Tannin Extracts as a Preservative for Pine Thermo-mechanically Densified Wood

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Thermo-mechanical densification modifies wood to produce a more dense and resistant lignocellulosic material and may degrade extractives that contribute to the increased susceptibility of wood to attack by xylophagous organisms. This study evaluated the efficiency of tannin extracts of *Acacia mearnsii* in the treatment of thermo-mechanical densified pine wood in relation to physical, mechanical, and biological resistance (*Cryptotermes brevis*) properties. *Pinus elliottii* samples were pretreated with oxalic acid in a Parr reactor, then treated by diffusion in tannin solutions at concentrations 5, 10, and 15%, and finally hot pressed. The apparent density of the modified wood was 87.8% greater than that of the *in natura* wood (control) with tannins at 15%. The mechanical strength increased, especially the parallel compressive strength, which had an average increase of 169% for the wood with tannins at 10 and 15%, compared with the *in natura* wood. There was an increase in termite mortality and a reduction in damage for the modified wood treated with 15% tannins, obtaining the best results in mechanical and biological resistance and for the physical parameters. Thermal densification pine wood and preserved with tannin extractives proved to be a potential alternative as a high performance material.

DOI: 10.15376/biores.18.1.641-652

Keywords: Biodeterioration; Thermo-mechanical densification; X-ray densitometry; Preservatives; Tannins

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INTRODUCTION

Thermomechanical densification or thermal densification is a modification process in which temperature and pressure is applied to wood to decrease the space between the pores and, consequently, increase the density and mechanical strength (Laine *et al.* 2016; Pelit and Yalçin 2017; Pertuzzatti *et al.* 2018). It is performed on low-density woods, such as pine, adding value and expanding use by improving physical and mechanical properties (Pertuzzatti *et al.* 2018), making it possible to produce high-performance building lignocellulosic materials.

Some authors have proposed the use of thermomechanical modification in conjunction with a chemical pretreatment (pre-hydrolysis) with oxalic acid or sodium hydroxide and sodium sulfite, in order to partially remove hemicelluloses and lignin, making the wood more pliable, and resulting in improved ease of compression and reduced damage to the wood structure during thermomechanical densification (Pertuzzatti *et al.* 2018; Song *et al.* 2018; Silva 2019).

However, chemical pretreatment and thermal modification in the press can contribute to increased susceptibility of wood to attack by xylophagous organisms and termites due to the degradation of extractives (Unsal *et al.* 2008; Lesar *et al.* 2013; Khalil *et al.* 2014). Preservative treatment in conjunction with thermal densification can be an alternative for improving the biological resistance of modified wood.

Among wood preservatives, tannins extracted from *Acacia mearnsii* bark, which are flavan-3-ol and/or flavan-3,4-diol polymers, are a potential alternative in replacement of conventional water-soluble preservatives, such as chromated copper arsenate (CCA) and chromated copper borate (CCB), due to low toxicity to humans and the environment, and efficiency against wood biodeterioration by termites and rotting fungi (Monteiro *et al.* 2005; Tascioglu *et al.* 2012, 2013; Ogawa and Yazaki 2018). As a disadvantage, tannins are leachable due to extreme solubility with water (Tondi *et al.* 2013; Hu *et al.* 2017). However, they can be an alternative in the preservation of wood used in internal structures in construction, where contact with water is limited. To improve the biological resistance of thermo-mechanical densified pine wood, tannin extracts from *Acacia mearnsii* may be an alternative in the technological rout of lignocellulosic materials used in the manufacture of internal structures in construction.

This study evaluated the effect of tannin extracts of *Acacia mearnsii* incorporated along with the treatment of thermomechanical densified pinewood in relation to physical, mechanical, and biological resistance properties.

EXPERIMENTAL

Materials

The wood used was *Pinus elliottii* from a plantation located in Viçosa, Minas Gerais, Brazil (20º 45′ 14″ S, 42º 52′ 53″ W). The timber was felled by tangential cuts, from which 3 m long x 8 cm wide x 4 cm thick boards were removed (Fig. 1). From each board, 27 x 7 x 3.3 cm samples were taken (longitudinal, tangential, and radial), following the orientation of the growth rings, totaling 16 samples per treatment free of defects (knots and cracks).

