PRESERVATIVE TREATMENT OF TASMANIAN PLANTATION *EUCALYPTUS NITENS* USING SUPERCRITICAL FLUIDS

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Abstract. Short rotation plantation forests in Tasmania, Australia, are dominated by *Eucalyptus nitens* (common name: shining gum). These forests were primarily planted to provide material for pulp and paper production, but the timber is increasingly sought after for higher value and more enduring applications.

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Plantation *E. nitens* has a high proportion of low-durability heartwood that resists penetration by conventional fluid preservatives. This limits its use to indoor applications. One approach to overcoming the refractory nature of *E. nitens* is to modify the treatment fluid. We investigated the use of supercritical carbon dioxide to deliver biocides deep into the wood. Timbers varying in thickness from 19 to 35 mm and 900-mm long were treated with a multicomponent biocide under supercritical conditions in a commercial facility in Denmark. The resulting timber was cut into zones inward from the surface. Wood from these zones was grounded and extracted for HPLC analysis for tebuconazole and propiconazole. Preservative was detected in the inner portion of every sample examined, indicating that the process resulted in treatment throughout the boards, with concentrations meeting and on average exceeding the targeted amounts.

Keywords: *Eucalyptus nitens*, preservative treatment, supercritical carbon dioxide, tebuconazole, propiconazole, wood preservation.

INTRODUCTION

Timber durability is a key consideration for architects and engineers concerned with reducing the environmental impact of buildings. Building construction contributes 39% of the world's carbon emissions, with building materials making up 11% of those emissions (Global ABC 2019). Structurally and aesthetically, wood can rival nonrenewable materials like steel and concrete that are also far more carbon intensive to produce (Churkina et al 2020; Allen et al 2021). In terms of durability, however, wood is a biological material that can be degraded by a number of abiotic and biotic agents under the proper conditions (Archer and Lebow 2006; Zabel and Morrell 2020). Designers can limit biodegradation risk, eg with water shedding designs, or by only specifying timber in interior applications. In instances where long-term wood contact with moisture is unavoidable, species that produce naturally durable heartwood may be specified, but the supply cannot always meet the demand for durable timbers. Alternatively, timber can be artificially impregnated with preservatives to protect against fungal, insect, and even marine borer attack.

In most regions, preservative treatment specifications address the easily treated sapwood of coniferous species, such as Scots pine, radiata pine, or the southern pines. Heartwood of these species is generally far more difficult to impregnate, and most treatment standards specify a high percentage treatment of the sapwood coupled with a shallow penetration of the heartwood (eg a 5-10 mm envelope). While softwoods are important in global commerce, many countries have expanding supplies of low-durability plantation hardwood timbers that require preservative treatment for exterior exposures. For example, Tasmania has extensive plantations of *Eucalyptus nitens* that were originally planted and managed for pulp and paper production, but are increasingly viewed as a potential resource for building construction including in exterior applications, where biodeterioration is likely (Wood et al 2020).

Plantation *E. nitens* is classified as Durability Class 4 in Australian Standard AS 5604 (Standards Australia 2005) and is characterized by shallow bands of sapwood surrounding a large core of lower durability heartwood that is highly resistant to conventional fluid preservative treatment. These refractory characteristics make it extremely difficult to meet the current Australian Standards for exposure in exterior Hazard Classes (H3, outside, above ground; or H4, in ground contact) as described in Standard AS/NZS 1604.1 (Standards Australia 2021), limiting its potential uses to indoor applications. It is also notoriously difficult to kiln dry without excessive defects and air-drying usually requires prolonged periods of a year or more depending on the thickness of the board, followed by reconditioning, final kiln dry, and planing or molding. Any preservative treatment that significantly rewets the wood after the material has been planned would potentially require another lengthy drying period, followed by a costly second reconditioning, final kiln dry, and planing or molding.

