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DURABILITY PERFORMANCE OF *EUCALYPTUS NITENS* IMPREGNATED USING SUPERCRITICAL CARBON DIOXIDE

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ABSTRACT: Wood remains one of our most important carbon neutral structural materials, but many wood species are susceptible to biodegradation. Preservative treatment can minimize degradation, but some species are exceptionally resistant to preservative penetration. An excellent example is shining gum, *Eucalyptus nitens*, which is globally planted and especially abundant in Tasmania. This species has low decay resistance and is exceedingly difficult to effectively treat using conventional processes. One alternative approach is to modify the treatment media using supercritical carbon dioxide (SC-CO). A previous study showed that shining gum could be effectively treated using a mixture of fungicides in SC-CO. However, the ability to deliver biocides into the wood may not necessarily translate into biological performance. The impregnated materials were subjected to laboratory decay tests using an aggressive brown rot fungi and above ground field tests in Queensland and Tasmania, Australia. Laboratory tests were inconsistent, owing to the wide variations in preservative retention. Field trials are nearly three years old and beginning to show results.

KEYWORDS: *Eucalyptus nitens*, shining gum, durability, supercritical carbon dioxide, biodegradation

1 – INTRODUCTION

Timber will play an increasingly important role in the development of carbon neutral structures, but one negative aspect of wood is its susceptibility to biodegradation. Preventing decay primarily involves design to exclude moisture, but this is not possible in some applications. In these cases, supplemental biocides are often used to provide protection. The sapwood and in some cases the heartwood of many softwoods are easily impregnated with wood preservatives, but the heartwood of many Australian hardwood species is very resistant to treatment. An excellent example is shining gum (*Eucalyptus nitens*), which is fast growing and frost tolerant. The heartwood of this species is refractory and has little resistance to fungal or termite attack [1]. Efforts to treat timber of this species using conventional processes have consistently failed, limiting safe application to interiors [2]. One alternative approach to protection of this species is to use a different fluid to facilitate penetration through the normally refractory heartwood. Supercritical carbon dioxide (SC-CO) has been explored for over 30 years for treatment of refractory woods. SC-CO has many attractive properties for wood treatment [3,4]. It has relatively low critical temperatures (31°C) and pressures (73.8 bar) and can solubilise a wide range of organic biocides.

Previous studies have shown that SC-CO can effectively penetrate a range of refractory softwood species [5,6] and there is one commercial treatment facility in Denmark treating spruce (*Picea abies*). A further study conducted by Wood et al. [7] showed that SC-CO was able to achieve higher than targeted retention levels of a given biocide in the refractory heartwood of shining gum without causing undue collapse or deformation of the timber.

The samples were then exposed in laboratory and field decay trials, the results of which are discussed in this paper.

2 – BACKGROUND

In a previous study [7], the potential for using SC-CO to impregnate shining gum boards of varying thicknesses with preservative was explored at a commercial treatment facility, Superwood A/S, in Denmark (https://www.superwood.dk/). Forty-five boards, fifteen each of 900 mm (L) x 100 mm (W) x 19 mm, 25 mm or 35 mm (D), were included in a spruce treatment using a mixture of tebuconazole, propiconazole and 3-iodo-2-

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propynyl butylcarbamate (IPBC) at a 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 European Standard EN 599-1:2009 + A1:2013 for Use Class 3 exposure (outside, above ground).

Preservative retention in the shining gum was determined by cutting sections different distances from the surface of each treated board and analysing for the azole content. Azoles were present in greater concentrations closer to the surface of each board than at the core. Average retentions in the cross section for the tebuconazole and propiconazole were 174 g/m^3 in 19 mm boards, 165 g/m^3 in 25 mm boards and 154 g/m^3 in 35 mm boards, well above the targeted average of 120 g/m^3 for spruce (note these amounts didn't include IPBC which if included would increase the averages even further). Furthermore, there was no evidence of cracking or deformation [7].

While these retention results were promising in showing the ability to deliver preservatives far deeper in the wood than would be possible through conventional pressure treatment, field and laboratory exposure to fungi provide a more accurate indication of the performance of the treated material. The SC-CO treated shining gum was included in laboratory and field trials.

