of a unique mechanism (Dawson et al. 2015; Pearson et al. 2019b). In traditional kiln drying, cell collapse occurs from meniscus-driven forces—the evaporation of water through the meniscus creates a negative water tension in the lumen water, which exceeds the average strength in compression perpendicular to cell walls which can collapse (Chafe et al. 1992; Booker 1993, 1994).

Table 5 Mean pixel areas for *Eucalyptus nitens* pure shrinkage and pure collapse, washboard depression, internal check areas and radial:tangential shrinkage ratios for experimental factors of pre-treatment, drying treatment and their full treatment interaction

Experimental factor	Schedule	Pure shrinkage (% area) ^a	Pure collapse (% area) ^a	Internal check area (% area)	R:T shrinkage	Washboard depression (mm)
Pre-treatment	Green	7.8 A	21.7 A	5.6 A	1.04 A	4.1 A
	LWE ^b	7.5 A	5.1 B	7.9 A	1.02 B	1.07 B
Drying treatment	Air	7.4 A	4.1 C	0.5 B	1.03 B	1.28 B
	Kiln	8.0 A	14.2 B	1.8 B	1.05 A	2.25 A
	Oven	7.2 A	22.4 A	48.4 A	0.99 C	2.82 A
Full treatment	Green-Air	7.6 A	6.4 DC	0.9 B	1.04 BC	1.87 C
	Green-Kiln	8.3 A	22.8 B	0.3 B	1.07 A	4.21 B
	Green-Oven	7.2 A	35.1 A	50.7 A	0.99 E	7.23 A
	LWE-Air	7.3 A	1.7 D	0.2 B	1.03 DC	0.87 D
	LWE-Kiln	7.7 A	5.6 DC	4.5 B	1.03 BC	1.21 DC
	LWE-Oven	7.2 A	9.7 C	46.1 A	1.00 DE	1.1 D

Means in columns with different letters are significantly different; p < 0.5

^a% area reduction

^bLumen water expulsion



Fig. 4 Pure collapse in *Eucalyptus nitens* for full treatments. Error bars show 95% confidence intervals

In contrast, in the high pressure phase of the supercritical CO_2 lumen water expulsion cycle, the possibility of negative water tension arising is circumvented. The supercritical CO_2 fluid (critical point 31 °C, 7.1 MPa) has liquid-like density while the water in cells remains liquid (critical point 374 °C, 21.8 MPa) and has surface tension. At the supercritical CO_2 fluid-liquid water meniscus, CO_2 will diffuse into the water in regions accessible by the supercritical fluid (Pearson et al. 2019b). Diffusion of water into the supercritical CO_2 is likely to be almost negligible. On depressurisation, CO_2 bubble formation creates a back pressure, which shunts liquid water out of the cells.

It was observed that Eucalyptus nitens specimens, still averaging 98% moisture content after supercritical CO₂ treatment, had greatly reduced collapse on subsequent oven, kiln or air drying down to 12% moisture content (Fig. 4). It is proposed that the menisci moved from the outside of the saturated wooden specimens to the interior because of the voids created by water removal from parenchyma, and vessel and tracheid tissue. As water is removed from these cell elements by LWE, the water column is broken throughout the specimen thereby disrupting the development of meniscus-induced water tension as subsequent drying occurs. Water exits from the specimen along the same pathways as CO₂ enters and exits. There is no evidence that the high pressure hold of 20 MPa has damaged tissue structure and is not thought to affect permeability except through water removal. The fibres in hardwoods, as proposed earlier, retain the bulk of the remaining water as they are not water conductive.

For both green and LWE treatments, there was no statistical difference in internal checking between the air drying or kiln drying treatments but the oven drying treatment was different to both other treatments and an order of magnitude higher (Table 5). Lumen water expulsion treatment had no effect on internal checking. Booker (1994) considered that internal checking and collapse were both caused by negative water tension. He predicted when cells had low compressive strength perpendicular to the cell wall, collapse without internal checking may occur if the collapsed region included board surfaces, which agrees with the present study where collapse was observed over multiple specimen surfaces.

Salvo et al. (2017) reported that the drying behaviour of 17 years old *Eucalyptus nitens* samples, from annual rings 6–10 resulted in higher collapse and internal checking compared with inner and outer wood. In contrast, specimens in the present work, showed collapse in rings 6–10, but little internal checking for the air- and kiln-dry treatments with stronger checking for the harsher oven-dry treatment. The stronger drying stresses experienced in the oven-dry treatment were not able to be dissipated while in the two slower drying treatments stress relief was possible.