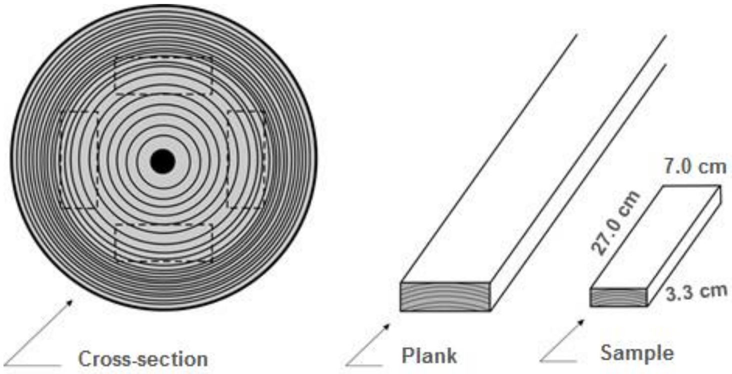


Fig. 1. Schematic of cutting and orientation of the wood samples. Adapted from Silva (2019)

Methods

The wood densification process was developed in three steps: acid hydrolysis, wood impregnation, and thermo-mechanical densification.

Acid hydrolysis

In a Parr reactor (Floor Stand Reactor) with an open circulation system and 18.75 L capacity, the samples were immersed in oxalic acid solution at a concentration of 25 g/L, at 120 °C for 90 min, based on previous studies (Pertuzzatti *et al.* 2018; Song *et al.* 2018; Silva *et al.* 2019) and preliminary tests. After treatment, the samples were washed in running water.

Wood impregnation

The pine samples submitted to acid hydrolysis were immersed in different concentrations (5, 10, and 15%) of tannin (manufacturer: Tanac®, Montenegro - RS, Brazil) powder, solubilized in distilled water, until the wood samples were completely saturated. The concentrations used were based on previous studies on the resistance to termite attack in treated wood (Tascioglu *et al.* 2012; Silveira *et al.* 2017; Yingprasert *et al.* 2021). After the preservative treatment, the samples were subjected to thermomechanical densification.

Thermomechanical densification

The woods removed from the tannin solutions were initially heated at 100 °C in water vapor in an autoclave, then transferred to a hydraulic press at 150 °C for 60 min, as established by Silva (2019). The pressing cycle was 30 min at a pressure of 3 atm, then the pressure was raised to 5 atm, remaining for another 30 min (Fig. 2). The pressing was carried out up to 40% (1.3 cm) of the initial sample thickness. At the end, the thermostat was turned off and the pressure was gradually reduced to ±0.1 atm, after which the samples remained in the press for another 40 min cooling to minimize defects. The samples were stored at 20 °C and 65% relative humidity until they reached the hygroscopic equilibrium moisture content to perform the tests.

Apparent density and X-ray densitometry

The bulk density ($g.cm^{-3}$) was determined by direct measurement with a pachymeter, dividing the mass (g) by the corresponding volume (cm^3), according to NBR 7190 (ABNT 1997) adapted due to the lower thickness dimension of the samples after thermomechanical densification. For X-ray bulk density analysis, the wood samples were cut 2 mm thick in a circular saw and conditioned in a climate controlled room at 20 °C and 65% relative humidity for 24 h according to Castro *et al.* (2020). The samples were inserted with the cellulose acetate calibration scale into the shielded compartment of the Faxitron digital X-ray equipment model LX-60 (manufacturer: Faxitron, Lincolnshire, England) previously calibrated for automatic reading, at 30 Kv for 19 s (Castro *et al.* 2020), and the digital images with ultra-contrast and resolution were saved in DICOM format (Faxitrom 2009). Bulk density profiles were constructed with the grayscale digital images, converted to rainbow scale in Adobe Photoshop software (Adobe, San Jose, CA, USA), and the calibration analyzed in ImageJ software (National Institutes of Health, Bethesda, MD, USA). In the grayscale images, shades closer to the colors of white and black were associated with higher and lower density, respectively. In the rainbow scale (color image) the shades of red, yellow, green, and blue were associated, in descending order, from highest to lowest density, respectively (Medeiros *et al.* 2020).