Globally, a variety of alternative methods have been explored for impregnating difficult to treat or refractory heartwoods, including modifying the solvent characteristics (Siau 1971), increasing

pressure, or using alternative pressure methods, but none of these can completely overcome the refractory nature of heartwood on their own (Hunt and Garratt 1967; Nicholas and Siau 1973; Cookson 2000). Timbers can be incised to increase the amount of cross-sectional area exposed to fluid flow, but this process mars the surface appearance, can reduce strength, and is mostly only effective to the depth of the incisions (Winandy et al 2022). Diffusible compounds, especially boron, have also been explored because of their ability to spread with moisture into normally refractory timber, but their water solubility renders them susceptible to leaching and, therefore, not suitable for exterior exposures (Lloyd 1998; Freeman et al 2009). Gaseous boron can also be used, but it has largely been limited to applications on composites (Burton et al 1990).

In Australia, the requirements for quality assurance of preservative treatments include visual assessment of the entire cross section of a treated board to ensure complete sapwood penetration as well as minimum heartwood treatment depths (AS/NZS 1604; Standards Australia 2021). This is usually done using colorimetric spot tests; however, a penetration tracer, such as zinc or copper, may be added when the preservative cannot be visually assessed (eg as in the case of azoles). The current Australian Standard (AS/NZS 1604) requires a minimum penetration of 5 mm in the heartwood of any hardwood board equal to or less than 35-mm thick, and complete penetration of the sapwood.

As noted previously, 5 mm of heartwood penetration of *E. nitens* is exceedingly difficult to consistently achieve as illustrated by two recent reports prepared for the Australian National Institute for Forest Products Innovation (Wood et al 2023a, 2023b), which outline a comprehensive suite of treatment strategies that were trialed to improve the durability of heartwood-dominant refractory Australian *Eucalyptus* species, including *E. nitens.* (The material used in both trials was from the same general source as the material used for the research described in this paper.) One of the strategies trialed was an extensive vacuum pressure

impregnation trial that used varied scheduling, pressure, solution strengths, and chemical composition, including both copper-based and azole-based Australian-approved biocides. In the study of azole-based biocides (Wood et al 2023a), chemical retention analysis was not undertaken, and theoretical retention was unable to be assessed as the biocide used was a commercially available, ready-to-use solution and limited information was provided on the schedule and solution strengths. However, extremely low uptakes averaging less than $10 \, L/m^3$ were recorded, which is far less than the typical targeted amount for a hardwood to meet exterior application requirements in Australia (ie approximately $40\text{-}45 \, L/m^3$). In addition, because the biocide included trace amounts of copper, visual penetration assessments were completed on oven-dried biscuits using a preservative indicator (PAN [1-(2-pyridylazo)-2-napthol]) and evaluated against the criteria in the Australian Standards (AS/NZS 1604; Standards Australia 2021) using a grid analysis (Wood et al 2023a [see Fig 14], p. 22). The results indicated that azole-based biocides barely penetrated the *E. nitens* boards. Similar trials using alkaline copper quaternary (ACQ) compound and micronized copper azole (MCA) showed that samples could not consistently meet the required retention targets (Wood et al 2023a). Although some better penetration was achieved using novel process enhancements, treatments still failed to produce consistent preservative penetration demonstrating the difficulty of treating *E. nitens* using conventional vacuum pressure impregnation.

One potential alternative for impregnating refractory heartwoods like *E. nitens* is supercritical fluid (SCF) treatment. SCFs are defined as materials that are at a temperature and pressure where distinct gas and liquid phases do not exist. SCFs can behave like a liquid in terms of density, which can aid in solubility and viscosity that can facilitate movement through the wood matrix (Krukonis 1988; Kayihan 1992; Sahle-Demessie et al 1995a, 1995b; Kjellow et al 2010). SCF density and viscosity are easily adjustable by varying pressure or temperature. While a variety of solvents can be used for SCF treatments, carbon dioxide (CO_2) is

more commonly used because of its low cost, minimal toxicity, and low critical temperature/ pressure. It can also be captured and recycled in a closed-loop treatment system.