3.3 Adoption of technology

Before supercritical CO_2 lumen water expulsion treatment technology can be applied commercially, there are challenges to be evaluated. Together with market knowledge data, this will lead to a value proposition for commercialisation.

- Treatments to date have been carried out on a pilot scale $(3000 \times 100 \times 50 \text{ mm})$ for only *Pinus radiata* timber. Trials for *Eucalyptus nitens* must be carried out to obtain data for larger dimension timber along with mass and energy flows.
- Modelling the process for *Eucalyptus nitens* is at an advanced stage but is still to be completed.
- Using the results of the factorial study for *Eucalyptus nitens* and the model will define the range of parameters that can be used and this will inform plant design.
- The cost of a plant is predicated on pressure and cycling requirements as these determine the cost and longevity of the plant.
- Expanding the range of hardwood species to include high value but very slow drying hardwoods independent of whether they are collapse-prone or not.

4 Conclusion

The hypothesis tested was would supercritical CO₂ lumen water expulsion prevent the development of negative water tension and thereby remove the driver of collapse, washboard depression and internal checking in *Eucalyptus nitens* heartwood. Results showed that the lumen water

expulsion pre-treatment, followed by kiln drying, reduced collapse in *Eucalyptus nitens* by 75% and washboard depression by 71%. Oven drying produced the highest drying stresses as evidenced by most collapse and air drying the least. Internal checking was low in all treatments except oven drying. The results confirmed this hypothesis for difficult-to-dry *Eucalyptus nitens*. Supercritical CO_2 lumen water expulsion pre-treatment circumvents the accepted negative water tension model of collapse.

It was proposed that the parenchyma, vessel and tracheid tissue volume in *Eucalyptus nitens*, which represents the water conductive system, is the source of the water removed by the lumen water expulsion treatment. The residual water after LWE treatment therefore potentially resides in the cell tissue and fibre lumens. In general, fibres in hardwoods are not part of the water conductive system.

The application of supercritical CO_2 treatment technology may offer the prospect of increasing the conversion of green collapse-prone species timber into products if they have a medium lumen water expulsion efficiency of 40–60%. Future work will consider treatments of timber of different dimensions and position within the tree and with tree age, looking at the response variables of collapse, internal checking and radial-tangential shrinkage ratio.

Acknowledgements This work was supported by Scion through funding from the New Zealand Ministry of Business, Innovation and Employment. Graeme Manley from Southwood Exports Ltd is thanked for supplying *Eucalyptus nitens* log sections. Rosie Sargent (Scion) is thanked for the description of the supercritical CO_2 lumen water expulsion plant; John Lee and Jamie Agnew (Scion) are thanked for log section reduction and specimen preparation; Maxine Smith (Scion) is thanked for volume displacement measurements.

Compliance with ethical standards

Conflict of interest There are no conflicts of interest associated with this research.

Open Access This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons licence, and indicate if changes were made. The images or other third party material in this article are included in the article's Creative Commons licence, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons licence and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this licence, visit http://creativecommons.org/licenses/by/4.0/.