3 – MATERIALS AND METHODS

Laboratory trial

For the first laboratory trial, SC-CO treated samples were exposed to an aggressive brown rot fungus (*Fomitopsis ostreiformis*) following methods outlined in the Australian Wood Protection Committee protocols (AWPC Laboratory Decay, Hazard class 3, 4 and 5: nutrient medium test) [8]. Four thin crosscut sections, ~5 mm thick, were cut from each of ten SC-CO treated parent boards of each thickness (19 mm, 25 mm and 35 mm) and labelled A-B-C-D. Sample sections were then oven dried (60°C) and weighed (0.001 g). The sections were sterilized by steaming for 10 minutes at 100°C along with untreated control samples, and distributed among fourteen 1 % malt extract agar in boxes previously inoculated with an aggressive brown rot fungus (*F. ostreiformis*). Samples were incubated at 25°C for 9 weeks before being removed. Mycelium was scraped from the sample which were weighed, oven dried (60°C) and weighed again to determine mass loss. Every box had at least one untreated pine sapwood control to compare mass losses.

Field trials

In addition to the small-scale laboratory trial, SC-CO treated samples were exposed at two field sites, one in Tasmania (cool, temperate) and one in Queensland (sub-tropical, termites), Australia, in ground proximity tests following the methods outlined in Australian and American protocols (AWPC Field Decay, Hazard Class 3: ground proximity test; and AWPA E18-15 Standard field test for evaluation of wood preservatives to be used above ground [UC3B]) [8, 9]. The test method creates reasonably aggressive conditions for fungal attack out of soil contact.

Sample sections measuring 125 mm x 100 mm x varying thicknesses (19 mm, 25 mm and 35 mm) were cut from each of the forty-five treated parent boards and labelled. Samples were then placed on concrete blocks and the arrays were covered with a permeable shade cloth that allowed rainfall to penetrate but kept the samples shaded. The covers also helped maintain elevated humidity to facilitate fungal attack. Matching samples were installed at Upper Castra in Tasmania and at Nambour in Queensland. Samples were assessed annually using a pick test method and rated from 10 - 0 based on the AWPA Decay Rating Scheme (10 = sound, no evidence of decay; 0 = fail, broken or probe goes right through the sample) [9].

3 – RESULTS AND DISCUSSION

A primary reason for undertaking this research was to establish whether SC-CO treatment using SC200, a registered and approved preservative treatment for above ground applications in Denmark, provided any protection from biological attack in the treated shining gum material under Australian conditions. It is important to note that, although the retention amounts of the given preservative exceeded the targeted amount in each board in the previous trial [7], retention requirements for those same preservatives are much higher in the Australian/New Zealand Standards [10]. Australian Standards measure the amount of preservative treatment in each board as a % mass/mass based on the oven-dried mass of the test specimen. For light organic solvent preservatives, propiconazole and tebuconazole (1:1), the main ingredients in the SC200 treatment, AS/NZS1604:2021 requires an amount equivalent to or exceeding 0.06% mass/mass, but average combined retentions of tebuconazole and propiconazole for each thickness in this trial were

approximately 0.03% mass/mass [7]. Additionally, samples in the field trial in sub-tropical Queensland were exposed in an area where termites are known to be active, but the SC200 treatment does not contain an insecticide. Another limitation was that there are no indicators for detecting penetration of azoles, although chemical analysis of the inner zone of the samples suggested that azoles were present. Conceptually, preservative may be detected in composite samples, but the micro-distribution from the surface to the interior may be more sporadic and this could result in poor performance.

Laboratory trial 1

Table 1 shows that mass losses of non-treated radiata pine averaged over 61 % indicating that conditions were suitable for aggressive fungal attack. Results from agar block decay tests of SC-CO treated samples against the aggressive brown rot fungus, *F. ostreiformis* resulted in mass losses between 6 and 18 % in SCF treated blocks. Mass losses varied widely among the blocks, suggesting that SCF impregnation was ineffective. However, it is important to note that chemical analysis examined an average retention of biocide in a given area. The lack of an indicator for visualising biocide distribution on a finer scale makes it difficult to determine if the mass losses were due to localized areas of low retention or actual fungal resistance to the biocide. These results might suggest that SCF was less effective in penetrating shining gum than it appears from the retention analysis tests in the previous study [7]. However, it is important to note the limitations of the laboratory decay tests that are normally performed on uniformly impregnated intact blocks of sapwood. The azole retentions in these SC-CO treated heartwood samples were deposited in a gradient with higher concentrations near the surface and lower retentions further inward and then cut into wafers for this experiment, exposing the lesser treated core directly to the brown rot fungus. While mass losses averaged around 15-16% in SC-CO treated blocks, these levels were less than 25% of those found for the untreated pine. These results suggest that higher retentions may be needed where SC-CO treatments are used; however, it is important to note that azole performance is closely tied to other components of the preservative system, especially water repellents. Much further research will be needed to determine the potential performance differences between conventional solvent-based systems and SC-CO treatment.

