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Articles

THE INFLUENCE OF DIFFERENT IMPREGNATION FACTORS ON MECHANICAL PROPERTIES OF SILICA SOL-MODIFIED *POPULUS TOMENTOSA*

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Abstract. The low density of many fast-growing plantation species results in poor mechanical and flexural properties that limit their usefulness. Supplemental impregnation may represent one method for improving wood properties to create new applications for these materials. The potential for using varying silica sol impregnation processes to improve hardness and flexural properties was investigated on plantation-grown *Populus tomentosa*. Silica sol-gel impregnation resulted in significant improvements in hardness, MOR, and MOE. The results suggest that these supplemental processes have the potential to create modified woods with a broader range of potential applications.

Keywords: *Populus tomentosa*, fast-growing wood, silica sol, hardness, MOE, MOR.

INTRODUCTION

Because of its high strength-to-weight ratio, outstanding thermal insulation characteristics, ease of processing, carbon neutrality, recyclability, and beauty, wood is frequently used in construction and decorative applications (Brischke 2020). Nevertheless, increasing global demand for wood products will place increasing importance on the development of fast-growing plantations to fill supply shortages. One negative aspect of this material is that the resulting wood tends to have lower density which results in lower physical and mechanical properties that preclude many more valuable uses. As a result, the wood needs to be modified to improve its usefulness (Cheng 2017).

Most of the research in modification has concentrated on Chinese fir (*Cunninghamia lanceolata*), eucalypts, and pines (Yang et al 2020), but fast-growing poplars also have potential in this space. *Populus tomentosa* has a straight bole, fine-grained wood, and one of the shortest rotations for a timber species. Poplars are widely used for construction, furniture, and decorative products (Zhang 2012); however, low density, softness, and poor physical and reduced mechanical properties limit their applications (Lang 2016). Modifying wood properties may represent one approach to increasing the utilization of these resources.

Wood modification can be either physical or chemical. Physical modification densifies the cell structure per unit volume to improve mechanical

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properties (Yang et al 2020), but the improvements via these processes are limited. Gao et al (2019) explored surface compression and heat treatment of poplar. While heat treatment had no appreciable impact on the mechanical properties, the simultaneous use of pressure to densify the wood increased hardness.

Chemical modification can be accomplished by bulking the wood with monomers that polymerize in situ to increase wood properties or by using chemicals that react with cell wall constituents to form covalent bonds with the active components of wood (Lang 2016). Chemical modification can improve mechanical properties, but it can also introduce specialized properties such as hydrophobicity, flame retardancy, or corrosion resistance (Qiu et al 2018). The primary method for chemical modification is to impregnate the wood with a reactant that either reacts at room temperature or the process can be accelerated by heating (Qin et al 2021). The process can occur by simply soaking or fluid uptake can be expedited using combinations of vacuum and pressure to "force" the reactant into the wood (Bao 2021). For example, Wang and Wang (2019) vacuum impregnated fast-growing poplar with unsaturated polyester resin and found that the vacuum level significantly impacted mechanical properties. Similarly, Hou et al (2016) found slight improvements in the wear resistance of modified poplar.

Impregnation with silica-magnesium gel improved the physical and mechanical characteristics of Chinese fir, including hardness flexural properties and compression strength (Zhang et al 2022). Liu et al (2019) used heat treatment and impregnation with a water glass solution to improve the physical and mechanical qualities of poplar. Furfuryl alcohol has also been used to densify the wood surface while also improving dimensional stability and improve mechanical properties (Zheng et al 2022). Liu et al (2021) used a glucose-urea-melamine resin/sodium silicate compound modifier to enhance wood properties.

Silica sol is primarily composed of water and hydroxyl groups, has a low viscosity, small particle size, is nontoxic, and has minimal negative environmental attributes. The silica sol penetrates the wood pores creating a spatial network structure after gelation and drying that can enhance the mechanical and aesthetic qualities of wood (Sun et al 2021).

Lin (2008) found that silica sol impregnation of poplar improved flexural properties but at the expense of reduced compressive strength. Zhang et al (2021) used silica sol to enhance the mechanical properties of wood while also improving phenolic resin durability. Sun et al (2019) varied silica sol solution strength with an ambient pressure treatment to improve the mechanical properties and flame-retardance capabilities of poplar.

Shi et al (2019) impregnated poplar with acid and alkaline silica sols and found that acid silica sol-modified material had higher flexural resistance, whereas alkaline silica sol only improved MOR. Hong et al (2022) found significant improvements in the compressive strength of polyvinyl acetate (PVA)-nano silica sol-impregnated composite wood.

Although soaking treatments can result in high loadings of permeable wood species, combinations of vacuum and pressure can produce higher, more uniform uptakes. Jun et al (2022) found that thermal modification before treatment introduced channels into the wood that facilitated solution uptake, resulting in improved hydrophobicity, dimensional stability, thermal stability, and surface hardness.

The extensive prior research highlights the potential for enhancing the properties of low-density poplar, but a large number of variables complicate process optimization. The objective of our study was to explore a limited number of variables to improve silica sol modification of *P. tomentosa* using an orthogonal test design.

MATERIALS AND METHODS

Materials

Populus tomentosa was provided by Dehua Tubao Decoration New Material Co., Ltd. (Deqing, China). The material was treated for 2 h at

120°C with superheated steam and then dried to 0-3% MC before being cut into eighty 50 mm × 50 mm × 70 mm samples for hardness tests and eighty 20 mm × 20 mm × 300 mm (T × R × L) beams for flexural properties. Silica sol was purchased from Jinan Yinfeng Silicon Products Co., LTD (Jinan, China), and had a solids content of 20%, an average particle size of 8-15 nm, pH of 9.3, and a viscosity of 7.0 MPa. The materials were then allocated to one of nine groups of eight samples each for the hardness and bending tests and conditioned to constant mass at 65% RH and 25°C to an MC of approximately 12%. One group of eight hardness and bending samples was left untreated to serve as a control while the others were assigned to one of eight treatments. The high number of possible variables made complete replication of all possible treatment combinations difficult. Instead, an orthogonal test design was used where variables and treatments were chosen at random among the wide array of choices.

The variables examined were vacuum level (-04, -06, or -0.08 MPa), vacuum time (10, 20, or 30 min), pressure level (0.8, 1.0, or 1.2 MPa), and pressure time (1, 2, or 3 h) (Table 1). Samples were immersed in the silica sol and subjected to the desired vacuum/pressure conditions. The treated materials were conditioned at 25°C and 65% RH for 7 d, then dried at 50°C for 12 h, then 60°C for 12 h, and finally 90°C for 12 h. The material was then reconditioned to constant mass at 65% RH and 25°C. The silica sol treatment altered the EMC to between 9% and 15% MC.

The hardness and flexural elasticity of the two groups of samples were measured by the same superheated steam pretreatment, vacuum pressure impregnation, and dry aging treatment. In the hardness measurement, the MC of the test-modified material was adjusted, and the MC of the nine groups was balanced (close to 12%), which was converted to a value under 12% MC. In MOE and MOR measurements, the flexural modulus and flexural strength of the sample with 12% water content could be calculated when the water content of the sample was in the range of 9-15%.

Hardness Tests

The effect of silica sol treatment on hardness was assessed according to Chinese Standard GB1941-2009 where a 5.64 mm diameter steel indenter was pressed at two points into the radial, tangential, or cross-section of each 50 mm × 50 mm × 70 mm long sample using a universal mechanical testing machine (LX-WN-LL2T, Lixiong, Dongguan, China, UTM) The indenter was pressed at a rate of 3 mm/min or 6 mm/min to a depth of 5.64 mm, and the load required to achieve this depth was recorded (±10 N). After the test, a 20 mm × 20 mm × 20 mm sample was cut from the indented area, weighed, oven-dried

Table 1. Effect of silica sol treatment on the hardness of *Populus tomentosa*.

						Hardness at 12% MC (N)		
No.	Vacuum (min)	Vacuum (MPa)	Time (h)	Pressure (MPa)	MC (%)	End face	Chord plane	Diameter surface
0		Untreated			9.8	4.39 (±0.01)	3.02 (±0.02)	2.96 (±0.02)
1	10	−0.04	1	0.8	10.7	5.64 (±0.02)	4.22 (±0.01)	3.89 (±0.01)
2	10	−0.06	2	1.0	10.8	6.23 (±0.01)	4.80 (±0.02)	4.13 (±0.02)
3	10	−0.08	3	1.2	10.8	6.33 (±0.02)	4.78 (±0.02)	4.36 (±0.01)
4	20	−0.04	2	1.2	11.2	5.85 (±0.01)	4.43 (±0.01)	4.03 (±0.01)
5	20	−0.06	3	0.8	11.4	5.98 (±0.01)	3.90 (±0.02)	4.39 (±0.02)
6	20	−0.08	1	1.0	11.3	6.81 (±0.02)	4.91 (±0.02)	4.22 (±0.02)
7	30	−0.04	3	1.0	10.9	5.44 (±0.01)	4.43 (±0.02)	4.28 (±0.01)
8	30	−0.06	1	1.2	10.8	5.91 (±0.02)	4.14 (±0.01)	4.19 (±0.01)
9	30	−0.08	2	0.8	11.1	6.22 (±0.01)	5.42 (±0.01)	5.04 (±0.02)

Values represent the means of eight replicates per treatment group, whereas values in parentheses represent 1 SD (sample dimensions were 50 mm × 50 mm × 70 mm [T × R × L]).

(105 ± 2°C), and weighed to determine wood MC at the time of testing.

Flexural Testing

The conditioned 20 mm × 20 mm × 300 mm long beams were subjected to third point loading on the UTM according to Chinese Standard GB-1936-1-91 (*test method for MOE*) and GB1936-2-91 (*test method for MOR of wood*). The load was applied continuously at the center span of each beam using a 30 mm diameter load head at a rate of 3 mm/min. Load/deflection was continuously recorded as a failure. The linear portion of the load-deflection curve was used to calculate MOE, while the load at failure vs the beam dimension at that point was used to calculate MOR. The moisture content at the time of testing was determined by cutting a 20 mm cube from the failure zone of each beam. The section was weighed, oven-dried (105 ± 2°C), and weighed again to determine MC. These data were used to correct for slight moisture variations in individual beams.

Statistics

Statistical Package for the Social Sciences (SPSS) was used for auxiliary experimental design, data analysis and to generate the L16(44) orthogonal table to investigate the effects of prevacuum time (A), prevacuum pressure (B), impregnation pressure (C), and impregnation time (D), four influencing factors. The experimental conditions producing the best-modified wood were obtained by using the variance analysis and the range analysis models of SPSS.

The data were subjected to an analysis of variance to determine whether differences between the treatments were significant ($p < 0.1$).

RESULTS AND DISCUSSION

End surface hardness of pretreated and impregnated *P. tomentosa*-modified material increased by 24-55%; chord surface hardness increased by 29-79%; and radial hardness increased by 31-70%, indicating that silicon sol impregnation improved hardness *on all wood surfaces* (Table 1). Improved hardness might reflect the reinforcement of the wood cell wall or filling voids as well as physical, chemical, and mechanical interactions due to bonding.

The bending strength of the modified wood after pretreatment and impregnation increased by 23-70%; while MOE improved by 22-57% (Table 2). Increased MOR and MOE suggest that the silica sol not only bulks the wood but also interacts with the wood cell walls to improve properties. One interesting observation was that MOR and MOE both increased with the initial treatment and then declined with further pressure increases. In the increase of impregnation pressure, the weight gain rate of composite wood also increased, so the elastic modulus also increased.

Table 2. MOR and MOE of untreated and silica sol-modified *Populus tomentosa*.

No.	Vacuum (min)	Vacuum (MPa)	Time (h)	Pressure (MPa)	MC (%)	MOR (MPa)	MOE (GPa)
0		Untreated			8.9	65.7	7.29
1	10	−0.04	1	0.8	9.8	97.6	10.03
2	10	−0.06	2	1.0	9.6	95.2	9.43
3	10	−0.08	3	1.2	10.2	111.9	11.39
4	20	−0.04	2	1.2	11.3	99.9	10.15
5	20	−0.06	3	0.8	10.5	108.8	11.44
6	20	−0.08	1	1.0	9.9	103.6	10.41
7	30	−0.04	3	1.0	10.5	95.2	10.11
8	30	−0.06	1	1.2	10.6	95.5	9.99
9	30	−0.08	2	0.8	11.1	81.1	8.92

Values represent the means of eight replicates per treatment, whereas figures in parentheses represent 1 SD (sample dimensions were 20 mm × 20 mm × 300 mm [T × R × L]).

Table 3. Variance analysis and significance test of the hardness of modified *Populus*.

Properties	Factor	Sum of squares	DF	*F* value	Critical value	Pr > F
End hardness (N)	Prevacuum time	0.193	2	2.573	19	
	Prevacuum pressure	0.984	2	13.120	19	
	Immersion time	0.075	2	1.000	19	
	Immersion pressure	0.069	2	0.920	19	
	error	0.07	2			
Flat hardness (N)	Prevacuum time	0.101	2	1.000	19	
	Prevacuum pressure	1.037	2	10.267	19	
	Immersion time	0.478	2	4.733	19	
	Immersion pressure	0.113	2	1.119	19	
	error	0.10	2			
Edge hardness (N)	Prevacuum time	0.233	2	2.044	19	
	Prevacuum pressure	0.345	2	3.026	19	
	Immersion time	0.152	2	1.333	19	
	Immersion pressure	0.114	2	1.000	19	
	error	0.11	2			

Table 4. Variance analysis and significance test of designed parameters on bending strength and MOE of modified *Populus tomentosa*.

Properties	Factor	Sum of squares	DF	*F* value	*F* critical value	Pr > F
MOR (MPa)	Prevacuum time	308.936	2	39.801	19	*
	Prevacuum pressure	7.762	2	1.000	19	
	Immersion time	262.776	2	33.854	19	*
	Immersion pressure	67.909	2	8.749	19	
	error	7.76	2			
MOE (GPa)	Prevacuum time	1.506	2	25.525	19	*
	Prevacuum pressure	0.059	2	1.000	19	
	Immersion time	3.304	2	56.000	19	*
	Immersion pressure	0.443	2	7.508	19	
	error	0.06	2			

* - significant effect.

While higher pressures increased solution uptake, they could also be associated with increased damage to the wood thereby reducing bending strength.

The analysis of variance examining the effects of prevacuum time, prevacuum pressure, impregnation time, and impregnation pressure on mechanical properties showed that prevacuum pressure affected the end surface and chord surface hardness of the modified *P. tomentosa* (Table 3). The hardness of the radial surface was not significantly affected.

Prevacuum time and immersion time significantly affected the MOR and MOE of the modified *P. tomentosa*. Although both treatment elements were significant, prevacuum time appeared to have a greater effect on flexural properties.

Optimal Modification Process of *P. tomentosa*

Optimum hardness was obtained when the modification parameters were 30 min prevacuum pressure at −0.08 MPa, impregnation for 2 h, and an impregnation pressure of 1 MPa. Optimum improvement in flexural properties was found with a 20 min prevacuum time at −0.06 MPa, and a 3 h impregnation at 1.2 MPa (Table 4). The use of orthogonal comparisons allowed us to reduce the number of variables needed to identify the processes that produced the most improvement in physical and mechanical properties.

CONCLUSIONS

The use of vacuum/pressure treatment with silica sol significantly improved the hardness and

flexural properties of *P. tomentosa* although some reductions in flexural properties were noted with some treatments. The use of an orthogonal comparisons approach allowed us to reduce the number of variables examined while still optimizing property improvements within the parameters examined. The results illustrate the potential for improving the properties of fast-growing, low-density plantation *P. tomentosa*.

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PHYTIC ACID FOR DUAL WOOD PROTECTION AGAINST FUNGI AND FIRE

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Abstract. Phytic acid (PA) is a natural compound derived from plant seeds and cereals with excellent antifungal properties and fire resistance. However, the potential of PA for dual wood protection is yet to be reported. This study investigated the antifungal properties and fire performance of PA for wood protection. The antifungal properties of PA against common wood-decaying fungi, including two white-rot fungi, *Trametes versicolor* (*T.v.*) and *Irpex lacteus* (*I.l.*), and two brown-rot fungi, *Gloeophyllum trabeum* (*G.t.*) and *Rhodonia placenta* (*R.p.*), were studied for both in vitro and in vivo tests. The thermal stability (pine and polar) and fire resistance (pine) of wood samples treated with different concentrations of PA by vacuum impregnation were also evaluated. For the in vitro test, PA almost fully inhibited the growth of three of the four fungi tested at a PA concentration of 0.25 wt% except for fungus *R.p.*, which was less sensitive to PA and could still grow at 4 wt% PA. The in vivo durability test results showed that PA significantly improved the fungal resistance of both pine and poplar wood blocks for the brown-rot and white-rot fungi, respectively, as shown by lower mass losses of 5-25% compared with the control group's 25-45%. The results from thermogravimetric analysis under both air and nitrogen indicated that PA increased the thermal stability of both pine and poplar samples, which was further confirmed by the results from the mass loss calorimeter. The peak heat release rate and the total heat release rate of 10 wt% PA-treated samples were decreased by 39% and 48%, respectively, at 148 kW/m^2 and 34.6 MJ/m^2 compared with the control, whereas the residual mass increased by 137% at 48.4%. Overall, this research demonstrates the potential of using PA to improve both fungal resistance and fire performance of wood products.

Keywords: Phytic acid, dual protection, durability, bio-based wood preservative, fire performance.

INTRODUCTION

As one of the oldest building materials, wood is used worldwide for construction and indoor decoration due to its aesthetics, strength, thermal protection, and environmental benefits. Moreover, the carbon storage potential of wood makes it a promising material to help achieve the carbon neutrality goal. However, wood is susceptible to both fungal deterioration and fire attack (USDA FPL 2021), which lead to structural failures and pose potential safety concerns. For example, fungal damage on wood can reduce its strength (Lepage et al 2022) while the inherent combustible

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nature of wood-based structures can contribute to fire growth (Thomas et al 2021). There is also an increased risk of wildfire events due to climate change in the wildland-urban interface area where fire retardant treatment of wood structures is needed. Therefore, it is imperative to develop approaches that provide dual protection against fungal decay and fire and extend the service life of wood.