Supercritical CO_2 (SC-CO_2) can solubilize a variety of organic preservatives at levels capable of protecting wood from decay and insect attack and has been shown to penetrate into a variety of wood species as well as wood-based composites (Hassan et al 1995; Kjellow and Henriksen 2009). Supercritical wood impregnation has been found to have no significant effects on timber properties despite the elevated pressures as long as pressure differentials are minimized (Smith et al 1993; Anderson et al 2000; Schneider et al 2003, 2005; Oberdorfer et al 2006). Limited field trials suggest that plywood impregnated with tebuconazole using SC-CO_2 performed well in a subtropical H3 exposure, but there have been few other public long-term durability studies (Acda et al 1997, 2001; Morrell et al 2005; Kjellow et al 2013).

SCFs have been used to a lesser extent to treat hardwoods. Anderson et al (2000) explored the effects of treatment on flexural properties of white oak and sweetgum beams. Cookson (2009) reported on small-scale treatments of Australian hardwoods with permethrin for termite control, but the work was not commercialized. One major hurdle to the commercial use of SCFs is the large capital investment required for the treatment plant; however, the operating costs can be lower than regular treatment facilities and the ability to effectively impregnate species that are not currently suitable for exterior exposure creates a range of new market opportunities. There is already one commercial facility located in Denmark, which currently uses SCFs to impregnate timber with a biocide suitable for exterior exposure. Their treatment facility concentrates on Norway spruce (*Picea abies*) for above-ground applications, such as cladding and decking.

SCFs treatment might be an attractive alternative for properly impregnating the heartwood of Tasmanian plantation *E. nitens* with biocides for H3 or H4 exposures. The process also has some advantages from a processing perspective since it is nonswelling and can be used to treat finished and shaped timber without the need for posttreatment sanding or planing. However, the process can also induce collapse or internal checks as a result of excessive surface vs internal pressure differentials if pressurization and depressurization rates exceed the rate at which the wood can equilibrate pressure (Oberdorfer et al 2006). While SCF treatments have been explored for a number of timber-related applications, there is little in support of using this process to treat *E. nitens*. The objective of this study was to evaluate the effects of SCF treatment on preservative penetration and internal condition of *E. nitens* timber.

MATERIALS AND METHODS

E. nitens timber (90 × 35 × 900-mm long) with an average oven-dry density of 547 kg/m^3 was cut from trees in a 26-yr-old plantation that had been thinned and pruned over the rotation. The timber had been air-seasoned for approximately 6 to 9 mo, then reconditioned and kiln-dried to a target MC of 12% (mass % oven-dry basis) prior to final planing and cutting. On average, less than 5% of the boards contained any sapwood at all and of those boards, there was less than 2% sapwood on each sample, usually concentrated in an outer corner of the board due to the quartersawn cutting pattern. Fifteen samples each of 19, 25, or 35-mm thickness were all cut from the same parent material and the samples were sent to Danish commercial SCF facility, Superwood A/S (https://www. superwood.dk/), for inclusion in one of their commercial spruce treatment charges. Before treatment, the wood was conditioned to a constant weight at 85% RH and 20°C to a final MC of 19% MC. The higher MC was used because experience from commercial treatment of spruce shows that wood at this moisture level is less prone to cracking during treatment.

The treatment process consisted of pressurizing the treatment vessel with CO_2. During pressurization, the CO_2 was continuously circulated between the treatment vessel and a static mixer containing the biocide, gradually dissolving the biocides and bringing these to the wood. Treatment temperature

throughout the process was 45°C and maximum impregnation pressure was 15 MPa. Pressure was held to allow the biocides to diffuse inward, then gradually released back to atmospheric levels thereby depositing the biocides in the wood. Total treatment time was 7 h, which is longer than is normally used for spruce boards. The prolonged rates of pressure increase and decrease were used as previous tests on other dense species showed that they minimized the development of pressure gradients that induced internal stresses leading to either collapse or internal checking/splitting.

The samples were treated with SC200, a mixture containing tebuconazole, propiconazole, and iodopropynyl butylcarbamate (IPBC) in a relative ratio of 2:2:1. The target concentration for the spruce boards was 120 g/m^3 of total active ingredient, which is the biological reference value of SC200 against basidiomycetes determined by the biological test regime specified in EN 599-1:2009 + A1:2013 for Use Class 3 exposure (outside, above ground). In conventional treatments, it is possible to weigh the wood before and after treatment to determine net solution uptake; however, SCF treatments can solubilize wood extractives during the process while simultaneously depositing the biocides, potentially resulting in net weight losses. In addition, the target deposition of 120 g/m^3 corresponds to only about 0.00025 kg of actives per 1 kg of wood, so the uncertainty of a weight measurement would be too large. Chemical analysis was used instead to measure the amount of biocide through the cross sections of boards of each thickness.