Field trials

Parallel field trials of SCF treated samples in an above ground exposure test are only in their third year but already some decay is beginning to occur. The samples were rated for degree of decay on the top and undersurface. The undersurface typically remains wetter for longer periods of time presenting a far more severe risk of both decay and leaching.

Table 2 shows that decay tended to be more severe on the lower surfaces of samples exposed in Queensland, while they were similar to the upper surface on Tasmanian samples. These results reflect the higher decay hazard posed at the former site. Samples at the Queensland site showed higher levels of fungal decay than those in Tasmania, particularly in the 35 mm thick samples. This suggests that while the SC-CO treatment provide some protection across all samples, it was less effective in thicker boards. The lower levels of protection could reflect variations in preservative distribution since the cutting exposed inner zones that might have had lower biocide retentions, but they could also reflect conditions for decay as thicker samples are more likely to dry out more slowly after wetting, creating conducive decay conditions for longer time periods. As noted in the the laboratory decay trial (above), retention analysis results from the background treatment trial [7] showed that azoles were clearly deposited in higher concentrations closer to the surface, with minimal levels towards the core of each board. Cutting boards into smaller

Table 1. Average mass losses for SC-CO treated shining gum samples following exposure to *Fomitopsis ostreiformis* (brown rot fungus) in agar boxes for nine weeks

SC-CO treated shining gum heartwood original thickness	5 mm wafers	
	Reps	***Average % of mass loss***
19mm	35[c]	15.21 (3.82)
25mm	35	16.45 (3.46)
35mm	36	16.08 (6.3)
Pine sapwood control[b]	23	61.47 (26.26)

[a]Values represent means while figures in parentheses represent one standard deviation.
[b]At least one untreated *P. radiata* sample was included in each box as a control.
[c]The original number of repetitions was 40 for each thickness, however some specimens were damaged during the experiment.

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Table 2. Average decay ratings for SC-CO treated shining gum exposed in ground proximity decay tests at sites near Nambour, Queensland, and Upper Castra, Tasmania, Australia

Sample thickness	Average Decay Rating[a]					
	Reps	***30 months, TAS***		*Reps*	***30 months, QLD***	
		SC-CO (Top)	*SC-CO (Tag)*		*SC-CO (Top)*	*SC-CO (Tag)*
19mm	15	9.97 (0.13)	9.87 (0.23)	15	9.73 (0.59)	8.13 (0.92)
25mm	15	10 (0)	9.93 (0.18)	15	9.80 (0.56)	7.73 (0.96)
35mm	15	9.93 (0.18)	9.90 (0.21)	10	6.60 (2.50)	5.40 (2.27)
		35 months, TAS			***28 months, QLD***	
Control[b] 35mm	23	9.88 (0.33)	9.64 (0.76)		(not provided)	3.08 (2.97)

[a]Values represent means while figures in parentheses represent one standard deviation.
[b]Control samples are untreated *E. nitens*, 120 x 90 x 35 mm. Five control samples of untreated *E. nitens* at 19 mm and 25 mm were only recently installed at both sites and comparative data are not yet available for these thicknesses.

specimens for the field trial effectively exposed the less well-treated core to direct fungal attack and this was

more important for the thicker samples. Compared to pine controls of the same sample thickness, the 35 mm thick samples are still performing better at both sites, and the fact that thicker dimensioned boards are underperforming reinforces that the treatment is having some positive effect overall.

6 – CONCLUSION

The results from the laboratory and field trials varied, but suggest that SC-CO treatment provided some level of protection and further research is warranted. It is important to note that the treatment process was designed for treating spruce and may not have provided sufficient time for the biocides to diffuse into the wood. Further tests are planned to deliver higher retentions of a biocide that would be more appropriate for protecting this species, including an insecticide and a chemical that will react as a tracer so that penetration through the cross section is able to be determined.

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