There has been extensive research on imparting both durability and fire retardancy to wood for outdoor applications using combined treatments (Marney and Russell 2008). These technologies generally include modifying an existing preservative with a fire retardant (Baysal 2002), modifying wood using common fire retardants with biocidal activities (Lewin 1997; Lee et al 2000), fixing conventional preservatives that have good fire retardance into wood (Tsunoda 2001; Kartal et al 2004; Baysal et al 2006), and forming wood-inorganic composites (Yamaguchi 2003). Among these technologies, boron compounds have been most commonly used due to their dual functionalities but the leaching issue of boron remains (Marney and Russell 2008). A more recent study investigated the decay resistance and fire performance of two quaternary ammonia compounds, didecyl dimethyl ammonium chloride (DDAC) and didecyl dimethyl ammonium tetrafluoroborate (DBF), on both solid wood and plywood samples. Although DDAC- and DBF-treated samples showed resistance against the fungi tested, these samples were more flammable and had higher heat release rates (HRRs) than the controls (Terzi et al 2011). Similar results were also reported with lauric arginate-treated wood (Alorbu et al 2021) and laccase-assisted enzymatic grafting of wood with kraft lignin and sulfonated lignin (Bolaño et al 2021).

Phytic acid (PA) is a bio-based compound that can be found in grains and cereals (Kalali et al 2019). It has excellent metal-chelating properties (Zhang et al 2013) and a high phosphorus content (ca. 28 wt% to its molecular weight) (Costes et al 2017). These properties have allowed PA to be used for different applications, such as natural antimicrobials for food preservation and potential fire retardants in textiles or wood composites (Leng et al 2022; Zhang et al 2023). PA has been demonstrated to improve the bio-control efficacy of *Rhodotorula mucilaginosa* against postharvest gray mold spoilage and natural spoilage of strawberries (Zhang et al 2013) and inhibited the growth of pathogenic fungi, eg, *Fusarium oxysporum* (Li et al 2023). It also inhibited spore germination and vegetative cell growth of *Clostridium perfringens* (type A), which is one of the most widely distributed pathogenic microorganisms (Bloot et al 2022). In terms of fire retardancy, there has been extensive research on combining PA with metal ions or other chemicals to improve the fire performance of wood and wood composites. For example, PA chelating with metal ions, such as Cu, Fe, Zn, and Mg, has shown improved fire performance and reduced smoke release for loblolly pine (*Pinus taeda*) (Zhang et al 2022). Spraying PA solution along with sodium silicate on wood particles increased the fire retardance of wood composites without significantly affecting the key mechanical properties (Lin et al 2023). Moreover, unlike traditional halogenated fire retardants that can affect human health and the environment, PA could be a nontoxic and environmentally friendly alternative to improve the fire performance of wood while preventing fungal deterioration.

However, no studies have been found investigating both the antimicrobial properties and fire resistance of PA-treated wood. Based on the encouraging results from our preliminary study (Liang et al 2023), this research first investigated the antifungal properties of PA against common wood-decaying fungi, including two white-rot fungi, *Trametes versicolor* (*T.v.*) and *Irpex lacteus* (*I.l.*), and two brown-rot fungi, *Gloeophyllum trabeum* (*G.t.*) and *Rhodonia placenta* (*R.p.*), using both in vitro and in vivo assays. Second, the fire resistance of wood blocks treated with different PA concentrations was evaluated using thermogravimetric analysis (TGA) and mass loss calorimeter (MLC).

MATERIALS AND METHODS

Materials

PA (50 wt% in water, Tokyo Chemical Industry Co., Ltd., Portland, OR), malt extract (Oxoid Ltd., Lowell, MA), yeast extract (Oxoid Ltd., Lowell, MA), and agar (Fisher Bioreagents, Pittsburgh, PA) were purchased from Thermo Fisher Scientific (Shelton, CT). Two white-rot fungi, *Trametes versicolor* (Linnaeus: Fries) *Lloyd*, (ATCC#42462, *T.v.*), and *Irpex lacteus (Fr.) Fr.* (ATCC#11245, *I.l.*), and two brown-rot fungi, *Gloeophyllum trabeum* (Madison 617/ATCC 11539, *G.t.*), and *Rhodonia placenta* (*Fr.*) (ATCC#11538, *R.p.*), were purchased from ATTC and used for both in vitro and in vivo tests.

Softwood (loblolly pine [denoted as Pine], *Pinus taeda* L., 488 ± 23 kg/m^3) was donated by Weyerhaeuser, Bruce, MS, and hardwood (yellow poplar [denoted as Poplar], *Liriodendron tulipifera* L., 484 ± 19 kg/m^3) was purchased from a local store, Moscow Building Supply, Moscow, ID. Wood blocks (14 mm × 14 mm × 14 mm, $L \times T \times R$) of both pine and poplar were used for PA treatment and durability test against the brown-rot and white-rot fungi, respectively. Pine wood blocks with dimensions of 100 mm × 100 mm × 10 mm ($L \times T \times R$) were further used for the fire performance test by MLC. All wood samples were defect-free and prepared based on standard AWPA E10 (AWPA 2017). The wood samples without treatments or with DI water treatments were used as controls, depending on the testing specified below.

In Vitro Antifungal Properties of PA

The in vitro antifungal properties of PA were studied by measuring the growth rates of four common wood-decaying fungi (*T.v.*, *I.l.*, *G.t.*, and *R.p.*) in PA-amended malt agar media and the results were compared with those of the control (Cai and Kuo 2022). Specifically, the growth medium of the control group was prepared by mixing and autoclaving 2.0 wt% malt extract, 1.2 wt% agar, and 0.2 wt% yeast in distilled water. PA-amended (0.125-4 wt%) malt agar media were obtained by adding different amounts of PA solutions into the sterilized control malt-agar solution. Subsequently, 20 mL of either the control or PA-amended culture media was cast into a Petri dish (90 mm diameter) and solidified in a biosafety hood. A 5 mm diameter mycelial plug was cut from the edge of an actively growing fungal colony and placed upside down at the center of the growth media. The inoculated plates were incubated at 25°C and 75% RH for 2 wk. Three replicates were prepared for each treatment. Finally, the growth of each fungus in the plates was observed and photographed every 2 d. The fungal growth area was captured using the ImageJ software and the fungal growth rate was calculated by Eq 1

$$Fungi\ growth\ rate\ (\%) = \frac{A_i - A_0}{A} \times 100 \quad (1)$$

where A_0, A_i, and A are the measured areas of the initial fungal plugs, the area where mycelium covered on the growth medium on Day i (i = 2, 4, 6, …, 14), and the area of the Petri dish used for the study, respectively.

PA Treatment on Wood Samples

The wood samples for the fungal resistance and fire performance tests were conditioned at 60°C for 48 h or until the consistent mass was recorded ($m_{trt.1}$). For the durability test, the wood blocks were vacuum impregnated with either DI water (as a control group) or PA solutions (2.5-10 wt% at an interval of 2.5 wt%) for 30 min and were kept in treating solutions for 12 h. The samples were reconditioned at 60°C until consistent weight ($m_{trt.2}$) was recorded. The mass gain after treatment was obtained following Eq 2:

$$\text{Mass gain}\ (\%) = \frac{m_{trt.2} - m_{trt.1}}{m_{trt.1}} \times 100 \quad (2)$$

where $m_{trt.1.}$ and $m_{trt.2}$ are the mass of the conditioned wood blocks before and after vacuum (or vacuum-pressure) impregnation, respectively.

Similarly, PA-treated samples for the fire performance test were vacuum-impregnated with 5 wt% and 10 wt% PA aqueous solutions for 2 h, followed by pressure treatment (827 kPa) for 1 h, whereas samples without treatment were used as the control group in the MLC test.

The treated samples were then conditioned at 60°C until a consistent weight before TGA and MLC tests. The mass gain of the samples was also calculated using Eq 2.

In Vivo Antifungal Properties of PA

The decay resistance of PA-treated wood was evaluated using the soil block method described in standard AWPA E10-16 (AWPA 2017). Before exposure, both treated and untreated wood samples were sterilized by spraying 70% ethanol solution on the surface and drying in the biosafety hood for 1 h. This process was repeated at least twice. Two wood blocks per bottle were placed on the top of the feeder strips containing the actively growing test fungus. At the end of inoculation, all the culture bottles were placed in the environment chamber at 25°C and 75% RH for 8 wk. Pine samples were only exposed to brown-rot fungi, whereas the poplar samples were only exposed to white-rot fungi. Six replicate blocks were prepared for each treatment level for all the testing fungi. The mass loss of the decayed wood was calculated by Eq 3

$$\text{Mass loss } (\%) = \frac{m_{\text{unexpo}} - m_{\text{expo}}}{m_{\text{unexpo}}} \times 100\% \quad (3)$$

where m_{unexpo} and m_{expo} are the mass of wood blocks conditioned at 60°C before and after fungal exposure, respectively.

TGA Analysis

The thermal degradation behaviors of PA-treated wood blocks and control water-treated wood blocks were studied by TGA on a PerkinElmer TGA-7 instrument (Shelton, CT) under both N_2 and air conditions. The samples (around 5-6 mg for each treatment) were heated from 38 to 850°C at a heating rate of 20°C/min under the gas flow of 30 mL/min. Three replicates were tested for each treatment.

Fire Performance Test

The fire resistance of PA-treated wood was evaluated using an MLC (Fire Testing Technology Ltd., East Grinstead, UK) at an irradiance of 50 kW/m^2. The HRR, total heat release (THR), mass changes, and time-to-ignition (TTI) were recorded and analyzed. Samples without treatment were used as a control group. Two replicates were tested for each treatment.

Statistical Analysis

Data (mass gain and mass loss) were statistically analyzed using SAS (9.4, SAS Institute Inc., Cary, NC), which included a normality test, homogeneity of variance test (unequal for all the data in this study) and a nonparametric post hoc analysis approach (Games-Howell test) for the group differences comparison. The results from the analysis were interpreted at a 5% significance level.

RESULTS AND DISCUSSION

In Vitro Antifungal Properties of PA

The growth rates of two white rot, *T.v.* and *I.l.*, and two brown rot, *G.t.* and *R.p.*, in PA-amended malt agar substrates over the 14-d incubation period are shown in Tables 1-4, respectively, and the photos of their overall growing status in the Petri dishes on day 14 are displayed in Fig 1. Overall, the growth of these four fungi was significantly inhibited as the concentration of PA increased and their growth was different across the fungal species. Specifically, in the control

Table 1. Average growth rates of four wood-decaying fungi *T.v.* exposed to different concentrations of phytic acid (PA)-amended malt agar medium over a 14-d incubation period.

Average growth rate of *T.v.* (%)					
	Concentration (wt% PA)				
Day	0	0.125	0.25	0.375	0.5
0	0	0	0	0	0
2	10.59 ± 2.00	1.94 ± 0.07	0	0	0
4	55.25 ± 6.43	3.92 ± 0.80	0	0	0
6	98.65 ± 0.01	6.89 ± 2.41	0	0	0
8	98.65 ± 0.01	9.82 ± 2.84	0	0	0
10	98.65 ± 0.01	13.15 ± 2.92	0	0	0
12	98.65 ± 0.01	16.42 ± 2.54	0	0	0
14	98.65 ± 0.01	18.93 ± 2.96	0	0	0

Table 2. Average growth rates of four wood-decaying fungi *I.l.* exposed to different concentrations of phytic acid (PA)-amended malt agar medium over a 14-d incubation period.

Average growth rate of *I.l.* (%)					
	Concentration (wt% PA)				
Day	0	0.125	0.25	0.375	0.5
0	0	0	0	0	0
2	19.40 ± 1.45	10.56 ± 1.26	0	0	0
4	84.70 ± 3.06	37.33 ± 2.71	0	0	0
6	98.58 ± 0.14	73.47 ± 7.39	0	0	0
8	98.58 ± 0.14	98.56 ± 0.04	0	0	0
10	98.58 ± 0.14	98.56 ± 0.04	0	0	0
12	98.58 ± 0.14	98.56 ± 0.04	0	0	0
14	98.58 ± 0.14	98.56 ± 0.04	0	0	0

group, the mycelium of the two white-rot fungi (*T.v.* and *I.l.*) fully occupied the whole Petri dishes on day 6, whereas those of the two brown rot of *G.t.* and *R.p.* were significantly delayed to days 14 and 10, respectively, which were consistent with previous reports (Alorbu and Cai 2022; Cai and Kuo 2022). In comparison, at a PA concentration of 0.125 wt%, the growth of *T.v.* and *G.t.* was significantly inhibited with growth rates of 19% and 30%, respectively, on day 14. Although this concentration did not significantly change the growth of *I.l.* and *R.p.*, their growth rates were almost completely inhibited as the concentration of PA concentration increased to 0.25 and 4 wt%, respectively. Further increasing of PA concentration to 0.25 wt% also led to little growth

Table 3. Average growth rates of four wood-decaying fungi *G.t.* exposed to different concentrations of phytic acid (PA)-amended malt agar medium over a 14-d incubation period.

Average growth rate of *G.t.* (%)					
	Concentration (wt% PA)				
Day	0	0.125	0.25	0.375	0.5
0	0	0	0	0	0
2	3.70 ± 0.03	1.63 ± 0.27	0	0	0
4	14.49 ± 2.13	5.40 ± 0.88	0	0	0
6	28.60 ± 4.54	8.57 ± 0.31	0.38 ± 0.66	0	0
8	42.66 ± 7.23	12.74 ± 0.62	0.94 ± 1.06	0	0
10	61.47 ± 7.97	18.56 ± 0.95	2.62 ± 2.02	0	0
12	74.78 ± 6.99	24.67 ± 1.13	2.39 ± 2.12	0	0
14	88.80 ± 7.08	30.46 ± 2.22	4.15 ± 4.39	0	0

Table 4. Average growth rates of four wood-decaying fungi *R.p.* exposed to different concentrations of phytic acid (PA)-amended malt agar medium over a 14-d incubation period.

Average growth rate of *R.p.* (%)																	
	Concentration (wt% PA)																
Day	0		0.125		0.25		0.375		0.5		0.625		0.75		1		1.5		2		3		4	
0	0		0		0		0		0		0		0		0		0		0		0	
2	4.64	± 0.31	6.65	± 0.21	6.14	± 0.39	5.20	± 0.40	5.09	± 0.89	4.80	± 0.88	5.96	± 0.25	5.31	± 0.28	3.24	± 0.47	2.70	± 0.14	0		0	
4	30.69	± 1.14	35.69	± 0.56	28.02	± 0.02	21.81	± 2.06	18.57	± 1.43	17.26	± 0.46	17.03	± 0.87	13.07	± 0.66	10.65	± 3.14	8.49	± 1.48	2.83	± 0.44	0.11	± 0.19
6	60.17	± 3.27	64.30	± 3.97	59.63	± 5.10	49.78	± 5.41	42.76	± 1.35	36.94	± 1.58	29.18	± 0.76	24.41	± 1.16	19.67	± 4.28	15.21	± 5.09	4.52	± 0.07	0.60	± 0.52
8	71.58	± 5.12	81.45	± 0.67	76.82	± 5.78	71.10	± 6.75	65.72	± 2.02	58.74	± 3.36	46.99	± 0.56	34.64	± 3.11	29.02	± 7.80	19.44	± 5.14	5.33	± 0.15	0.83	± 0.33
10	95.39	± 5.57	98.69	± 0.06	98.64	± 0.04	98.62	± 0.01	93.01	± 9.54	75.41	± 2.37	64.85	± 5.40	47.50	± 4.66	36.70	± 6.34	32.88	± 7.82	7.83	± 0.55	1.79	± 0.13
12	98.59	± 0.09	98.69	± 0.06	98.64	± 0.04	98.62	± 0.01	98.55	± 0.08	98.64	± 0.05	80.75	± 7.61	64.73	± 2.45	46.29	± 5.84	38.17	± 6.20	10.37	± 0.37	2.19	± 0.21
14	98.59	± 0.09	98.69	± 0.06	98.64	± 0.04	98.62	± 0.01	98.55	± 0.08	98.64	± 0.05	98.61	± 0.05	78.98	± 4.11	55.40	± 4.86	43.50	± 5.92	14.60	± 1.30	2.54	± 0.31

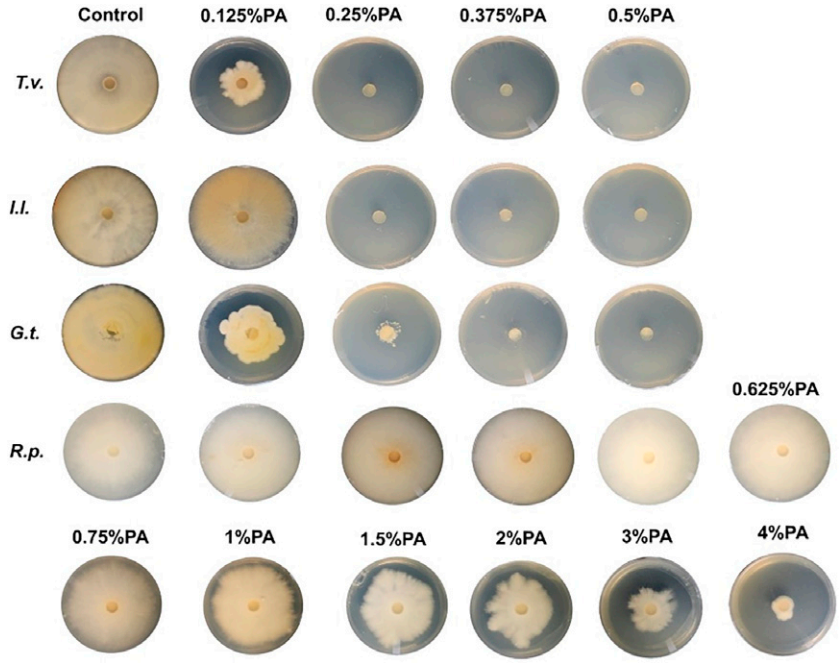

Figure 1. Photos of the wood-decaying fungi on PA amended and unamended plates on day 14.

of *T.v.* and *G.t.*, as shown in Tables 1 and 3, respectively. These results indicated that the white-rot fungi, *T.v.* and *I.l.*, and brown rot fungus *G.t.* used in this study were more sensitive to PA than brown rot fungus *R.p.* Moreover, fungus *R.p.* is the least sensitive to PA and could still grow at the PA concentration of 4 wt%, the highest concentration that the PA-amended malt agar media could solidify under the hood at room conditions.