After treatment, each 900-mm long parent board was cut into five sections with three 200 mm long planks retained for durability testing in a separate study, and two 150 mm cross sections for retention analysis. A 5-mm thick cross section was cut from each of the two 150-mm long cross sections and these were further divided into corresponding inner and outer zones for the 19- and 25-mm thick timbers and inner, middle, and outer zones for the 35-mm thick timber (Figs 1 and 2). Outer zones were 0-5 mm from the surface for the 19- and 25-mm thick timbers, while the inner zones corresponded to the zone 6-9 mm inward and 6-12 mm
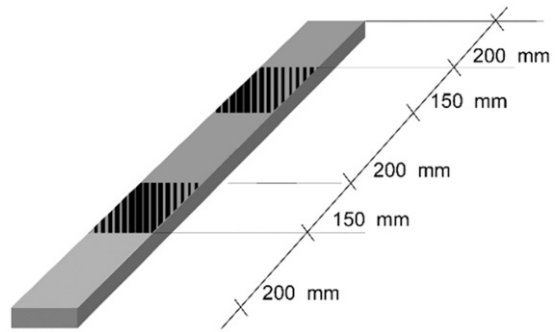

Figure 1. Schematic for 900-mm long SCF treated cut into 3×200 mm long planks and 2×150-mm long planks following treatment. Hatching indicates area from which a 5-mm biscuit was obtained for retention analysis.

inward for the 19- and 25-mm thick timbers, respectively (accounting for some loss due to the saw kerf). The 35-mm thick samples were cut into outer, middle, and inner zones corresponding to 0-5, 6-11, and 12-17 mm from the wood surface, respectively (Fig 2). Samples from a given zone were ground to pass a 20 mesh screen, oven-dried for 2 h at 80°C, and stored in a desiccator until cool. Approximately 0.5 g of ground wood from a given section and distance from the surface was added to a screw cap tube along with 25 mL of methanol and sonicated for 180 min at room temperature. The resulting liquid was filtered through a SAX SPE cartridge to remove particulate and interfering compounds.

The resulting filtrate was analyzed for tebuconazole and propiconazole on a Shimadzu High Performance Liquid Chromatograph equipped with an Intersil ODS-3 (150×4.6 mm, 3 μm) column. The mobile phases were 1) 55:45 acetonitrile:buffer, 2) acetonitrile, and 3) 2% methanol in HPLC water introduced at a flow rate of 1.00 mL/min. The two azoles were detected at 195 nm and quantified by comparison with similar analyses of prepared standards. The third preservative component (IPBC) was not analyzed in this experiment.

RESULTS AND DISCUSSION

There was no visible evidence of excessive crushing, splitting, or collapse in the samples following
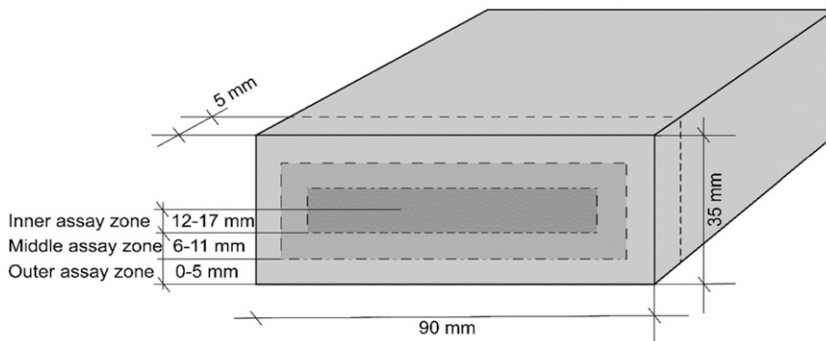
Figure 2.   Schematic showing the cutting pattern for the three assay zones on a 35-mm thick sample.