References

- Abramoff MD, Magalhaes PJ, Ram SJ (2004) Image processing with ImageJ. Biophotonics Int 11(7):36–42
- Berry SL, Roderick ML (2005) Plant-water relations and the fibre saturation point. New Phytol 168(1):25–37. https://doi.org/10. 1111/j.1469-8137.2005.01528.x
- Booker R (1993) The importance of the S3 cell wall layer in collapse prevention and wood hardness. In: Paper presented at the proceedings 24th forest products research conference, 15–18 Nov 1993, 3/17, CSIRO Division of Forest Products, Clayton, pp 1–13
- Booker R (1994) Internal checking and collapse–which comes first. In: Paper presented at the proceedings of the 4th IUFRO wood drying conference: improving wood drying technology. Forest Research Institute, Rotorua, pp 133–140
- Budgen B (1981) Shrinkage and density of some Australian and South-east Asian timbers (Division of Building Research Technical paper). CSIRO, Melbourne
- Carlquist S (2001) Comparative wood anatomy. Systematic, ecological, and evolutionary aspects of dicotyledon wood. Springer, New York
- Chafe S, Barnacle J, Hunter A, Ilic J, Northway R, Rozsa A (1992) Collapse: an introduction. CSIRO, Division of Forest Products, Melbourne, p 9
- Cown D, Bigwood S (1979) Some wood characteristics of New Zealand-grown western red cedar (Thuja plicata D. Don). N Zeal J For 24:125–132
- Dawson BSW, Pearson H (2017) Effect of supercritical CO₂ dewatering followed by oven-drying of softwood and hardwood timbers. Wood Sci Technol 51(4):771–784. https://doi.org/10.1007/ s00226-017-0895-8
- Dawson BS, Pearson H, Kroese HW, Sargent R (2015) Effect of specimen dimension and pre-heating temperature on supercritical CO_2 dewatering of radiata pine sapwood. Holzforschung 69(4):421–430
- Denig J, Wengert E, Simpson W (2000) Drying hardwood lumber. (Gen. Tech. Rep. FPL-GTR-118). US Dept. of Agriculture, Forest Service, Forest Products Laboratory, Madison
- Franich R, Gallagher S, Kroese H (2010) Improvements related to wood drying. NZ Patent 582932 (2010), p 46
- Franich RA, Gallagher SS, Kroese HW (2013) Wood drying. US Patent 8,578,625, p 29
- Haslett AN (1990) Properties and utilisation of exotic speciality timbers grown in New Zealand. Part VI: Eastern blue gums and stringy barks. Eucalyptus botryoides Sm. Eucalyptus saligna Sm. Eucalyptus globoidea Blakey. Eucalyptus muellerana Howitt. Eucalyptus pilularis Sm. FRI Bulletin (119, pt. 6)
- Kauman W (1964) Cell collapse in wood. CSIRO Division of Forest Products, Melbourne, p 59
- Kininmonth JA (1972) Permeability and fine structure of certain hardwoods and effects on drying: II. Differences in fine structure of *Nothofagus fusca* sapwood and heartwood. Holzforschung 26(1):32–38
- Kininmonth JA (1991) Wood/water relationships. In: Kininmonth JA, Whitehouse LJ (eds) Properties and uses of New Zealand radiata pine, vol 1. Wood properties. New Zealand Ministry of Forestry, Forest Research Institute, Rotorua, pp 1–23
- Lausberg M, Gilchrist K, Skipwith J (1995) Wood properties of *Eucalyptus nitens* grown in New Zealand. NZ J For Sci 25:147–163
- Pearson H, Dawson B, Kimberley M, Davy B (2019a) Modelling and optimisation of ceramic and wood dewatering using supercritical CO₂. J Supercrit Fluids 146:15–22. https://doi.org/10.1016/j. supflu.2019.01.004

- Pearson H, Dawson B, Kimberley M, Davy B (2019b) Predictive modelling of supercritical CO₂ dewatering of capillary tubes. J Supercrit Fluids 143:198–204
- Redman AL, Bailleres H, Turner I, Perre P (2016) Characterisation of wood-water relationships and transverse anatomy and their relationship to drying degrade. Wood Sci Technol 50(4):739– 757. https://doi.org/10.1007/s00226-016-0818-0
- Salin J-G (2008) Drying of liquid water in wood as influenced by the capillary fiber network. Dry Technol 26(5):560–567. https://doi.org/10.1080/07373930801944747
- Salin J-G (2010) Problems and solutions in wood drying modelling: history and future. Wood Mater Sci Eng 5(2):123–134. https://doi. org/10.1080/17480272.2010.498056
- Salvo L, Leandro L, Contreras H, Cloutier A, Elustondo D, Anaias R (2017) Radial variation of density and anatomical features of *Eucalyptus nitens* trees. Wood Fiber Sci 49(3):301–311
- Simpson W, TenWolde A (1999) Physical properties and moisture relations of wood. In: Wood Handbook: wood as an engineering

material (vol. Gen. Tech. Rep. FPL-GTR-113. Forest Products Laboratory, Madison, p 463

- Wilkes J (1988) Variations In wood anatomy within species of *Eucalyptus*. IAWA J 9(1):13. https://doi.org/10.1163/22941932-90000 461
- Yang L, Liu H (2018) A review of *Eucalyptus* wood collapse and its control during drying. BioResources 13(1):2171–2181
- Ziemińska K, Butler DW, Gleason SM, Wright IJ, Westoby M (2013) Fibre wall and lumen fractions drive wood density variation across 24 Australian angiosperms. AoB Plants 5:plt46. https:// doi.org/10.1093/aobpla/plt046

Publisher's Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.