Mass Gain of PA-Treated Wood Blocks

The mass gain of wood blocks after PA treatment is shown in Fig 2, which generally increases as PA concentration increases. The mass gain of poplar samples was significantly higher (p-value < 0.05) than those of pine samples at all treated concentrations, which is possibly related to the wood anatomical differences (Alorbu and Cai 2022). Moreover, the relatively simple structures of pine samples likely caused more leaching of the extractives in DI water-treated pine control samples than those of poplar samples.

In Vivo Antifungal Properties of PA

The mass loss of decayed samples with and without PA treatment is shown in Fig 3. In general, the PA-treated samples recorded significantly lower (p-value < 0.05) mass loss than those of

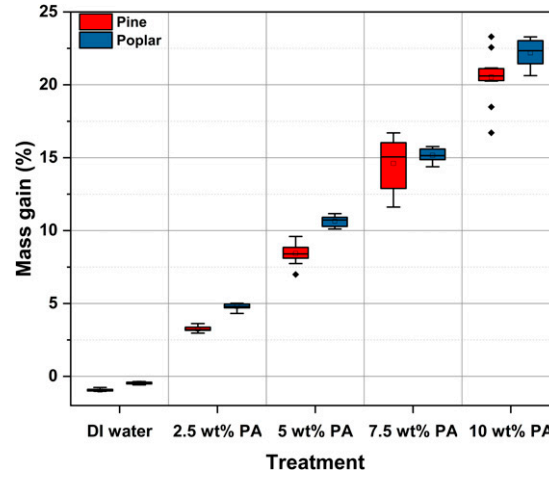

Figure 2. Mass gain of wood blocks after treatment.

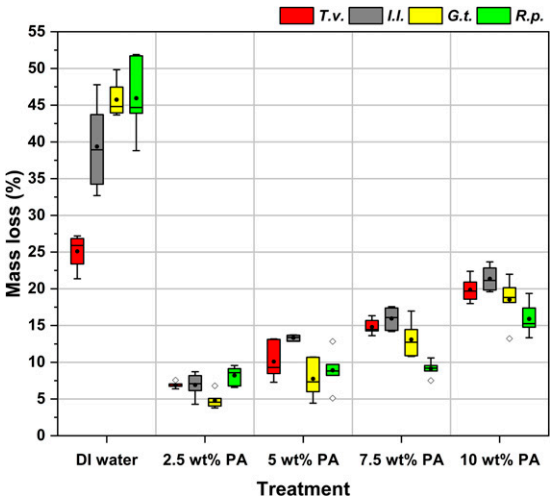

Figure 3. Mass loss of untreated and PA-treated wood blocks without leaching test after 8-wk exposure to white-rot and brown-rot fungi.

DI water-treated samples. For example, the average mass loss of the DI water-treated samples ranges from 25 to 50%, depending on fungal species, whereas the average mass loss of all PA-treated samples is below 25%, indicating PA's promising effectiveness against the common brown-rot and white-rot fungi. Among the PA-treated groups, the mass loss due to decay for each fungus generally increased with an increasing PA concentration (p-value < 0.05) except for fungus *R.p.* This is probably due to the leaching of PA from treated wood blocks during the fungal exposure period. The mass loss of the wood blocks due to fungi exposure was influenced by various factors, including the sample size, fungal exposure time, and chemicals used for the treatment. The results show that the growth of both brown-rot and white-rot fungi on wood has been significantly inhibited by PA treatments. The antifungal mechanism of PA against these four decaying fungi remains unclear but possible reasons could include the disruption of the fungal cell membrane, and inhibits the activity of pathogenesis-related enzymes (Li et al 2023).

TGA Analysis

The pyrolysis and thermal degradation behaviors of the tested wood samples under N_2 and air conditions, respectively, were studied using TGA, as shown in Fig 4, and their average onset temperatures and residues are summarized in Table 5. The TGA thermograms of pine and poplar samples showed no significant differences and generally involved three main stages, including dehydration, active pyrolysis (N_2) or oxidative degradation (air), and passive pyrolysis (N_2) or burnout (air).

Specifically, the dehydration of all the tested wood samples under both N_2 and air was observed below 170°C with less than 5% mass loss. As the temperature increased, all the samples under N_2 experienced active pyrolysis with significant Derivative thermogravimetry (DTG) peaks (Fig 4[a] and [c]). However, the active pyrolysis process for PA-treated samples was advanced (170-450°C) with onset temperatures at ~189-195°C, as compared with that of DI water-treated controls (~300-500°C) at around 310-320°C. Moreover, the onset temperatures of 10% PA-treated wood samples were significantly lower than those of 5% PA-treated, indicating a higher amount of acid could lead to an accelerated pyrolysis process of wood samples (Fu et al 2008). Further increasing of temperature to 800°C resulted in passive pyrolysis of both PA-treated and DI-water-treated controls, which were associated with the decomposition of lignin into cross-linked aromatic charcoal (Yang et al 2006). This observation was also consistent with the nearly zero mass loss rate in their DTG curves (Fig 4[c]). The residues of PA-treated samples were 33-39%, which were significantly higher than those of DI water-treated controls of ~10%.

The weight percentage changes of the samples under air were similar to those of under N_2 conditions before 450°C (Fig 4[b]). As the temperature increased, the samples under air were further oxidized and decomposed, which is evidenced by DTG peaks at ~480°C and above (Fig 4[d]). This process also led to significantly lower residues of the samples as compared with those under N_2 pyrolysis. For example, the residues for DI water-treated pine and poplar controls were nearly 0% (~10% under N_2) while the residues of 5% and 10% PA-treated wood samples were 5% and 10%

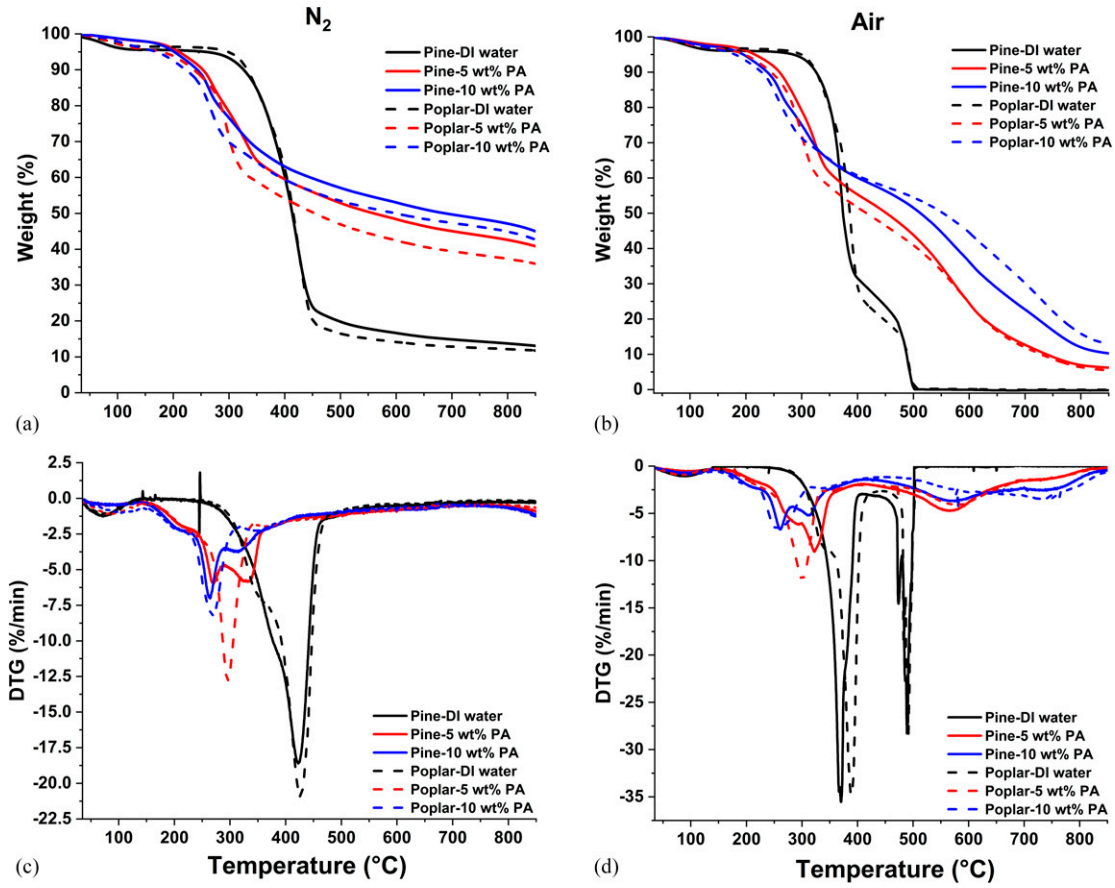

Figure 4. TGA curves of testing samples under (a) N_2 and (b) air; DTG curves of testing samples under (c) N_2 and (d) air.

(~36% under N_2), respectively. Regardless of the purge gas used, PA-treated samples have significantly lower onset temperatures (p-value < 0.05) than those of DI water-treated controls. The improved thermal stability of PA-treated wood samples is likely related to the increased char formation promoted by phosphoric acid from PA, which is less thermally stable and decomposes earlier than cellulose (Daneluti and Matos 2013; Yuan et al 2021). The formation of residual char

Table 5. Onset temperature and residue of pine and poplar samples of the TGA test.

	N_2		Air[a]		
Sample	Onset (°C)	Residue (%)	1st onset (°C)	2nd onset (°C)	Residue (%)
Pine-water control	317 ± 1	12 ± 1	309 ± 5	478 ± 8	0 ± 0
Pine-5 wt% PA	188 ± 5	38 ± 1	192 ± 2	488 ± 13	6 ± 2
Pine-10 wt% PA	178 ± 3	41 ± 1	180 ± 6	479 ± 14	10 ± 4
Poplar-water control	309 ± 3	11 ± 1	307 ± 5	485 ± 5	0 ± 0
Poplar-5 wt% PA	195 ± 4	34 ± 1	201 ± 9	518 ± 5	5 ± 2
Poplar-10 wt% PA	178 ± 3	38 ± 1	187 ± 5	537 ± 18	13 ± 4

[a]For samples exposed to the air, 1st and 2nd onset refers to the initial and 2nd temperatures testing samples start breaking down.

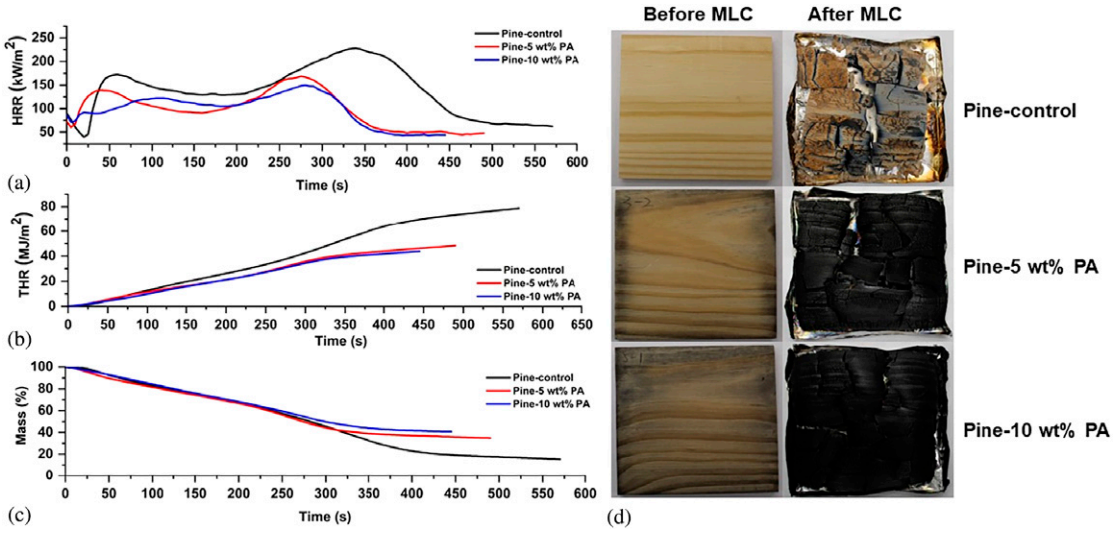

Figure 5. Representative (a) heat release rate (HRR) curves, (b) total heat release (THR) curves, (c) mass curves, and (d) pine samples with different treatment levels before and after the MLC test.

helped reduce the release of flammable gases and heat/mass transfer, contributing to significantly higher (p-value < 0.05) residue at 850°C.

Fire Performance Test

The combustion properties of control wood samples, 5 wt% and 10 wt% PA-treated wood samples were shown in Fig 5. Specifically, in the HRR curves (Fig 5[a]), two peaks were observed in all the testing samples. The first and second peaks correspond to the formation and breakdown of the char layer, respectively (White and Dietenberger 2004; Wang et al 2022). The latter also has a higher HRR value than the former and is noted as peak HRR (pHRR), which helps determine the maximum combustibility and flashover potential of a fire retardant-treated material. For example, the pHRR of the control group is the highest at 241 kW/m^2, whereas those of 5 wt% and 10 wt% PA-treated samples decreased by 26% and 39% to 178 and 148 kW/m^2, respectively. Similarly, the THR(Fig 5[b]) of 5 wt% and 10 wt% PA-treated samples are 47.7 and 34.6 MJ/m^2, respectively, 29% and 48% lower than that of the control at 66.8 MJ/m^2. Moreover, as shown in Fig 5(c), the 5 wt% and 10 wt% PA-treated samples recorded 80% and 137% higher mass residue (36.6% and 48.4%), respectively, than that of the control (20.4%). The increased mass residues of PA-treated wood samples were also evidenced by their improved char formation after burning (Fig 5[d]). In comparison, the residue in the control group was mainly ash. It is also worth noting that the TTI (Table 6) of control wood samples was 21.5 s whereas those of the 5 wt% and 10 wt% PA-treated wood samples were shortened to 7.0 and 5.5 s, respectively. The shortened TTI could be related to the changed chemical structure of PA-treated wood samples, which might have been degraded by the strong PA solution causing the darkening of the surface of wood samples (Shi et al 2018) (Fig 5[d]). Collectively, the shortened ignition time, together with the significantly reduced values in pHRR and THR, and increased

Table 6. Mass Loss Cone data (averaged) of pine-treated samples.

Sample	TTI (s)	pHRR (kW/m^2)	THR (MJ/m^2)	Residue (%)
Pine-control	21.5	241	66.8	20.4
Pine-5 wt% PA	7.0	178	47.7	36.6
Pine-10 wt% PA	5.5	148	34.6	48.4

pHRR refers to the peak HRR value. THR refers to heat release calculated from the start of the test to 2 min after flaming out.

mass residue after flame out indicate PA might help lower the onset decomposition temperature (as shown in TGA analysis above) and catalyze the carbonization of wood compounds, thus facilitating the formation of residual char as insulation and reducing the heat and mass transfer (Yuan et al 2021). Our findings are also consistent with the previous results found in the combined treatment of PA (6 wt%) and uracil on poplar wood samples (Zhang et al 2021).

CONCLUSIONS

The in vitro tests demonstrated the effectiveness of PA in inhibiting the growth of two white-rot fungi, *T.v.* and *I.l.*, and the brown-rot fungus, *G.t.* at a concentration of 0.25 wt%. However, fungus *R.p.* was the least sensitive to PA and continued to grow at a PA concentration of 4 wt%. Soil block tests further evidenced the improved resistance of PA-treated wood blocks to wood-decay fungi with around 5-25% mass loss, compared with the control group's 25-45%. Thermal stability of wood samples under both air and N_2 conditions was significantly improved with higher residue and lower DTG peak after PA treatment. The MLC tests of pine samples showed that PA treatment significantly reduced the pHRR and THR of wood samples from 241.0 and 66.8 kW/m^2 to 147.8 and 34.6 kW/m^2, respectively, and increased the charring from 20.4 to 48.4% during combustion. Our research findings indicated PA provides both antifungal properties and fire retardance to wood products.

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A COMPARATIVE STUDY ON THE BENDING STRENGTH OF EUROPEAN SPRUCE AND FUJIAN CHINESE FIR

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Abstract. This paper compared the bending properties of European spruce (*Picea abies (L.) H. Karsten*) and Chinese fir (*Cunninghamia lanceolata (Lamb.) Hook.*) with differing densities and knot patterns to identify the most appropriate uses for the latter species. As expected, reduced density and increased knot size negatively affected the modulus of elasticity (MOE) and the modulus of rupture (MOR) of both species. The MOEs of European spruce were higher than those of Fujian Chinese fir and were higher in samples without knots. The effect of knots on bending strength was more pronounced in European spruce. The results indicated that Fujian Chinese fir and European spruce could be substituted for each other in some less-demanding structural applications, which helps improve the utilization of the latter species.

Keywords: European spruce (*Picea abies (L.) H. Karsten*), Fujian Chinese fir (*Cunninghamia lanceolata (Lamb.) Hook.*), modulus of elasticity, modulus of rupture, knots, density.

INTRODUCTION

Chinese fir (*Cunninghamia lanceolata (Lamb.) Hook.*) has the advantages of fast growth, good mechanical properties, and resistance to fungal attack. Acetone, methanol, and ethyl acetate extracts of Chinese fir heartwood had inhibitory effects on the growth of the white-rot fungus (*Trametes versicolor* (L. ex Fr.) Quél.) and the brown-rot fungus *Gloeophyllum trabeum* (Pers. ex Fr.) Murr (Yan et al 2019; Zhang et al 2020; Liu et al 2023). Chinese fir is widely used for engineered wood products as well as in wood-frame buildings (Longuetaud et al 2022; Ponzecchi et al 2022; Shen et al 2022). Chinese fir may also have potential applications in mass timber buildings (Zhang and Qiu 2023).