treatment; however, some minor collapse was observed in the samples after several months stored under ambient conditions indoors (approximately 17-25°C) indicating that there may have been excess moisture in the timber that subsequently dried during the storage period indoors (Fig 3). This was particularly evident on the surface of the 35-mm thick samples and was more apparent when samples were cut into assay zones for retention analysis. The long lag between treating and collapse makes it unlikely that the collapse was due to pressure differentials during SCF treatment, but rather due to the reconditioning of the timber to 19% MC prior to the treatment. This conditioning was done to aid heat transfer through the wood and make it slightly more plastic to mitigate potential cracking. Ovendry MC was not determined directly following treatment or in the months after the treatment; however, we do not believe that MC changed as a result of treatment because there was minimal potential for drying during treatment.

It is worth noting that interior and exterior timber in Australia is required to be at 9-14% MC at time of sale (AS 2796.1, Standards Australia 1999). This would require some redrying if the moisture conditioning to 19% was included in the treatment process. Further studies to determine the optimum moisture level that minimizes physical damage are recommended.

Chemical analyses revealed that both tebuconazole and propiconazole were detectable in all assay zones of the cross sections of every sample tested (Fig 4, Table 1), and all samples met and exceeded, on average, the targeted requirement of $120 \, g/m^3$ for treatment of spruce using SC200. While retentions in the middle and inner zones of the three thicknesses of wood were much lower than those near the surface, both azoles were detected in every treated sample analyzed, indicating that $SC\text{-}CO_2$ was capable of carrying the biocides well into the heartwood. These analyses did not include the levels of IPBC in the wood.
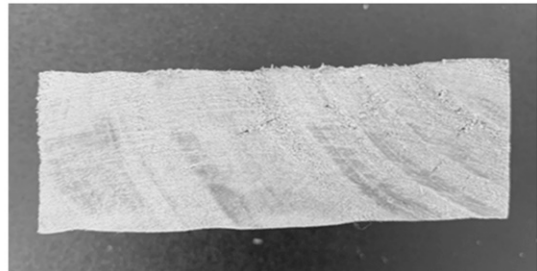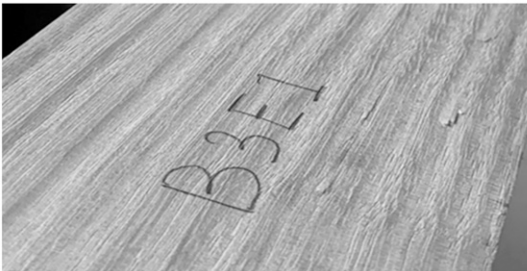