Chinese fir is one of the most important plantation trees in China in terms of planting area and basal volume (Löf et al 2023; Yu et al 2023). However, the boles of this species have many knots and low mechanical properties making it difficult to use in some high-grade furniture and decorative applications (Zarna et al 2023; Zong et al 2023). As a result, imported European spruce is widely used in construction, infrastructure, and other applications (Jian et al 2023). However, it is difficult to control the quality of the material source since the timber is imported in the sawn form (Walsh-Korb and Avérous 2019; Li et al 2021). Substitution of home-grown Chinese fir in the construction would allow for tighter quality control and increase the utilization of this resource (Kumar et al 2016; Meijer et al 2021).

The physical and mechanical properties of wood vary widely among species and are critical for proper utilization (Reynolds et al 2016; Zhan et al 2019; Palizi and Toufigh 2022; Martineau et al 2023). Mechanical properties of wood are closely related to density, knots, and other factors and vary widely between species (De Santis and Fragiacomo 2021). The main focus with Chinese fir has been on growth rates, with less concern for wood quality, including density and knot sizes (Zhao et al 2021).

Zhong et al (2011) showed that the process of compression in spruce included elastic, yield, and compaction stages. They also showed that the

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axial compression failure mode of spruce was mostly from buckling and folding of wood fiber, whereas the radial or tangential failure modes were mostly due to slippage and layering of wood fiber. The axial compressive yield strength of spruce was about nine times that of radial and tangential yield strength which were nearly equal. Yuka et al (2018) showed that wood density significantly impacted mechanical properties, whereas the shape and deformation of wood cells also played a key role in the structural characteristics. Liu et al (2007) examined the modulus of elasticity (MOE) and the modulus of rupture (MOR) to create a mathematical model to predict the bending performance and fracture mode of black spruce. Fischer et al (2016) established the relationship between MOE and MOR with tree characteristics, including altitude, latitude, tree age, and density by conducting tests on spruce trees at 17 sites in eastern Norway. This model evaluated wood performance but also predicted the impact of silvicultural practices on wood quality.

Several standards 《GB/T 26899-2011 Structural glued laminated timber》, 《GB/T 29897—2013 Visual grading rules for dimension lumber in light wood frame construction》, and 《LY/T 2228—2013 Finger jointed structural dimension lumber in light wood frame construction》 all relate the quality and grade of structural sawn timber to mechanical properties and engineering wood uses. European spruce (*Picea abies*) and Chinese fir (*Cunninghamia lanceolata*) structural sawn timber need to meet the requirements of the above standards.

The purpose of this paper was to compare the properties of Chinese fir with those of European spruce for structural applications in relation to variations in density and knots.

MATERIALS AND METHODS

Materials

Kiln-dried European (Norway) spruce was obtained from Sweden through Suzhou Kunlun Green Building Wood Structure Technology Co., Ltd (Table 1). The lumber was sawn to 17 mm × 38 mm × 330 mm long and conditioned to 12-15% MC.

Chinese fir was cut from 20- to 30-yr-old second rotation trees (180-250 mm diameter at breast height) obtained from the Shengsheng Wood Industry Co., Ltd. of Shunchang County, Fujian Province, China (Table 1). The lumber was sawn into 40 mm × 140 mm × 4000 mm sections that were kiln-dried at low temperatures from the original 60-80% MC to 12-15% MC before being further processed into 17 mm × 38 mm × 330 mm long beams.

The European spruce samples were sorted to produce 20 specimens in three density ranges (Table 2). The Chinese fir samples were similarly sorted to produce four density ranges each containing 20 specimens. Four groups were sorted for Chinese fir to account for the slightly wider density range for this species. A total of 500 specimens were examined. In both cases, only specimens with a slope of grain less than 15°, excluding the area around any knots, were selected. The effect of the knot area on flexural properties was assessed in a separate test where beams were cut so that knots of different diameters were positioned to be within 100 mm on either side of where the load would be applied.

The beams were cut so that they contained a single knot with the dimensions divided into four groups: ($d < 10$ mm, $d =$ 10-20 mm, $d =$ 20-30 mm, $d > 30$ mm).

Bending Test

The specimens were loaded on the narrow face to failure in a third-point bending on an Instron 3369 microcomputer electronic universal mechanical testing machine according to procedures described

Table 1. Range and average density of European spruce and Chinese fir specimens used in the experiments.

	Specimens	Density range (g/cm^3)	Average density (g/cm^3)
European spruce	60	0.30 -0.40	0.36 g/cm^3
Chinese fir	80	0.30-0.50	0.40 g/cm^3

Table 2. Treatments used to assess the effects of density and knots on flexural properties of European spruce or Chinese fir.[a]

Treatment group	European spruce	Chinese fir
Density 1	0.344-0.361 g/cm^3	0.327-0.382 g/cm^3
Density 2	0.370-0.382 g/cm^3	0.391-0.430 g/cm^3
Density 3	0.391-0.445 g/cm^3	0.446-0.485 g/cm^3
Density 4	—	0.494-0.543 g/cm^3
Knot 1	<10 mm diameter	<10 mm diameter
Knot 2	10-20 mm diameter	10-20 mm diameter
Knot 3	20-30 mm diameter	20-30 mm diameter
Knot 4	>30 mm diameter	>30 mm diameter

[a]Each treatment was replicated on 20 beams per wood species.

in Chinese Standard GB/T 50329-2012 (standard for test methods of timber structures, GB/T 1936.1-2009 test methods for bending strength of timber) with a span to the depth ratio of 16.5 and a loading rate of 5 mm/min. Displacement and load were continuously recorded. According to the standard GB/T 1936.1-2009, the effects of shear were ignored. Each test took 2-3 min.

The linear portion of the load/deflection curve was used to calculate MOE, whereas the ultimate load was used to calculate MOR:

$$\mathrm{MOE}=\Delta PL^3/(4bh^3\Delta y) \quad (1)$$

$$\mathrm{MOR}=3P_{\max}L/(2bh^2) \quad (2)$$

where L is the span, b is the width of the specimen, h is the height of the specimen, y is the stress-strain diagram value, P is the load for which stress-strain diagram, and $P_{\max}$ is the breaking load.

RESULTS AND DISCUSSION

Effect of Density on Bending Resistance of European Spruce and Fujian Chinese Fir

Effect of density on bending resistance of European spruce. The density range of European spruce was relatively narrow, ranging from 0.3 to 0.4 g/cm^3, whereas that of Fujian Chinese fir was slightly larger, ranging from 0.3 to 0.5 g/cm^3 (Table 1). For this reason, four density groups were used for Chinese fir instead of the three used for European spruce.

MOEs of the three groups of European spruce were 10.41, 12.42, and 13.96 GPa for the low, medium, and high-density groups, respectively, and were similar to commercial values with fairly low coefficients of variation (COV) of 8%, 10%, and 11%, respectively (Table 3). MORs of the same material were 35.85, 40.88, and 42.80 MPa, with COVs of 8%, 10%, and 9%, respectively. The lower COVs likely reflected the fact that the timber was further segregated from the general population. As the density is generally well-correlated with MOE and MOR, this would tend to narrow the variation in a given group. The data were subjected to an analysis of variance and tests of normality to identify treatments that differed significantly from one another ($\alpha = 0.01$) (Table 3).

As can be seen from Fig 1, the load was applied to European spruce from the narrow side, and failures tended to be in tension. The bending strength of the European spruce groups was divided by the average density of each group to more closely examine the relationships between these two properties. The results still indicated that denser materials still retained more capacity (99.58 MPa[cm^3]/g, 113.56 MPa[cm^3]/g, and 118.89 MPa[cm^3]/g for the three groups). These results were analyzed using the Min-Max normalization method. Bending strength values of the above three groups of European spruce and Chinese fir under unit density were linearly transformed, and the resulting values were 0, 0.06, and 0.09, respectively. The results suggest that Chinese fir has slightly higher bending strength per unit of mass than European spruce. These differences may be reflected in the anatomical arrangement of the cells.

Table 3. Effect of density on MOR and MOE of European spruce.[a]

Density range (g/cm^3)	MOE (GPa)	MOR (MPa)
0.344-0.361	10.41 ± 0.81	35.85 ± 2.85
0.370-0.382	12.42 ± 1.03**	40.88 ± 4.26**
0.391-0.445	13.96 ± 1.54**	42.79 ± 3.68**

[a]Values represent averages of 20 species per group. Values followed by ** signify very significant differences ($0.01 \le \alpha < 0.05$) between each group.

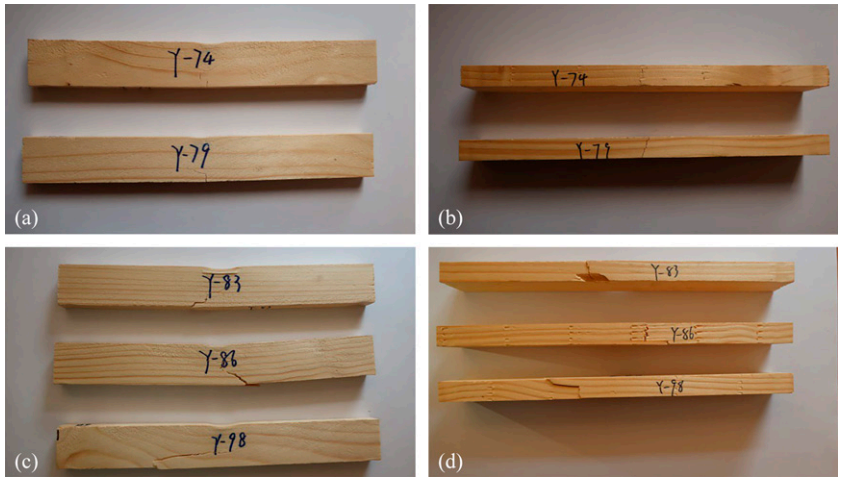

Figure 1. Examples of failure modes of knot-free European spruce subjected to loading to failure in third-point bending.

Effect of density on bending resistance of Fujian Chinese fir. MOEs of the four density groups of Chinese fir were 6.11, 6.20, 6.05, and 6.57 MPa (Table 4). These values were similar to previous reports for this species. However, variations were much higher with COVs of 14%, 21%, 22%, and 22%, respectively. Density was not as well-correlated with MOE as with European spruce.

MORs of the four density groups of Chinese fir were 84.12, 102.17, 108.56, and 126.11 MPa, with COVs of 8%, 8%, 10%, and 14%, respectively. There was a strong correlation between density and MOR with this species. As with the European spruce, the samples primarily failed in tension (Fig 2).

MORs of three groups of European spruce were divided by the average density (35.85 MPa/0.36 g/cm^3, 40.88 MPa/0.36 g/cm^3, and 42.80 MPa/0.36 g/cm^3) to obtain MORs on a unit mass basis of 99.58 MPa(cm^3)/g, 113.56 MPa(cm^3)/g, and 118.89 MPa(cm^3)/g, respectively. The Chinese fir data were similarly divided by average density (84.12 MPa/0.4 g/cm^3, 102.17 MPa/0.4 g/cm^3, 108.56 MPa/0.4 g/cm^3, and 126.11 MPa/0.4 g/cm^3) to produce MORs per unit mass of 210.30 MPa(cm^3)/g, 255.43 MPa(cm^3)/g, 271.40 MPa(cm^3)/g, and 315.28 MPa(cm^3)/g, respectively. The above results were analyzed using the Min-Max normalization method, and the MORs/unit of mass of the above seven groups of European spruce and Fujian Chinese fir were linearly transformed, and the results mapped to the range [0-1]. The values suggested that Chinese fir was slightly stronger per unit of mass than European spruce.

Table 4. Effect of density on modulus of elasticity (MOR) and modulus of rupture (MOR) of Fujian Chinese fir.[a]

Density group (g/cm^3)	MOE (GPa)	MOR (MPa)
0.327-0.382	6.11 + 0.87	84.12 + 7.05**
0.391-0.430	6.20 + 1.27	102.17 + 8.67**
0.446-0.485	6.05 + 1.36	108.567 + 11.21**
0.494-0.543	6.57 + 1.44	126.11 + 17.10**

[a]Values represent 20 replicates per group.

Effect of Knots on Flexural Properties

European spruce and Chinese fir specimens with knots both tended to fail in tension around the knots (Figs 3 and 4). This would be a typical mode of failure given that knots present a void in the wood as well as deviations in grain direction that lead to reduced properties. Load/displacement curves tended to be higher for European spruce and tended to have higher failure values (Fig 5).

Effect of knots on bending resistance of European spruce. The European spruce in this study had only a few knots that ranged from 10 to 30 mm in diameter and were mostly sound.

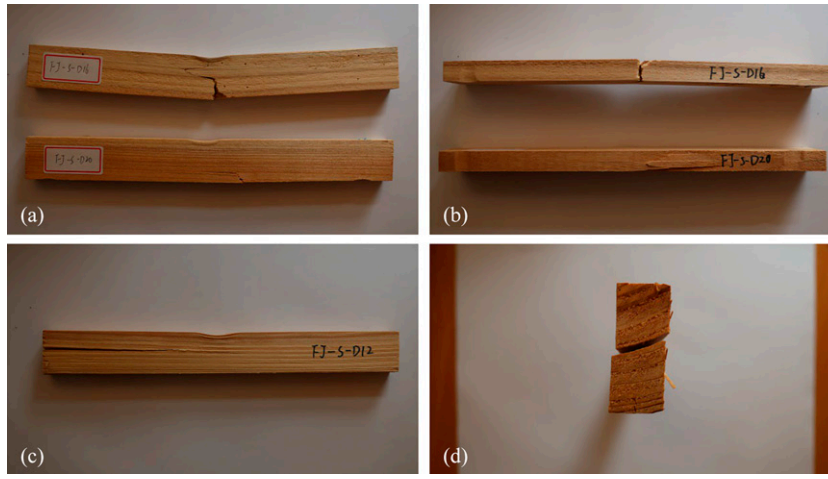

Figure 2. Examples of failure modes of knot-free Fujian Chinese fir subjected to third-point loading.

The MOEs in compression parallel to the grain of four groups of European spruce with different knot sizes were 13.16, 12.95, 12.67, and 11.04 GPa and were similar to the reported values (Table 5 and 6). The COVs were 29%, 30%, 28%, and 37%, which were much higher than COVs for wood.

MORs of three groups of European spruce with different knot sizes were 43.49, 38.27, 36.01, and

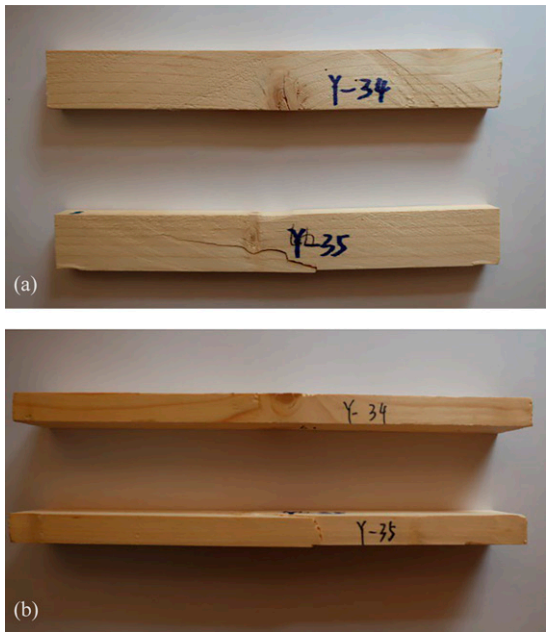

Figure 3. Examples of failure modes on European spruce beams with knots of various sizes subjected to third-point loading.

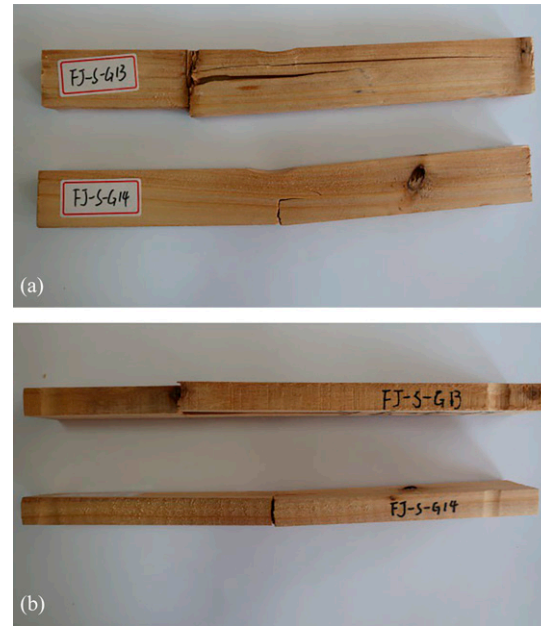

Figure 4. Examples of failure modes of Fujian Chinese fir with knots of varying sizes that were tested to failure in third-point loading.

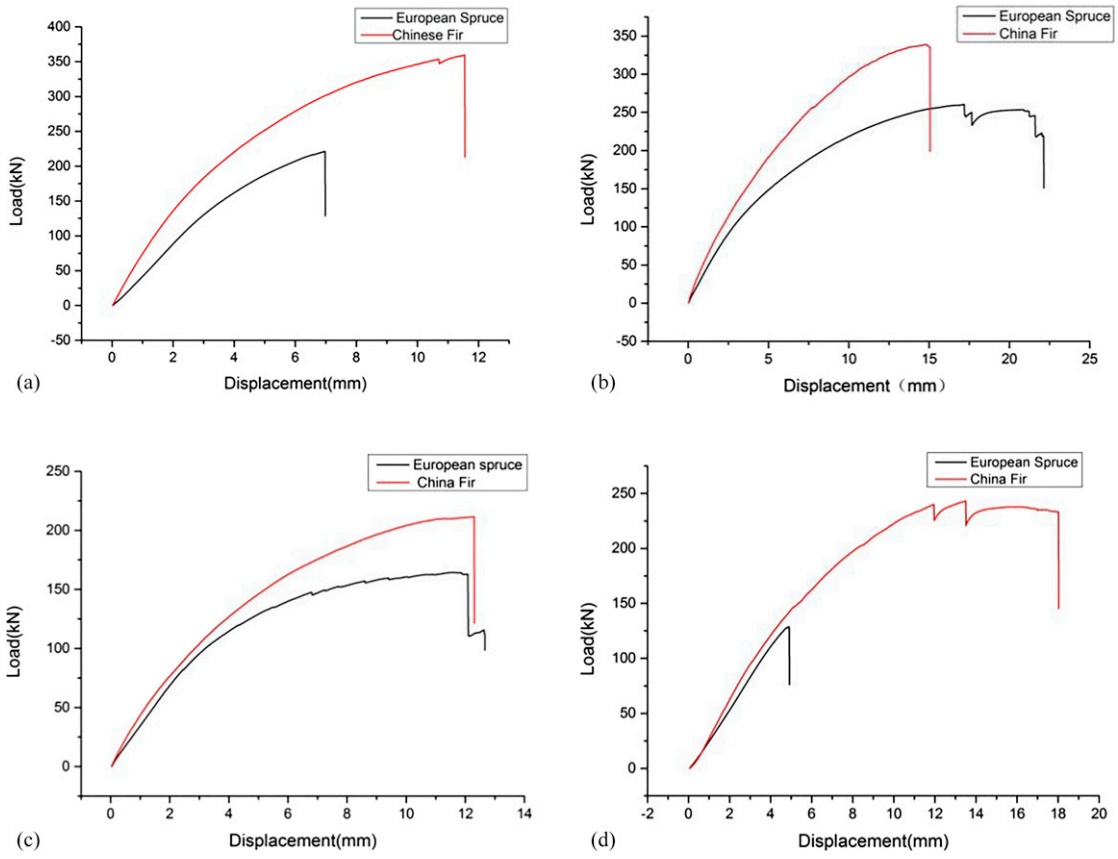

Figure 5. Load/displacement curves of typical European spruce and Chinese fir beams with knot diameters of (a) $d < 10$ mm, (b) $d = 10$-20 mm, (c) $d = 20$-30 mm, and (d) $d > 30$ mm.

28.20 Mpa with COVs of 18%, 16%, 22%, and 35%, respectively. COVs were again higher, reflecting the variability induced by the variable grain around the knots.

MOE and MOR both decreased with increased knot size, although the effects were small for knots less than 20 mm in diameter.

Effect of knots on bending resistance of Fujian Chinese fir. MOEs of the five groups of Fujian Chinese fir with different sizes increased from 6.09 Gpa to 6.24 MPa, 6.39 GPa, and 6.93 GPa, and the COVs were 29%, 36%, 39%, and 25% as the knot size increased. As discussed, these variations were higher than typical values for wood. Although the values were much lower than those for European spruce, it is unclear why they increased with larger knots. It is possible that the density of the Chinese fir knots affected the test results.

MORs of the knot groups of Fujian Chinese fir with different knot sizes declined slightly with increasing knot sizes ranging from 111.99, 115.005, 105.62, and 99.69 Mpa with COVs of 23%, 22%, 21%, and 13%, respectively. As with the MOE values, the COVs tended to be higher than those for clear beams, likely reflecting the variations induced by more variable grain. Unlike European spruce, Chinese fir MORs declined with knot size which would be a more typical response.

As can be seen from Fig 5, Fujian Chinese fir with the same knot sizes had better mechanical

Table 5. Effect of knots on MOE and MOR of European spruce with different knot sizes.

Knot size (mm)	MOE (GPa)[a]	% Reduction	MOR (MPa)[a]	% Reduction
<10	13.16 ± 2.23	10	40.21 ± 3.79	8
10-20	12.95 ± 2.35**	11	38.27 ± 5.61**	12
20-30	12.67 ± 2.64**	13	36.01 ± 8.08**	17
>30	11.04 ± 2.71**	24	28.20 ± 7.50**	35

[a]Values represent the means of 20 samples per group. Values with an asterisk differ significantly from the no knot group at $\alpha = 0.05$.

Table 6. Effect of knot diameter on flexural properties of Fujian Chinese fir.

Knot diameter (mm)	MOE (GPa)	% Reduction	MOR (MPa)[a]	% Reduction
<10	6.09 ± 1.78	0.9	111.99 ± 25.33**	33
10-20	6.29 ± 2.27	2.5	107.68 ± 16.21**	28
20-30	6.39 ± 2.51	4.1	105.62 ± 21.78**	26
>30	6.92 ± 1.71	12.8	99.69 ± 12.63**	19

[a]Values represent the means of 20 samples per knot category. Values with an asterisk differ significantly from the knot-free control ($\alpha = 0.05$).

properties than European spruce although both timbers experienced declined with increased knot size. European spruce tended to experience fracture failures, whereas the Chinese fir exhibited more slippage or shear that allowed some recovery when the load was removed.

CONCLUSIONS

Increased density was associated with increased MOE and MOR of both species, but the effect was more enhanced with Chinese fir. Increased knot diameter was associated with reduced MOE for European spruce but this effect was only found with the larger diameter knots for Chinese fir. MOR was negatively affected by both species.

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COPPER MIGRATION FROM TREATED WOOD GARDEN BOXES INTO SOIL AND VEGETABLE BIOMASS PART I: THE FIRST TWO GROWING SEASONS AFTER INSTALLATION

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Abstract. Pressure-treated wood is a commonly used material for constructing garden boxes and concerns about metal leaching into garden soils and garden vegetables persist among the public. This study describes efforts to quantify copper migration from copper azole-treated garden bed frames into garden soil and vegetable biomass. Two garden bed frames were constructed from copper azole 2 × 12-inch nominal Douglas-fir lumber and two were constructed with untreated Douglas-fir lumber before filling with a mixture of native soil and compost. An assortment of common garden vegetables was planted in identical patterns in each of the beds for two growing seasons. During this 2-yr study, we found no difference in copper concentrations between identical vegetables grown in beds constructed with treated or untreated lumber. After 1 and 2 yr, average copper concentrations in soil 0-25 mm from the bed frames were about 23 ppm and 21 ppm higher than soils in the same location in untreated beds, respectively ($p < 0.05$, Tukey's HSD). Elevated copper levels were not detected in beds constructed with treated lumber at 76-102 inches from the frames or the bed center, indicating that metal migration was limited. This study shows use of treated wood garden beds did not lead to increases in copper concentrations in vegetables grown in those beds. Treated bed materials did lose some copper to garden soil but increases in copper are limited to about 20 ppm immediately next to the treated wood frames and were not detectable at any greater distances from the wood.

Keywords: Metal leaching, bioaccumulation, wood durability, copper azole.

INTRODUCTION

Wood is commonly used in the construction of garden boxes for residential flower beds and vegetable gardens but suffers severe decay risk in this environment due to frequent wetting by irrigation and contact with nutrient-rich soil. Using pressure-treated lumber can improve the lifespan of wooden garden boxes and is economically desirable. However, concerns over chemical contamination of garden soils and vegetables persist among the public.

Some of the concerns arise from the past use of chromated copper arsenate (CCA) as a wood preservative for residential applications and the associated fears of arsenic contamination of vegetable matter. It has been over 20 yr since CCA-treated lumber was voluntarily removed from the residential market in the United States (EPA 2002). Since then, nonarsenical copper-based preservative systems such as copper azole (CA-C), micronized CA-C, and alkaline copper quaternary have been used for residential applications including ground contact applications like the construction of garden boxes. Despite the absence of arsenic in current preservative formulations, many online blogs recommend avoiding pressure-treated wood in garden boxes because of the risk of contaminating produce with hazardous

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chemicals. The United States Department of Agriculture Organic regulations also exclude the use of pressure treated wood in new construction on organic certified land (NOP 2016). This suggests regulators believe the use of treated wood will impact the quality of organic produce.

Excessive copper exposure to plant tissues can be detrimental to plant growth if present at sufficient levels in the growth medium or plant tissues. High levels of heavy metals in vegetables originating from plants grown in contaminated soils are potentially hazardous to human health if accumulated in plant biomass at high levels (Intawongse et al 2006). Given these risks of metal accumulation observed in some hydroponic copper exposure experiments, it is important to assess the impact of treated wood garden boxes on garden soils that come into contact with edible vegetables (Shabbir et al 2020). There is a surprisingly small amount of published scientific investigation into the impacts of pressure-treated wood on garden vegetables specifically. A study done at Oregon State University investigated this topic using CA-C-treated Douglas-fir as a test material (Love et al 2014). This study showed that there was no difference in the metal content of the edible portions of vegetable biomass whether it was grown in a treated or untreated box. There was, however, a significant increase in the copper content of carrot tops sourced from treated boxes as compared with untreated boxes, indicating that there may be the potential for metal exposure for the aboveground portions of carrots.

The risk of vegetable contamination from treated wood has also been assessed in a controlled culture where metals are intentionally introduced into growth media and results vary by plant. Grapevines exposed to copper, chromium, and arsenic in a hydroponic growth medium did not accumulate metals above those of the controls, indicating that the plants regulated metal uptake via their roots (Ko et al 2007). Another study showed that CCA-treated wood sawdust amendment of soil led to increases in CCA metal concentration in the fibrous root portion of beetroot but less so in the large edible portion of the root (Speir et al 1992). Other studies show metal accumulation in plant parts in hydroponic and soil cultures where high copper loadings in the form of soluble copper are added to plant cultures (Shabbir et al 2020). These studies are not representative of the real-world use of treated wood bed material and further investigation of metal accumulation potential in a representative system would be beneficial for uncovering the true impact of treated wood's use in garden boxes.

In this study, metal migration from treated wood used in vegetable garden construction was investigated in a multiyear study. Garden boxes were constructed with CA-C-treated Douglas-fir lumber as well as untreated wood to quantify the migration of copper out of the treated boxes. Copper content in soils and vegetable biomass were measured at equivalent locations and plant types in each garden box. This study summarizes the first 2 yr of soil and vegetable data in an ongoing study.

METHODS

Garden Box Construction and Planting

Two garden box frames were constructed out of untreated or pressure-treated 5.1 × 30.5 cm (2 × 12-inch) nominal Douglas-fir lumber (Fig 1). The treated frame boards were pressure-treated to ground contact retention (2.4 kg/m^3) with CA-C. The raised beds were 1.2 × 3 m, constructed from a single 2.4-m piece of lumber that was halved for the box ends and two 3 m pieces of lumber for the sides which were held together with exterior screws. No additional remedial treatment was applied to the cut surfaces of the pressure-treated lumber. After the beds were constructed, the native soil was excavated to about 45 cm depth to loosen and layered into the beds with compost. The raised beds were then topped with a ~5-cm layer of compost before planting.

The four beds were planted in patterns identical to one another in each growing year. A different group of vegetables was selected for planting in years 1 and 2 and the planting plan is diagramed in Figs 2 and 3. Vegetable varieties used are summarized in Table 1. A mixture of common vegetables was seeded or planted into the beds at appropriate times based on their hardiness. The beds were watered by drip irrigation fed by 12.5 mm

Figure 1. Raised bed construction from the construction of boxes (top left) to bed digging (top right) to finished (bottom left) and in the first summer of growth with tomato trellising installed (bottom right).

irrigation tubing. Watering was done in response to weather and during the summer months was done daily. Trellising made from either galvanized 8-foot cattle panels, repurposed steel shelves, or untreated Douglas-fir trellising was used to support tomatoes and other vining plants (Figs 1 and 4). No copper input was expected from any of the trellising materials that would confound results.

Vegetable and Soil Collection

Produce was collected from the gardens as it matured. For each type of vegetable, a comingled sample of vegetable biomass was collected from each of the four beds. This was done to ensure that enough biomass was available for each bed for metal analysis. The size and productivity of vegetables varied in different boxes and because of

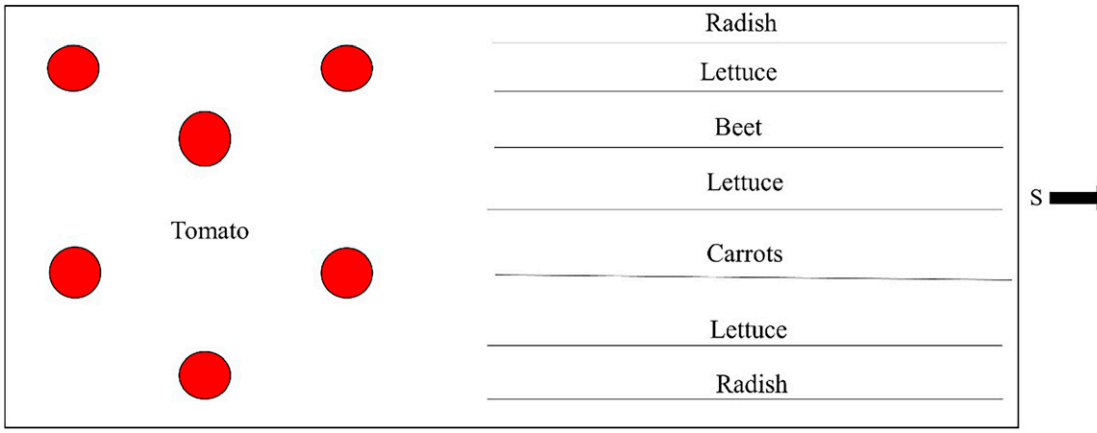

Figure 2. Planting diagram for year 1 for treated and untreated raised beds. Varieties grown for each vegetable are listed in Table 1 and circles indicate an individual plant. Basil was succession planted after beets were harvested in year 1.

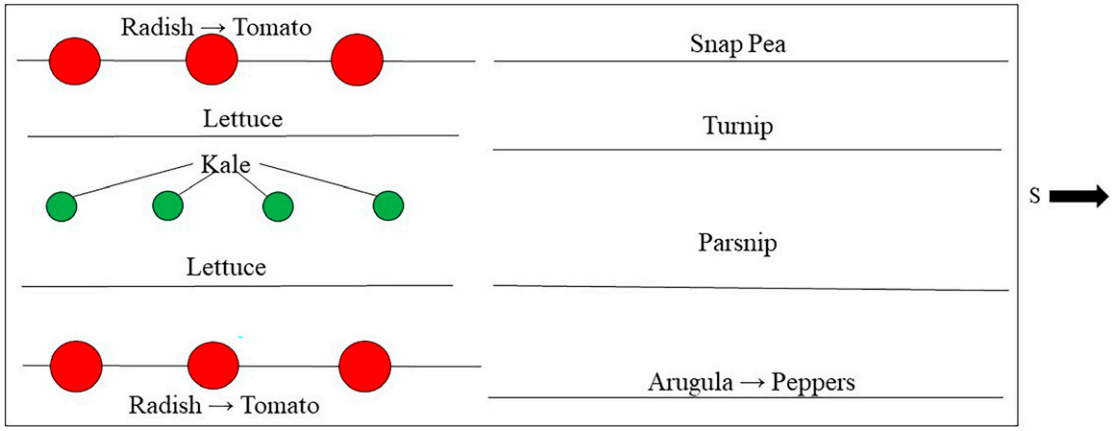

Figure 3. Planting diagram for year 2 for treated and untreated raised beds. Varieties grown for each vegetable are listed in Table 1 and circles indicate an individual plant. Arrows denote succession planting after the harvest of the first crop.

this, the total number of each plant harvested for analysis differed in some cases. For root crops such as carrots, beets, and parsnips, the leafy tops were separated from the roots and were analyzed separately. In some cases, inedible portions of non-root vegetables were analyzed in addition to the edible portion.

Soil samples were collected from the bed centers upon installation to serve as background. A single comingled sample of four samples was collected for each bed. At the end and start of each season (after fresh compost addition), soil samples were collected using a 25 × 305 mm soil corer from 0 to 25 mm from the bed edge, 76-102 mm from the bed edge, and the bed center (Fig 5). A total of four soil samples of each type were taken from each bed at each sampling point according to the diagram shown in Fig 5. The four equivalent samples at each sampling point were comingled and homogenized before analysis except for samples taken at the end of year 2 which consisted of four separate soil cores for each location in each bed. Soil samples were collected from the top of the soil surface to the full depth of the wood border.

Measurement of Copper

Soil or vegetable biomass was microwave digested in triplicate from comingled samples and analyzed for copper content using inductively coupled plasma mass spectrometry (ICP-MS). Soil or plant biomass samples were homogenized, oven-dried, and microwave-digested according to EPA method 3052. Briefly, 0.25 g of soil or 0.5 g of plant biomass was placed into PTFE microwave extraction tubes and 10 mL of concentrated nitric acid was added. Samples were digested for 9.5 min at 180°C with a total microwave digestion time of about 15 min. The resulting digestate was rinsed from the tube with DI water and brought up to a volume of 35 mL with DI water and analyzed for Cu using ICP-MS and expressed on a µg/g (PPM) basis.

Copper in extracts was measured using a Thermo-Elemental iCAP RQ ICP-MS. Each ICP-MS analytical session began by allowing the ICP-MS to warm up for 30 min before being tuned first in standard robust and then in kinetic energy discrimination robust (KEDR) modes utilizing a multiple element 1 part per billion (ppb) standards prepared in a 2% HNO_3 + 0.5% HCl matrix. Calibration standards were prepared from single-element standards and were spiked with 0.5 ppb indium for use as an internal standard. Calibration curves generated for 65Cu included nine standards and a blank. All standards and blanks were prepared using ultra-pure 2% HNO_3. Samples were spiked with 0.5 ppb indium as an internal standard and diluted 10-fold using laboratory-grade water. The analyte 65Cu was analyzed in KEDR mode. Each analysis

Table 1. Description of vegetable samples harvested from the raised beds in years 1 and 2 and which plant parts were analyzed.