Figure 3.   Examples of surface checking (left) and internal collapse (right) in 35-mm thick *Eucalyptus nitens* boards after SCF impregnation.
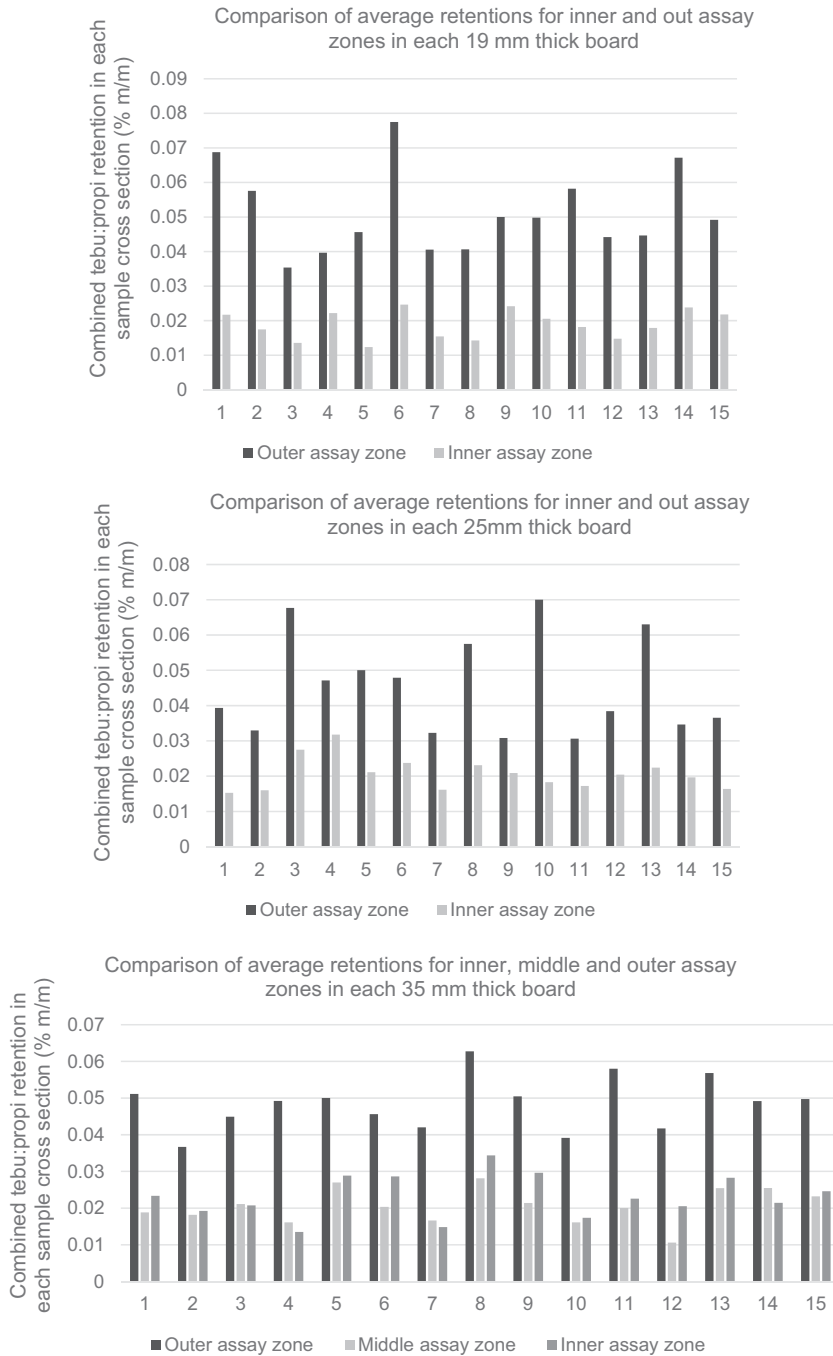
Figure 4.   Retention of tebuconazole/propiconazole at selected depths of (top) 19-mm thick (middle), 25-mm thick and (bottom), 35-mm thick *E. nitens* timbers treated using supercritical carbon dioxide. Note: in the 19-mm thick sample set, two vessels were lost/broken during extraction which may affect the averages for the inner assay zones in boards 7 and 14. Also, although the middle assay zones appear lower than the inner assay zone in some of the 35-mm thick boards, this variation between the two averages was insignificant.

Table 1. Average g/m^3 of propiconazole/tebuconazole in *E. nitens* timbers treated using supercritical carbon dioxide[a].

Sample thickness (mm)	Assay zone	Average g/m^3 by assay zone[b]	Total g/m^3 in cross section[b]
19	Outer 0-5 mm	277 (68)	191
	Inner 6-14 mm	104 (26)	
25	Outer 0-5 mm	226 (77)	165
	Inner 6-19 mm	103 (28)	
35	Outer 0-5 mm	265 (52)	161
	Middle 6-11 mm	103 (32)	
	Inner 12-24 mm	116 (35)	

[a] Samples were treated to the spruce target retention of 120 g/m^3 of the azole/IPBC mixture.

[b] g/m^3 is a less precise treatment measure as SCF treatments can solubilize wood extractives during the process while simultaneously depositing the biocides, potentially resulting in net weight losses. Values represent analyses of 30 replicates per assay zone for three board thicknesses, and 60 or 90 analyses for the combined cross sections for the 19/25 mm and 35-mm thick samples, respectively. Values in parentheses represent one standard deviation.

IPBC is highly soluble in SC-CO_2 and would have further increased the retentions (Hassan et al 1995).