Vegetable	Part analyzed	Variety	Seed source	Planting year
Basil	Leaf	Sweet Italian	Burpee	1
Basil	Stem	Sweet Italian	Burpee	1
Beet	Root	Early Wonder Tall top	Territorial Seed Co.	1
Beet	Tops	Early Wonder Tall top	Territorial Seed Co.	1
Carrot	Root	Carrot, Giants of Colmar	Territorial Seed Co.	1
Carrot	Tops	Carrot, Giants of Colmar	Territorial Seed Co.	1
Lettuce	Leaf	Mixed greens gourmet	Burpee	1
Radish	Leaf	Cherry Belle	Territorial Seed Co.	1
Radish	Root	Cherry Belle	Territorial Seed Co.	1
Tomato	Fruit	Rutgers	Territorial Seed Co.	1
Tomato	Vine	Rutgers	Territorial Seed Co.	1
Basil	Leaf	Sweet Italian	Burpee	1
Basil	Stem	Sweet Italian	Burpee	1
Beet	Root	Early Wonder Tall top	Territorial Seed Co.	1
Beet	Tops	Early Wonder Tall top	Territorial Seed Co.	1
Carrot	Root	Carrot, Giants of Colmar	Territorial Seed Co.	1
Carrot	Tops	Carrot, Giants of Colmar	Territorial Seed Co.	1
Lettuce	Leaf	Mixed greens gourmet	Burpee	1
Radish	Root	Cherry Belle	Territorial Seed Co.	1
Tomato	Fruit	Rutgers	Territorial Seed Co.	1
Tomato	Vine	Rutgers	Territorial Seed Co.	1
Arugula	Leaf	Roquette	Territorial Seed Co.	2
Radish	Root	Cherry Belle radish	Territorial Seed Co.	2
Radish	Leaf	Cherry Belle radish	Territorial Seed Co.	2
Turnip	Root	Turnip purple top globe	Territorial Seed Co.	2
Turnip	Greens	Turnip purple top globe	Territorial Seed Co.	2
Lettuce	Leaf	Lettuce Bibb	Burpee	2
Kale	Leaf	Kale Prism	Territorial Seed Co.	2
Pea	Pod	Super Sugar Snap	Territorial Seed Co.	2
Parsnip	Root	Gladiator F1	Territorial Seed Co.	2
Parsnip	Tops	Gladiator F1	Territorial Seed Co.	2
Tomato	Fruit	Rutgers	Territorial Seed Co.	2
Pepper	Fruit	Gold Star	Territorial Seed Co.	2

consisted of five 80 sweep runs with 65Cu analyzed with 10 ms dwell times. Quality control and internal precision were monitored by repeated measurements of the prepared standards following every 25th sample.

Statistical comparisons were made between copper levels in treated and untreated beds using a single factor ANOVA and a Tukey's honestly significant difference post hoc test, $\alpha = 0.05$.

RESULTS AND DISCUSSION

Copper concentration was measured on a dry weight basis for vegetables grown in years 1 and 2 are shown in Figs 6 and 7, respectively. There was no obvious difference in plant growth among the different beds although yields were not measured as part of this study. Copper levels are shown as averages for material sourced from treated and untreated beds are treated or untreated garden bed and little difference was detected among vegetables sourced from different bed types. There was no clear pattern of higher or lower copper levels based on bed type. In some instances, such as some radish roots and tomatoes, untreated beds produced vegetables with slightly higher copper content while in others such as some lettuce, treated beds may have had slightly higher copper

Figure 4. Progression of vegetable growth in the second growing season March (left), April (center), and May (right).

levels. However, none of these differences were statistically different than one another ($p > 0.05$, Tukey's HSD).

This is contrary to previous observations where carrot tops were identified as a potential accumulator of copper from garden bed materials (Love et al 2014). This study shows no difference in copper levels found in carrot tops grown in treated garden boxes or untreated garden boxes. Another study has shown that some plant tissues in beans, particularly roots, can show increased copper concentrations in soils when grown in copper-amended soils (Apodaca et al 2017). Intawongse and Dean (2006) showed that copper can accumulate in lettuce, spinach, and radish roots grown in copper-contaminated compost. However, copper levels in the growing medium in that study were over 10 times higher than those found in the current study and represent a severely metal-contaminated growing medium. Regardless, no increase in copper concentrations for root crops was measured in this study.

Copper levels in vegetables grown in year 2 followed a similar pattern where most average copper values in produce grown in treated or untreated beds were similar. Average copper levels in tomato fruit grown in treated beds trended slightly higher than in untreated beds, but the difference was not significant ($p > 0.05$, Tukey's HSD). Average copper levels trended higher in arugula and turnip greens grown in untreated boxes, but these were also not significantly different from levels in vegetables grown in untreated beds ($p > 0.05$, Tukey's HSD). These data indicate that for both years, copper levels in vegetables grown in treated and untreated beds were indistinguishable from one another.

Copper levels in soils were measured at the installation of the boxes, at the end of the first growing season, after the addition of compost for the second growing season, and after the second growing season (Fig 8). Copper concentrations in soil taken from treated garden boxes were about 23 ppm higher than equivalent samples taken from untreated boxes after the first year and the difference was statistically significant ($p < 0.05$, Tukey's HSD). This was a much lower increase in copper content than was observed in soils in direct contact with CCA-treated vineyard posts which increased by about 7-fold on average (Robinson et al 2006). No difference was seen in soils from the two-bed types at the 76-102 mm sampling point or the center bed sampling point, indicating that copper migration was limited to a small distance from the bed.

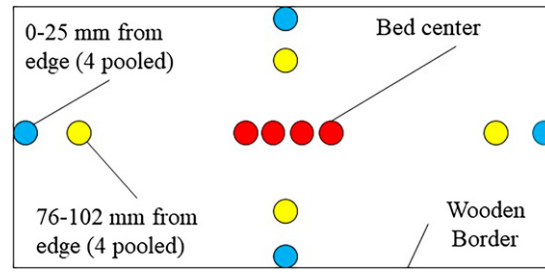

Figure 5. Sampling diagram for soil samples taken at the beginning and end of each growing season.

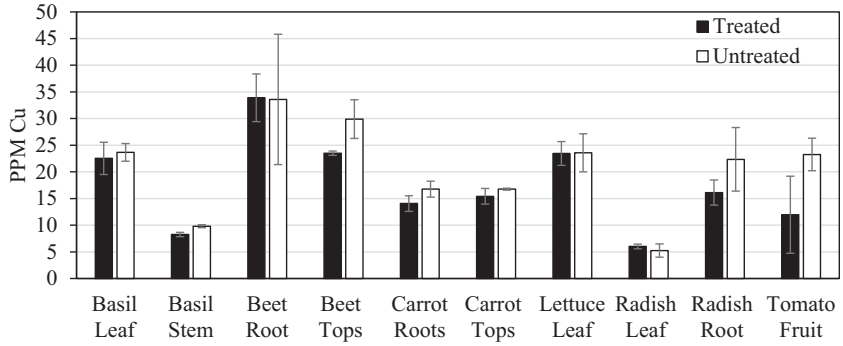

Figure 6. Copper levels were found in vegetable biomass taken from the raised beds in year 1. Error bars are plus or minus one standard deviation of three replicate extracts of two comingled pools of biomass.

Soil samples taken after compost addition at the start of the second year of growth did not show the same pattern and all equivalent samples taken from treated and untreated beds were indistinguishable from one another. This suggests that the compost addition washed out the copper signal from the treated bed by diluting the bed soil with fresh material. At the end of the second year, a nearly identical pattern to the first year emerged where average copper levels 0-25 mm from the bed edge were 21 ppm higher in the treated beds and the difference was statistically significant ($p < 0.05$, Tukey's HSD). No other sampling locations showed elevated copper levels in treated beds vs untreated beds, indicating that copper migration remained limited to the bed margins in the second year as well.

Copper levels measured are well within normal soil levels for the south Willamette Valley, OR which averages 38 PPM with a standard deviation of 30 PPM (Oregon Department of Environmental Quality 2013). Soil copper levels found in this study also fall well within normal ranges observed around the world which can range up to 495 PPM in the United States and even as high as 1508 PPM in the United Kingdom (Rehman et al 2019). The highest average Cu levels measured near treated wood boxes in this study was about 57 PPM and this is well within a range that would be considered statistically indistinguishable from natural copper levels in the Willamette Valley, OR. It is important to note that a fungal attack was observed on the untreated bed material as early as one year after installation whereas it was not on pressure-treated

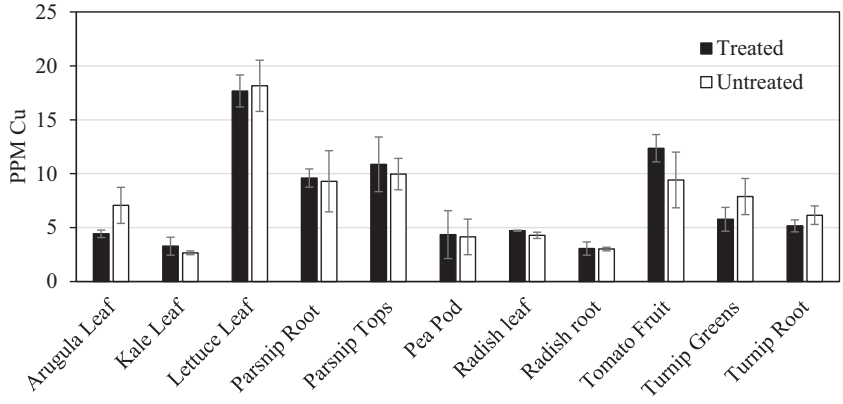

Figure 7. Copper levels were found in vegetable biomass taken from the raised beds in the year 2 season. Error bars are plus or minus one standard deviation of three replicate extracts of two comingled pools of biomass.

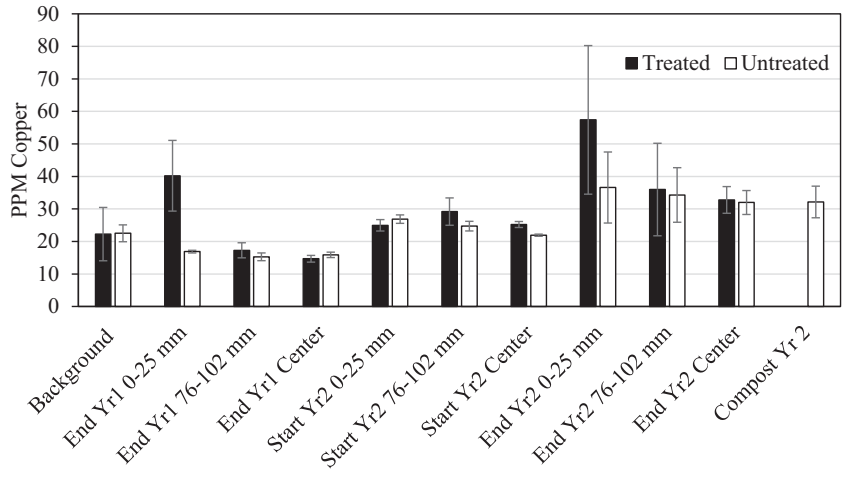

Figure 8. Copper levels were found in soils taken from treated and untreated garden boxes at the start of the study, the end of the first season, and the start of the second season of growth. Error bars are plus and minus one standard deviation three replicate extracts of two comingled pools of biomass, except for the end second-year sampling which consisted of four separate core samples from each bed extracted once.

beds. This illustrates the durability improvement from the use of pressure-treated bed material. While some increase in copper levels was observed within 25 mm of the treated wood beds, it was not enough to cause concern and it appeared to be limited to within a short distance from the bed material.

Copper migration from CA-C-treated wood has previously been studied using several different experimental methods. The potential for copper migration from CA-C treated decking to impact soil was studied by capturing decking runoff and applying it to columns packed with soil (Kennedy and Collins 2001). The previous study found that the amount of copper lost in runoff from the decking was insufficient to increase copper levels in the soil above their initial values. Soils in direct contact in this study showed an increase of about 20 PPM throughout a growing season. This is likely due to higher chemical loadings for wood in ground contact used in this study as opposed to decking and sustained contact of the wood with the soil to facilitate diffusion of the metal into the soil.

Another study that measured copper migration from CA-C treated wood stakes in soil-filled pots showed that soil immediately around the stakes increased in copper concentration by about 53-182 PPM Başkal et al (2023). These levels are about 2.5-9 times higher than observed in this study, but it is important to note that the CA-C retention levels in wood tested by Başkal and colleagues were 4.7-9.2 times higher than the 2.4 kg/m^3 ground contact retentions used for treated bed materials in this study. With that said, our results appear to show a proportional decrease in copper migration with the lower retention levels produced for the western United States market that are in line with previously observed migration rates.

This study analyzed copper migration from two treated or untreated garden boxes. While replication was built into the study through replicate sampling around each bed, the study would have benefited from greater replication. Soil as well as wood is a highly variable matrix of organic and inorganic matter and metal migration through these matrices can be highly varied from one location to another. Greater replication in the number of beds per treatment would have provided a better understanding of how copper migration varies among a greater population of treated wood. Subsequent studies on this topic should include a greater number of smaller beds.

Soils from the end of year 1 were extracted and analyzed for propiconazole and tebuconazole using high-performance liquid chromatography. Neither of these compounds were found in the initial sampling where the detection limit for propiconazole and tebuconazole was about 10 PPM in the soil (data not shown). No further analyses were done because it was determined that azoles were unlikely to be identified in any subsequent sampling. In CA-C, azoles are present at only 4% of the mass of copper. Even if migration is occurring in this system, it is likely entering soil at concentrations that are below the detection limits of our method.

CONCLUSION

This study shows that no increase in copper concentration was detectable in vegetables grown in garden boxes made with CA-C treated wood over vegetables grown in untreated beds. No effect of the treated beds was observed over two growing seasons. Treated wood garden bed material does increase copper concentrations in soils within 25 mm of the bed edge about 20 ppm above levels measured in untreated garden boxes. These increases were seen in both years of the study within 25 mm of the bed edge but were not measured at other locations farther from the bed edge. Increases in soil copper concentration near the bed edge were minor and did not increase copper levels beyond the natural range for the area.

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EFFECT OF DRYING OF DIFFERENT LIGHT HARDWOOD TROPICAL TIMBER SPECIES ON DURABILITY AGAINST *COPTOTERMES CURVIGNATHUS* HOLMGREN UNDER LABORATORY AND FIELD TESTS

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Abstract. The effects of drying temperature and duration on the durability of *Lagerstroemia* sp. and *Cinnamomum* sp. against subterranean termites, *Coptotermes curvignathus*, were evaluated in no-choice and choice laboratory tests as well as in the aboveground test. Samples measuring 25 mm × 25 mm × 6 mm and 100 mm × 40 mm × 20 mm were dried in an oven at three different temperatures: 60°C, 80°C, and 100°C for two different time periods: 10 and 15 d. In comparison between the control sample and the treated sample, the control sample showed the highest MC, the lowest visual rating, the lowest termite mortality, and the highest weight loss. For the treated samples, the results show that the samples for both wood species have a low resistance limit to termites at low temperatures and a short-drying time. The weight loss is also high for samples with high MC. The mortality rate of termites was also high in samples dried at high temperatures over a long period of time compared with samples dried at low temperatures for a short period of time. The visual rating results also showed the same trend as the weight loss results. The results for these three categories are identical for the no-choice, choice, and aboveground tests. The analysis demonstrates that the high material resistance of tropical wood species is mostly dependent on the temperature and length of time spent in the kiln.

Keywords: *Lagerstroemia* sp., *Cinnamomum* sp., drying, durability, *Coptotermes curvignathus.*

INTRODUCTION

When we deal with wood, we cannot escape the attack of pests, especially termites, as the humid temperatures in Malaysia strongly favor the activity of these pests. The presence of cellulose, the main food source in wood, is a center of attraction for termites. This can only be avoided if the wood used is properly treated. It is estimated that the damage caused by these pests in Malaysia amounts to RM 400 million per year (Anon 2021) and is estimated at over USD 40 billion for all places where termites are found (Subekti et al 2015).

Drying is one of the ways or alternative methods to improve the durability of wood against insects, decay, and other pests without using preservatives. It can be considered as a biocide-free alternative to improve the performance of wood species with low natural durability. The drying process can be seen as a hygrothermal wood treatment (Sehlstedt-Persson and Wamming 2010). In this process, some of the water is removed from the wood, which leads to a reduction in MC. This water removal process is necessary for further utilization of wood. Generally, temperature, time, species, and process precision all affect durability. As the temperature and treatment duration increased, density, swelling, and surface roughness decreased (Korkut et al 2008).

High MC is one of the factors that favor attacks on wood by biological agents, especially termites and fungi (Sajap et al 2008). The significance of moisture for termite survival has already been

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extensively discussed in previous studies (Oberst et al 2019). For example, the *Coptotermes acinaciformis* is a species of termite that prefers moist wood over dry wood and changes its MC depending on local conditions (McManamy et al 2008). *Reticulitermes flavipes* cannot maintain an infestation of wood without soil contact at an MC <24% as it requires a constant water supply. They require at least 30% wood moisture to survive for 6 mos.

Drying has been shown to have an impact on the durability of wood species. Fatima et al (2015) examined the resistance of three wood species drying at various drying temperatures and drying times against termites. The authors found that the wood-drying process had an effect on the durability of the wood. They also found that wood drying at a temperature (100°C) for 15 d has a higher durability than other combinations. Aihetasham et al (2012) examined the feeding of microcerotermes championsi on eight commercial wood species (*Cedrus deodara*, *Acacia arabica*, *Tectona grandis*, *Mangifera indica*, *Morus alba*, *Azadirachta indica*, *Ficus religiosa*, and *Melia azedarach*) drying at different temperatures. The study found that wood weight loss (WL) increased as the drying temperature increased, but did not have an effect on MC. Doi et al (2005) reported that the wood of Japanese larch (*Larix leptolepis*) dried at high temperatures (120-130°C) was susceptible to infestation by decay fungus and termite. In their study on heartwood samples of *Cryptomeria japonica* dried at high temperatures, Kano et al (2004) found that chemical compounds, such as sequirin C and agatharesinol, were vaporized, which caused the wood to be less resistant to subterranean termites (*Reticulitermes speratus*). In another study, pine (*Pinus sylvestris*) was significantly attacked when thermally modified at a temperature of 210°C for 15 min (Shi et al 2007). This means that drying wood at high temperatures is not suitable, as it alters the chemical components of the wood.

The leaves, roots, and bark of this species, *Lagerstroemia* sp., are traditionally used in medicine, especially for diabetes and to reduce body weight. Locally known as Bungur, it is a lesser-known timber species of the Lythraceae family originating in the Indo-Malayan region (including Indochina), extending into Indonesia and the Philippines, and often cultivated as an ornamental. Six new monomeric and dimeric ellagitannins, three new ellagitannins, 3-*O*-methylprotocatechuic acid, caffeic acid, *p*-coumaric acid, kaempferol, quercetin, and isoquercitrin are among the chemical compounds extracted from the leaves (using aqueous acetone) that have antioxidant, antibacterial, antiviral, antinociceptive, antidiarrheal, antiinflammatory, cytotoxic, antidiabetic, antifibrotic, and antiobesity effects (Chan et al 2014).

Cinnamomum sp. is an aromatic tree that belongs to the Lauraceae family. It is mainly distributed in Asia, China, and Australia (Jayaprakasha and Rao 2011) and is widely known as a culinary herb and traditionally used in medicine (Yanakiev 2020). It grows mainly in the tropical and subtropical regions of Southeast Asia, Australia, and America (North, Central, and South). With more than 500 compounds found in this plant, it has the potential for immunomodulatory, antiinflammatory, antitumor, antimicrobial, antioxidant, antifungal, antitermitic, insecticidal, and anticancer activities (Chang and Cheng 2002; Cheng et al 2009; Mdoe et al 2014; Wu et al 2020).

The wood of *Lagerstroemia* sp. is moderately durable, whereas *Cinnamomum* sp. is not durable when exposed to the weather or in contact with the soil (Wong 1982). A study by Febrianto et al (2015) on *Lagerstroemia* sp. wood grown in West Java, Indonesia, found that the sapwood of *Lagerstroemia* sp. is classified as resistant (natural durability class II) to *Coptotermes curvignathus* according to SNI 01-7207 (2006). *Cinnamomum* sp. only shows a WL of 0.98% when exposed to *C. curvignathus,* which classifies as very resistant according to EN 118 (2013) and Kadir et al (2017).

This study aims to find out if and how drying processes affect the durability of *Lagerstroemia* sp. and *Cinnamomum* sp. These two species were chosen to find alternatives to wood species that are commonly used, especially less well-known

species that are still not used commercially. Indirectly, it can also diversify the final product from wood. The focus was to assess how different drying times and temperatures affect termite feeding and mortality of subterranean termites, *C. curvignathus*, in the laboratory and aboveground tests.

MATERIALS AND METHODS

Raw Materials

Two Malaysian tropical timber species, *Lagerstroemia* sp. and *Cinnamomum* sp., were sampled from the Forest Research Institute Malaysia (FRIM) timber yard. Both species grow naturally on the FRIM site and were identified by FRIM's anatomist, Dr. Nordahlia Abdullah Siam. *Lagerstroemia* sp. was about 30 yr old and had a diameter of 62.3 cm, whereas *Cinnamomum* sp. was about 28 yr old and had a diameter of 49.5 cm. Only one tree was used for each species.

Each log is split into 50 mm × 100 mm × 200 mm-sized pieces of wood using a rip saw machine and labeled between sapwood and heartwood. The wood blocks, measuring 25 mm × 25 mm × 6 mm (laboratory) and 100 mm × 40 mm × 20 mm (aboveground), were cut from the heartwood of the basal part of each tree species. The difference between sapwood and heartwood is identified by color.

The wood blocks were then oven-dried according to six different drying sequences for each species and weighed after (initial weight). All samples were conditioned for 2 wk before testing began. For these tests, five (no-choice in the laboratory) and 10 (choice in the laboratory and aboveground) replicates were used for each drying series, resulting in a total of 350 samples. The samples for the laboratory and aboveground tests came from the same trees.

Moisture Content Determination

The MC of both wood species was measured in the oven-dried state immediately after treatment but before the termite test was carried out. The MC of the control sample (without drying treatment) was also measured before the termite test was carried out. To determine the MC, a total of five replicates (25 mm × 25 mm × 6 mm) were cut from the basal parts of each wood species. Before oven drying, each replicate was first weighed (W_i) and then dried at 103 ± 2°C until it reached a constant weight (W_o). The MC was calculated based on Eq (1)

$$\text{Moisture content } (\%) = \frac{Wi - Wo}{Wo} \times 100\% \quad (1)$$

Termites

Subterranean termites, *C. curvignathus* Holmgren, were collected by breaking and carefully tapping infested rubber trees (*Hevea brasiliensis*) at the Forest Research Institute Malaysia (FRIM) and placing them in plastic trays with moist paper toweling. Termite species were identified using the key from Tho (1992). The stock of termites was brought back to the laboratory on the same day the test began.

Termite Test

Laboratory tests (no-choice and choice tests). The samples were subjected to a termite bioassay according to the FRIM in-house (IHM/WEL/4 2014) no-choice and choice (the termites are given two things to eat, control vs heat-treated of the same species) test procedure.

Each 8 cm × 13 cm screw-cap bottle was filled with 200 g of sterilized sand (test medium) and 30 mL of distilled water. The bottle is left overnight to equilibrate the moisture in the sand after being moistened with distilled water before testing begins. The test block (one block for the no-choice test and two blocks for the choice test) was placed at the bottom of the bottle. Then, 400 healthy termites in their natural ratio (360 workers and 40 soldiers) were added to each bottle. All bottles were kept in an incubator at 22 ± 2°C and 65 ± 5% RH for 28 d. If all termites are found dead (no tunneling activities) during this test period, the test bottle is removed, and the number of days until 100% death is recorded. At the end

of the 28 d, the blocks were removed, cleaned, dried overnight (103 ± 2°C), and weighed again (final weight). The remaining live termites were counted and recorded for each of the bottles. The termite mortality is calculated based on Eq (2)

$$\text{Termite mortality } (\%) = \frac{\text{initial number of termite used} - \text{number alive at the end of test}}{\text{initial umber of termite used}} \times 100 \quad (2)$$

Each test sample (either choice samples or no-choice tests) was scored according to the standard method [IHM/WEL/4 (2014)]: 0 = failure (almost complete loss of strength); 4 = very heavy infestation, 50-75% of the cross-sectional area affected; 6 = heavy infestation, 30-50% of the cross-sectional area affected; 7 = moderate/heavy infestation, penetration, 10-30% of the cross-sectional area affected; 8 = moderate infestation, 3-10% of the cross-sectional area affected; 9 = light infestation up to 3% of the cross-sectional area affected; 9.5 = traces, nibbling on the surface allowed, and 10 = sound. The WL is calculated based on Eq (3)

$$\text{Weight loss } (\%) = \frac{\text{initial weight} - \text{final weight}}{\text{initial weight}} \times 100 \quad (3)$$

Aboveground test. The aboveground test was carried out on the basis of FRIM's in-house method (IHM/WEL/1 2004). The field test sites are located on cleared areas in the FRIM substation in Mata Ayer, Perlis. The soil is lateritic, with a pH between 4.97 and 5.25 (Md Noor 2003) and an average annual precipitation of 112.76 mm (Anon 2022). The area is overgrown with grass, weeds, shrubs, and pines and is infested with Asian subterranean termites, *C. curvignathus*, and *Coptotermes gestroi*. Certain parts of the area are also heavily infested with *Macrotermes* spp. and *Globitermes* spp.

The wood blocks for the aboveground test were oven-dried according to six different drying sequences for each species and weighed (initial weight). All samples were conditioned for 2 wk before the test began. Nine drums were placed in the area on February 20, 2022. A layer of highly susceptible woody substrate, *H. brasiliensis*, was placed close to the ends at the bottom of the drums. Then a square section of galvanized welded mesh (25 mm square opening) was placed on the top of the wood substrate. The test specimens covered with *H. brasiliensis* were placed on the galvanized welded mesh. The specimens were randomly arranged to avoid mutual contact. All specimens were left for 16 wk without disturbing them. The same control sample (not oven-dried) as in the no-choice and choice tests was used as a control sample. At the end of the 16 wk, the test samples were removed, cleaned, dried overnight (103 ± 2°C), and weighed again (final weight). Each test sample was assigned a classification according to the standard method [IHM/WEL/1 (2004)]: 0 = intact; 1 = traces of infestation, surface nibbling allowed; 2 = light and superficial infestation, up to 3% of the cross-sectional area affected; 3 = moderate/severe infestation, penetration, 4-40% of the cross-sectional area affected; 4 = severe/very severe infestation, 41-70% of the cross-sectional area affected; and 5 = failure (almost complete loss of strength). The WL is calculated based on Eq 3.

Statistical Analysis

Variations in WL, degree of infestation, and termite mortality with different drying methods of wood were compared and analyzed by analysis of variance (ANOVA, one-way) using Microsoft Excel 2003 to determine which groups differed significantly at the 5% significance level (α) when ANOVA indicated a significant difference between temperature and drying time and also between percentage WL and visual assessment in samples of both wood species. The assumptions of normality of the data were tested on the raw data using the Shapiro–Wilk test. The threshold for significance was set at $\alpha = 0.05$. If the assumptions were met, a one-way ANOVA was performed using PROC GLM, followed by a comparison of means using the Duncan multiple range test. If the assumptions were not met, a logarithmic transformation was used to normalize the data.

RESULTS AND DISCUSSIONS

Laboratory Test

No-choice. Table 1 shows the results of the no-choice test for two different types of wood drying at different temperatures, and drying times. The wood dried by different methods degraded the termites according to WL and visual assessment, with the degree of degradation varying according to temperature and drying time. The temperature and wood moisture played a significant and complementary role in the wood consumption of *C. curvignathus*.

It was found that the sample of *Cinnamomum* sp. dried at 80°C for 10 d was more susceptible to termites with a higher WL of 4.4% than the samples dried at 100°C for 15 d, which had the lowest WL of 0.5%. For *Lagerstroemia* sp., the most susceptible samples (WL of 4.1%) were found at 60°C for a 10-d drying period, and the least susceptible (2.0%) were found in samples dried at 100°C for 15 d. This shows that the resistance of both species of wood to termite attack increases with increasing intensity of the drying temperature and longer drying. Higher and lower WLs of *Cinnamomum* sp. occurred in test samples with high (19.1%) and low (13.2%) MC, respectively. The same trend was observed for the test samples of *Lagerstroemia* sp. Higher WL was observed in the high MC sample (17.1%), and lower WL was observed in the low MC sample (13.3%) (Table 1). ANOVA analysis also showed significant WL (except for a few cases) when the samples of the two wood species were dried at different temperatures and drying times in the no-choice test (temperature [df = 6; $F = 11.5294$, $p = 0.0037$ for *Cinnamomum* sp. and df = 6; $F = 14.458$, $p = 0.00194$ for *Lagerstroemia* sp., respectively] and drying time [df = 6; $F = 9.3637$, $p = 0.00748$ for *Cinnamomum* sp. and df = 6; $F = 6.5374$, $p = 0.00358712$ for *Lagerstroemia* sp.]).

The test results show that samples with a high MC (dried at 60°C for 10 and 15 d) show a higher WL. There is a reason for this when the wood MC is high, there is a lot of free water in the cell cavities, which contributes to the wood becoming softer. When the wood is dried, the OH group in the wood decreases due to the amount of water that has leaked out. This leads to a lack of OH groups that attract water and, therefore, a lower MC in the wood. The wood is less sensitive to changes in humidity, which further increases its stability and durability (Leggate et al 2020). Therefore, temperature plays an important role and is also complementary to WL at all moisture

Table 1. The average percentage of weight loss, termite mortality and MC, and visual rating of dried woods against *C. curvignathus* in no-choice laboratory tests.

	Treatments					
Wood species	Temperature (°C)	Days	Wood weight loss (%)	Visual rating	Termite mortality (%)	Moisture content (%)
Cinnamomum sp.	Control	Control	6.4 (0.26)a	6.0 (1.20)d	44.3 (5.36)d	46.3 (2.12)a
	60	10	4.0 (0.97)bc	6.4 (1.11)d	75.1 (8.67)c	15.0 (1.47)c
	60	15	3.5 (1.07)c	7.0 (0.00)c	76.2 (1.11)c	14.6 (1.17)c
	80	10	4.4 (0.83)b	7.0 (1.00)c	86.9 (2.56)b	19.1 (0.94)b
	80	15	3.0 (1.44)d	7.4 (0.95)c	89.3 (2.58)b	15.0 (3.09)c
	100	10	1.7 (1.28)d	8.2 (1.64)b	100 (0.00)a	12.7 (0.68)d
	100	15	0.5 (0.41)e	8.6 (1.22)a	100 (0.09)a	13.2 (0.47)d
Lagerstroemia sp.	Control	Control	5.5 (0.08)a	6.0 (1.00)d	36.5 (2.22)d	40.1 (1.87)a
	60	10	4.1 (1.31)b	6.2 (1.67)d	71.2 (2.68)c	17.1 (0.86)b
	60	15	3.2 (1.29)c	7.0 (0.00)c	76.2 (1.33)c	16.0 (2.34)c
	80	10	3.5 (0.41)c	6.4 (0.45)d	76.5 (1.22)c	16.9 (1.11)c
	80	15	3.0 (1.65)c	7.0 (0.00)c	80.0 (0.00)c	13.6 (0.66)d
	100	10	2.3 (1.69)d	7.8 (0.00)b	92.5 (1.78)b	13.5 (1.18)d
	100	15	2.0 (0.99)d	8.5 (1.34)a	100 (0.000)a	13.3 (1.00)d

Mean (±SD) of five replicates for each timber species. Percentage values followed by the same letter (vertical) are not significantly different in the same group at the 0.05 level of probability.

levels. This is confirmed by Gautam and Henderson (2011), where the highest percentage of WL by *Coptotermes formosanus* occurred in wood blocks with high MC (125-150%) compared with low MC (22-24%). In other studies, Sponsler and Appel (1990) reported that termite nesting materials and wood with an MC >16% have air spaces that are saturated with moisture and thus ensure the survival of termites, although the permeability of their cuticle is the main process of drying out water from their body.

The mortality of *C. curvignathus* in both wood species samples treated at high temperatures (100°C) for a longer period (10 and 15 d) was significantly higher than at low temperatures (60°C) and shorter duration (10 d) (Table 1). Termite mortality increased with increasing drying temperatures and longer drying durations. The lowest mortality (36.5%) was observed in the control treatment of *Lagerstroemia* sp. after 28 d, and the highest mortality rate (100%) was observed in samples of treated *Lagerstroemia* sp. dried at 100°C for 15 d and samples of *Cinnamomum* sp. dried at 100°C for 10 and 15 d. The increase in temperature showed a significant difference (df = 6; $F = 9.99521$, $p = 0.01953$ for *Cinnamomum* sp. and df = 6; $F = 5.5896$, $p = 0.05596$ for *Lagerstroemia* sp., respectively) in termite mortality, except when the temperature was increased from 60 to 80°C for the species *Cinnamomum* sp.

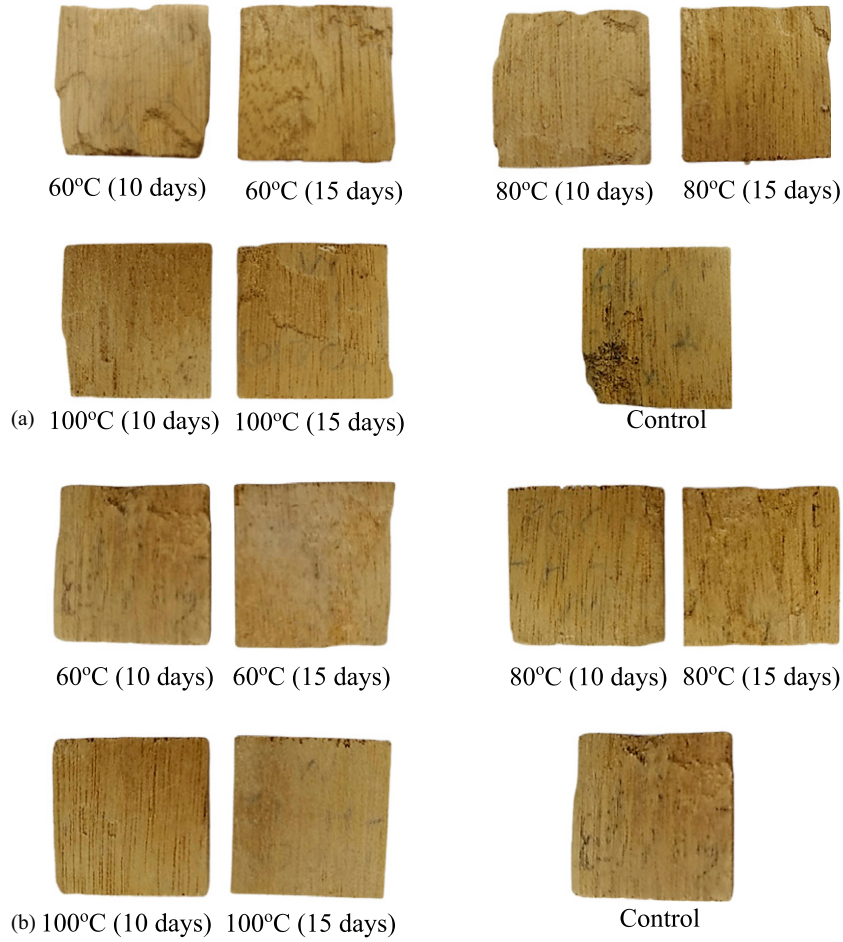

Figure 1. A representative sample of *Cinnamomum* sp. after 28 d exposure to *C. curvignathus* in (a) no-choice and (b) choice tests.

Increasing the drying time (10-15 d) also showed no significant difference (df = 6; $F = 3.144497$, $p = 0.126549$ for *Cinnamomum* sp. and df = 6; $F = 1.957738$, $p = 0.180843$ for *Lagerstroemia* sp., respectively) in termite mortality, except for the samples of *Cinnamomum* sp. dried at a temperature of 100°C.

The results of the visual assessment of the no-choice test show that both samples of *Cinnamomum* sp. and *Lagerstroemia* sp. had higher visual ratings (8.6 and 8.5, respectively) when dried at higher temperatures (100°C) and the longest drying time (15 d), which were rated as slightly to moderately impaired (3-10% of cross-sectional area impaired) (Table 1). The lowest visual score (6.4 for *Cinnamomum* sp. and 6.2 for *Lagerstroemia* sp.) was observed when treated at 60°C and dried for 10 d (Fig 1). Both wood species control samples had a visual rating of 6.0. Significant differences (df = 6; $F = 14.8256$, $p = 0.00846$ for *Cinnamomum* sp. and df = 6; $F = 5.12517$, $p = 0.064205$ for *Lagerstroemia* sp., respectively) in the visual ratings in the no-choice test were observed between the samples of the two wood species, with few exceptions. The results of the visual ratings for the choice test also showed the same trend as for the no-choice test. The highest score (9.6 for *Cinnamomum* sp. and 8.5 for *Lagerstroemia* sp.) was obtained for a 15-d treatment at 100°C and the lowest (8.5 for *Cinnamomum* sp. and 6.9 for *Lagerstroemia* sp.) for a 10-d drying at 60°C.