As noted in the introduction, Australian Standards for quality assurance of preservative treatments use visual assessments to ensure complete sapwood penetration as well as minimum heartwood treatment depths (AS/NZS 1604; Standards Australia 2021). Unfortunately, the chemicals used in the treatment of spruce at Superwood lacked either a specific color or a tracer making it difficult to determine whether the detected azole was uniformly distributed throughout a given zone. Establishing a method for visual evaluation of penetration will be critical for quality assessment of SCF azole treatment in an Australian context.

The Australian Standard also requires a minimum retention of 0.03% tebuconazole and 0.03% propiconazole mass/mass for H3 exposures based on the oven-dry mass each piece; a total of 0.06% m/m combined azoles. In this paper, analyzed retention % m/m was converted to g/m^3 for clarity using the following formula:

$$\text{Retention} (g/m^3) = \frac{\left\{ \begin{array}{l} retention\ (\%\ m/m)\ \times \\ oven\ dry\ timber\ density\ (g/m^3) \end{array} \right\}}{0.1}$$

Combined azole retentions in the SCF treated *E. nitens* boards were all below the Australian target level (Table 2); however, it is important to note that the system used for SCF treatment also contained IPBC, which was not measured in this study. In addition, the treatment process was designed for spruce and not specifically for this species. A further caveat is that the Australian Standard specifies analysis of the entire cross section. Average retentions for the combined inner, middle, and outer zones were 0.0348, 0.0330, and 0.0308% m/m, respectively, for the three thicknesses (Table 2).

Table 2. Retentions of propiconazole/tebuconazole in *E. nitens* timbers subjected to treatment using supercritical carbon dioxide (% m/m).

Sample thickness (mm)	Propiconazole/Tebuconazole retention (% m/m)[a]						Combined Avg
	Outer		Middle		Inner		
	Avg	Range	Avg	Range	Avg	Range	
19	0.0507 (0.0125)	0.0347-0.0853	—	—	0.0189 (0.0047)	0.0088-0.0292	0.0348
25	0.0453 (0.0140)	0.0224-0.0731	—	—	0.0207 (0.0052)	0.0118-0.0323	0.0330
35	0.0485 (0.0094)	0.0306-0.0687	0.0206 (0.0058)	0.0065-0.0323	0.0232 (0.0064)	0.0097-0.0361	0.0308

[a] Values represent 30 replicates per zone and sample thickness. Values in parentheses represent one standard deviation.

Figure 5. Example of extremely shallow and minimal penetration in the heartwood of conventional vacuum pressure treated *E. nitens* as indicated by darkened color on the corners of samples sprayed with PAN (1-[2-pyridylazo]-2-napthol) for the presence of copper.

The more important treatment parameter for this study was the retention away from the surface as an indicator of penetration, and, as noted above, it was clear from the chemical analysis that treatment was present in each assay zone, indicating that SC-CO_2 was able to carry biocides well into the heartwood of each sample. This result was in sharp contrast to those obtained using conventional vacuum treatment processes in a separate study (Fig 5).

While the levels of azoles in the treated materials were lower than the current requirements in the Australian Standards, the most obvious solution for achieving the Australian benchmarks would be to increase the amount of fungicide in the treatment vessel.

The use of longer treatment times would sharply reduce production capacity, but cosolvent addition to enhance azole solubility would increase the potential for diffusion. Previous studies have shown that solubilities of tebuconazole increased markedly with methanol addition and field trials of SC-CO_2 tebuconazole treated plywood in a subtropical environment showed that the resulting products performed well in above-ground exposures (Hassan et al 1995; Acda et al 1996, 1997a, 1997b, 2001).

CONCLUSIONS

SCF treatment resulted in far deeper preservative penetration in the heartwood of *E. nitens* than is typical for conventional preservative pressure treatments. The treatment target of $120\,g/m^3$ was reached and even exceeded in each board. While the retentions were below those required for azole treatment in an Australian context, this could be likely overcome by the addition of more fungicide in the treatment vessel, or by using modifiers to increase solubility. The results illustrate the potential for improved preservative protection of this species for above-ground applications and further research is planned.

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