The ANOVA analysis showed that there was no significant difference (df = 6; $F = 0.09756$, $p = 0.76535$ for *Cinnamomum* sp. and df = 6; $F = 0.094847$, $p = 0.768515$ for *Lagerstroemia* sp., respectively) in visual assessment between the samples of *Cinnamomum* sp. treated with different drying methods, except for the treatment at 100°C and the drying duration of 15 d. Significant differences (df = 6; $F = 28.5935$, $p = 0.0043$ for *Cinnamomum* sp. and df = 6; $F = 23.5739$, $p = 0.00284$ for *Lagerstroemia* sp., respectively) in visual assessment occurred only in some samples of *Lagerstroemia* sp. In general, visual assessment scores for *Cinnamomum* sp. increased from heavy infestation to light infestation in the no-choice test and from moderate infestation to allowed traces and superficial nibbles in the choice test. In *Lagerstroemia* sp., however, the visual rating increased from heavy infestation to moderate infestation.

Choice test. In the choice test, temperature and drying time also had significant effects on the

Table 2. The average percentage of weight loss, termite mortality and MC, and visual rating of dried woods against *C. curvignathus* in choice laboratory tests.

	Treatments					
Wood species	Temperature (°C)	Days	Wood weight loss (%)	Visual rating	Termite mortality (%)	Moisture content (%)
Cinnamomum sp.	Control	Control	6.4 (1.11)a	8.0 (0.00) c	42.6 (6.35)d	44.2 (2.38)a
	60	10	4.1 (1.86)b	8.5 (0.98)b	74.7 (1.46)c	15.0 (0.74)c
	60	15	4.6 (0.52)b	8.8 (1.11)b	79.8 (2.26)c	16.5 (2.53)b
	80	10	4.0 (0.99)b	8.8 (0.64)b	88.2 (1.00)b	14.8 (1.64)c
	80	15	3.6 (0.16)c	9.0 (0.00)b	89.5 (0.98)b	13.5 (0.87)d
	100	10	1.2 (1.64)d	9.2 (2.44)b	98.5 (1.00)a	12.9 (0.66)e
	100	15	0.5 (0.41)c	9.6 (1.84)a	100 (0.00)a	12.1 (0.53)e
Lagerstroemia sp.	Control	Control	6.0 (0.74)a	6.5 (1.00)c	30.1 (3.37)e	39.9 (1.88)a
	60	10	4.9 (1.26)b	6.9 (1.46)c	70.2 (1.48)d	16.5 (1.11)b
	60	15	4.0 (1.34)c	7.0 (0.00)c	74.5 (2.24)cd	15.4 (0.68)c
	80	10	4.7 (1.72)b	6.4 (1.32)d	77.8 (1.88)bc	16.3 (2.12)b
	80	15	4.5 (0.47)bc	7.0 (0.00)c	81.2 (1.08)b	13.7 (1.00)d
	100	10	4.2 (1.90)c	8.2 (1.22)b	92.2 (1.00)a	13.8 (0.98)d
	100	15	2.6 (1.38)d	8.5 (1.00)a	98.9 (0.560)a	13.5 (0.66)d

Mean (±SD) of ten replicates for each timber species. Percentage values followed by the same letter (vertical) are not significantly different in the same group at the 0.05 level of probability.

percentage WL of the test blocks of both wood species. As in the no-choice test, the WL of the wood blocks infested with *C. curvignathus* differed significantly difference with increasing temperature (df = 6; F = 14.5185214, p = 0.0776713 for *Cinnamomum* sp. and df = 6; F = 11.23461, p = 0.084267 for *Lagerstroemia* sp.) and drying time (df = 6; F = 44.286825, p = 0.005564 for *Cinnamomum* sp. and df = 6; F = 39.758081, p = 0.007422 for *Lagerstroemia* sp.). The highest percentage WLs were 4.6% (*Cinnamomum* sp. dried at 60°C in the 15-d dry period) and 4.9% (*Lagerstroemia* sp. dried at 60°C in the 10-d dry period) (Table 2). The lowest (less susceptible) were 0.5% (*Cinnamomum* sp.) and 2.6% (*Lagerstroemia* sp.). Both samples were dried at 100°C in a 15-d drying period (Fig 2). *Coptotermes curvignathus* consumed more *Cinnamomum* sp. (6.4%) compared with *Lagerstroemia* sp. (6.0%) for control samples. The highest and lowest WL values for both wood species occurred in samples with high (16.5% for *Cinnamomum* sp. and 16.5% for *Lagerstroemia* sp.) and low MC (12.1% for *Cinnamomum* sp. and 13.5% for *Lagerstroemia* sp.).

As with the no-choice test, the *Lagerstroemia* sp. sample is more likely to be eaten by termites due to the higher percentage of WL compared with

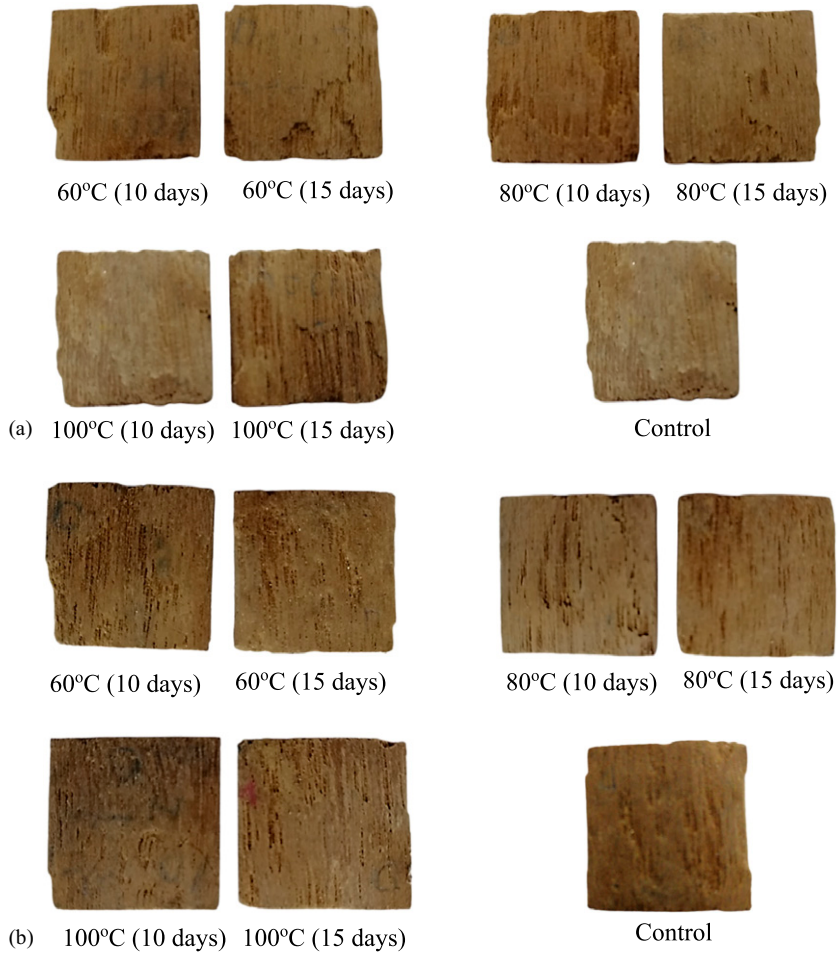

Figure 2. A representative sample of *Lagerstroemia* sp. after 28 d exposure to *C. curvignathus* in (a) no-choice and (b) choice tests.

Cinnamomum sp. Weekly monitoring revealed that the termites in the samples from the *Cinnamomum* sp. test bottle died earlier (only 2-3 wk of life) than the termites in the *Lagerstroemia* sp. test bottle (4 wk). For both wood species (*Cinnamomum* sp. and *Lagerstroemia* sp.), a significant (df = 6; F = 7.700679, p = 0.0032209 for *Cinnamomum* sp. and df = 6; F = 6.642439, p = 0.041922 for *Lagerstroemia* sp.) reduction in WL was observed at the highest temperature (100°C) and the longest drying time (15 d) compared with the lowest temperature (60°C) and the shortest drying time (10 d).

The mortality of *C. curvignath*us in wood samples treated at high temperature (100°C) for a longer period (15 d) was significantly higher (df = 6; F = 27.60096, p = 0.001913 for *Cinnamomum* sp. and df = 6; F = 23.00843, p = 0.003011 for *Lagerstroemia* sp.) than at low temperature (60°C) and shorter duration (10 d). Termite mortality increased with increasing drying temperatures and longer drying durations. The lowest mortality (30.1%) was observed in the control treatment of *Cinnamomum* sp. after 28 d in the choice test, and the highest mortality rate (100%) was observed in samples of *Cinnamomum* sp. dried at 100°C for 15 d. The increase in temperature showed a significant difference (df = 6; F = 268431, p = 0.0048 for *Cinnamomum* sp. and df = 6; F = 22.0723, p = 0.00333 for *Lagerstroemia* sp., respectively) in termite mortality. Increasing the drying time (10-15 d) also showed no significant difference (df = 6; F = 2.888526, p = 0.108567 for *Cinnamomum* sp. and df = 6; F = 0.033058, p = 0.858009 for *Lagerstroemia* sp., respectively) in termite mortality, except for the samples of *Cinnamomum* sp. dried at a temperature of 100°C. The high mortality of termites at high temperatures can be caused by a lack of moisture. Termites, especially subterranean termites, need moisture to continue living. Actually, subterranean termites cannot survive without moisture (Ferreira et al 2019). Temperature is the most important abiotic factor that affects the ability of termites to survive because it can cause movement, reduce survival, and cause rapid knockdown and death (Appel et al 1983; Quarcoo et al 2019).

In terms of visual rating, *Cinnamomum* sp. has a higher value (8.0-9.6) than *Lagerstroemia* sp. (6.4-8.5). Both control samples showed the lowest visual rating value (8.0 for *Cinnamomum* sp. and 6.5 for *Lagerstroemia* sp.) compared with the treated samples for the same species. There is no significant difference (df = 6; F = 0.615384615,

Table 3. The average percentage of weight loss and MC, and visual rating of dried woods against *C. curvignathus* in aboveground tests.

	Treatments				
Wood species	Temperature (°C)	Days	Wood weight loss (%)	Visual rating	Moisture content (%)
Cinnamomum sp	Control	Control	7.9 (1.22)a	3.6 (1.42)a	39.9 (3.41)a
	60	10	5.5 (0.68)b	3.4 (1.00)ab	20.1 (1.12)b
	60	15	5.7 (0.02)b	3.5 (0.98)a	20.5 (0.66)b
	80	10	5.1 (1.11)b	3.1 (0.07)b	16.3 (0.88)c
	80	15	4.8 (2.54)bc	2.8 (0.56)b	15.9 (2.12)c
	100	10	4.3 (1.11)c	2.3 (0.44)c	13.9 (1.00)d
	100	15	3.6 (0.98)d	2.0 (1.22)c	14.0 (1.43)d
Lagerstroemia sp.	Control	Control	12.1 (1.22)a	4.3 (2.22)a	42.1 (1.22)a
	60	10	10.0 (1.0)b	4.0 (1.11)a	21.7 (1.86)b
	60	15	9.9 (0.87)bc	3.8 (0.98)ab	19.6 (2.10)b
	80	10	8.8 (0.06)c	3.8 (0.012)ab	17.0 (1.46)c
	80	15	8.6 (1.11)c	3.6 (0.09)b	16.3 (2.22)c
	100	10	7.2 (2.74)d	2.8 (1.32)c	13.5 (0.48)d
	100	15	6.5 (2.22)c	2.5 (1.00)c	14.4 (0.56)d

Mean (±SD) of 10 replicates for each timber species. Percentage values followed by the same letter (vertical) are not significantly different in the same group at the 0.05 level of probability.

$p = 0.45537$ for *Cinnamomum* sp. and df = 6; $F = 0.285714286$, $p = 0.60751$ for *Lagerstroemia* sp., respectively) shown by ANOVA for the extension of drying time at the same temperature, but it is different when the temperature is increased, except for treatment with a temperature of 100°C for both drying times (10 and 15 d). A different situation occurred in samples of *Lagerstroemia* sp. Significant differences (df = 6; $F = 7.1227283$, $p = 0.0370837$ for *Cinnamomum* sp. and df = 6; $F = 5.6443$, $p = 0.05508$ for *Lagerstroemia* sp., respectively) occurred in samples treated at the same temperature but at different drying times. This situation is the exception for samples treated at 60°C for both drying times. However, all the treatments given to both species of wood improve the visual quality of the treated wood.

Aboveground test. The results of the aboveground test show that the percentage WL of *Cinnamomum* sp. is lower than that of *Lagerstroemia* sp. (Table 3). However, after 16 wk of exposure, both species showed the same trend in percentage WL (wood consumption) and also in visual assessment. The percentage WL decreased (from 5.5 to 3.6% for *Cinnamomum* sp. and from 10.0 to 6.5% for *Lagerstroemi*a sp.) when the temperature (from 60 to 100°C) and drying time (from 10 to 15 d)

Figure 3. A representative sample of *Cinnamomum* sp. after 16 wk exposure to *C. curvignathus* in the aboveground test.

were increased, although there were no significant differences in a few cases. The percentage WL for both wood species parallels the MC of the wood samples. The WL decreases as the MC decreases. There were signs of infestation in all test boxes, confirming that termites were present and widespread in each test box. A significant (df = 6; F = 10.28571429, p = 0.01248 for *Cinnamomum* sp. and df = 6; F = 010, p = 0.01335 for *Lagerstroemia* sp., respectively) reduction in WL was observed in both wood species (*Cinnamomum* sp. and *Lagerstroemia* sp.) at the highest temperature (100°C) and at the longest drying time (15 d) compared with the control treatment. Temperature is another important factor for the feeding and survival of subterranean termites (Nakayama et al 2005; Gautam and Henderson 2011; Aihetasham and Iqbal 2012).

The visual ratings of the two wood species samples ranged from 2.0 (light and superficial infestation, up to 3% of the cross-sectional area infested) to 4.3 (heavy or very heavy infestation, 41-70% of the cross-sectional area infested) for the percentage area of termite infestation on the wood surface. The control sample was the most severely

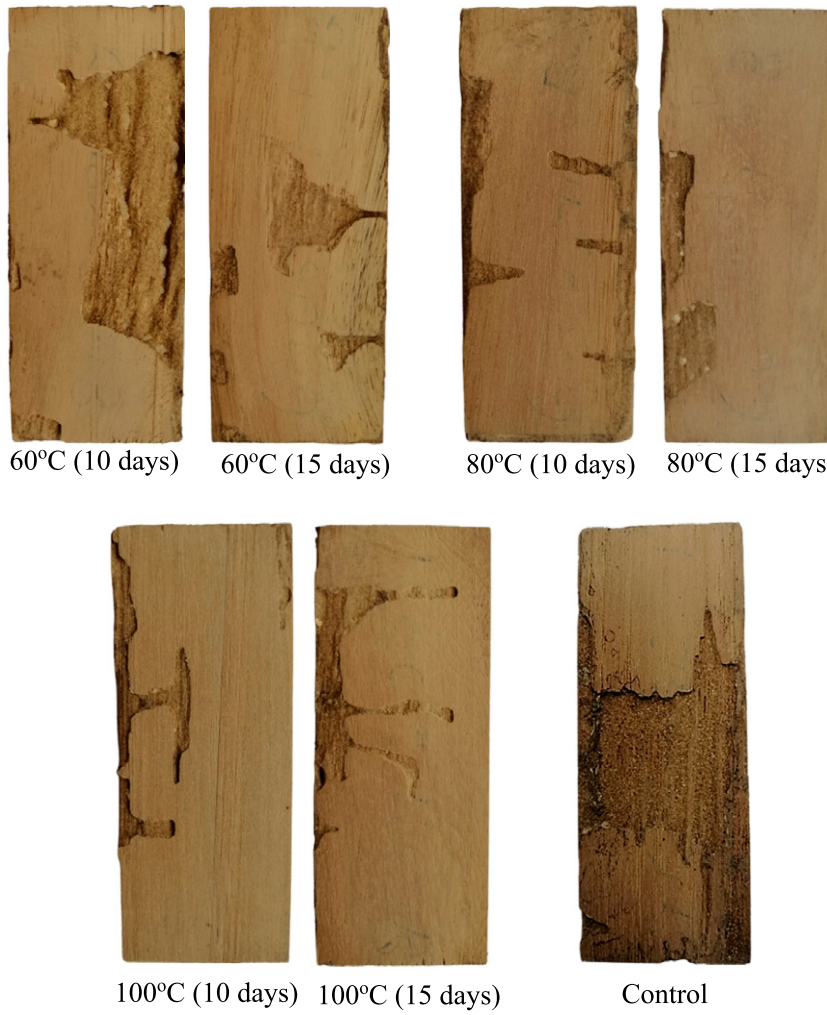

Figure 4. A representative sample of *Lagerstroemia* sp. after 16 wk exposure to *C. curvignathus* in the aboveground test.

damaged, with an average rating of 3.6 (*Cinnamomum* sp.) and 4.3 (*Lagerstroemia* sp.). In general, a higher temperature and a longer drying time reduced termite infestation on the wood samples for both species studied. The visual observations support the WL results (Figs 3 and 4).

The difference in WL caused by termite attacks on wood species can be caused by the fact that certain wood species have characteristics that make them palatable to termites and depend on the chemical, physical, and biological properties of the wood (Supriana 1988). *Cinnamon* groups consist of a variety of resinous compounds, including cinnamaldehyde, cinnamic acid, and numerous essential oils. Essential oils are among the substances that protect the wood from pest attacks (Senanayake et al 1978; Franzios et al 1997; Kadir et al 2013; Rao and Gan 2014), but not all extracts are toxic against destructive factors (Sjöstrom 1993; Wistara et al 2002).

CONCLUSIONS

The drying method of two different Malaysian woods was tested to determine whether the drying process affected the durability of the wood against subterranean termites. The drying temperature and drying time affect the WL and visual evaluation of the two tested wood species. In general, the higher the drying temperature and the longer the drying time, the lower the WL and the higher the visual evaluation value. There were no significant differences in terms of WL percentage or visual rating on samples from both wood species.

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