Mechanical properties of wood

The mechanical tests were performed according to NBR 7190 (ABNT 1997); however the thickness dimensions of the samples after densification were smaller than the dimensions suggested by the standard.

For the static bending test with the determination of the modulus of rupture (MOR) and modulus of elasticity (MOE), 12 test specimens of 27 cm length x 2 cm width x 1.3 cm thickness were used for each treatment, including the control (untreated and not densified wood). For the test of compression parallel to the grain, 12 specimens were used for each concentration of preservative treatment, with dimensions of 5 cm length x 2 cm width x 1.4 cm thickness. For the Janka hardness test, in the direction normal to the fibers, 8 specimens were used, with the dimensions 10 cm length x 5 cm width x 1.4 cm thickness.

Biological test with termites

The dry wood termite test was developed according to the method described by the Instituto de Pesquisas Tecnológicas do Estado de São Paulo, Divisão de Madeiras - IPT/DIMAD D - 2 (IPT 1980). Samples with adapted dimensions of 1.3 × 0.6 × 7.0 cm^3 (radial × tangential × longitudinal) were used for different concentrations of tannin extracts.

Each experimental unit consisted of a pair of samples, subjected to contact with 40 dry wood termites, 38 workers and two soldiers, of the species *Cryptotermes brevis* (Walker), family Kalotermitidae.

The experiment was kept for 45 days in laboratory conditions at 25 ± 2 °C and 65 ± 5% relative humidity. At the end, the mortality rate of the termites, number of holes, and the damage were evaluated by four examiners through a score ranging from 0 to 4, classifying as: (0) the sample that had no surface damage; (1) surface damage; (2) moderate damage; (3) severe damage; and (4) deep damage.

Statistical analysis

The experiment was set up in a completely randomized design with four treatments, one with wood *in natura* (control) and three concentrations (5, 10, and 15%) of tannins. The data were submitted to analysis of variance (ANOVA) at 5% significance level, and when there were significant differences, Tukey test was performed using the software SISVAR 5.8 (Ferreira 2011).

RESULTS AND DISCUSSION

X-ray Densitometry and Apparent Density

The average bulk density values of untreated and tannin extract treated and thermomechanically densified pine woods are presented in Fig. 2. There was a significant increase in the bulk density of the modified pine woods compared with the control. The apparent density increment of the modified woods treated with the 15% tannin concentration was 87.8% and, on average, 68.8% for those treated with the 5 and 10% tannin concentrations. These results are expected because the compression of wood by the thermomechanical densification process is responsible for the flattening of anatomical structures, with the reduction the empty spaces (lumens) and concentration of lignocellulosic mass per volumetric unit, having as a consequence, the formation of a more homogeneous material of higher density and mechanical strength (Laine *et al.* 2016; Pertuzatti *et al.* 2018; Silva 2019). With the high pressing temperature (150 °C), the lignin that constitutes the part of the wood and that is not removed during the pretreatment with oxalic acid (Silva *et al.* 2019) undergoes plasticizing, contributing to the molding and formation of the high-density wood.

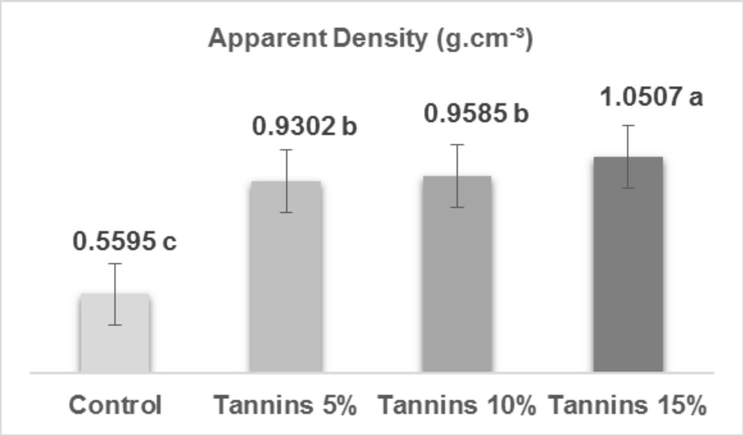


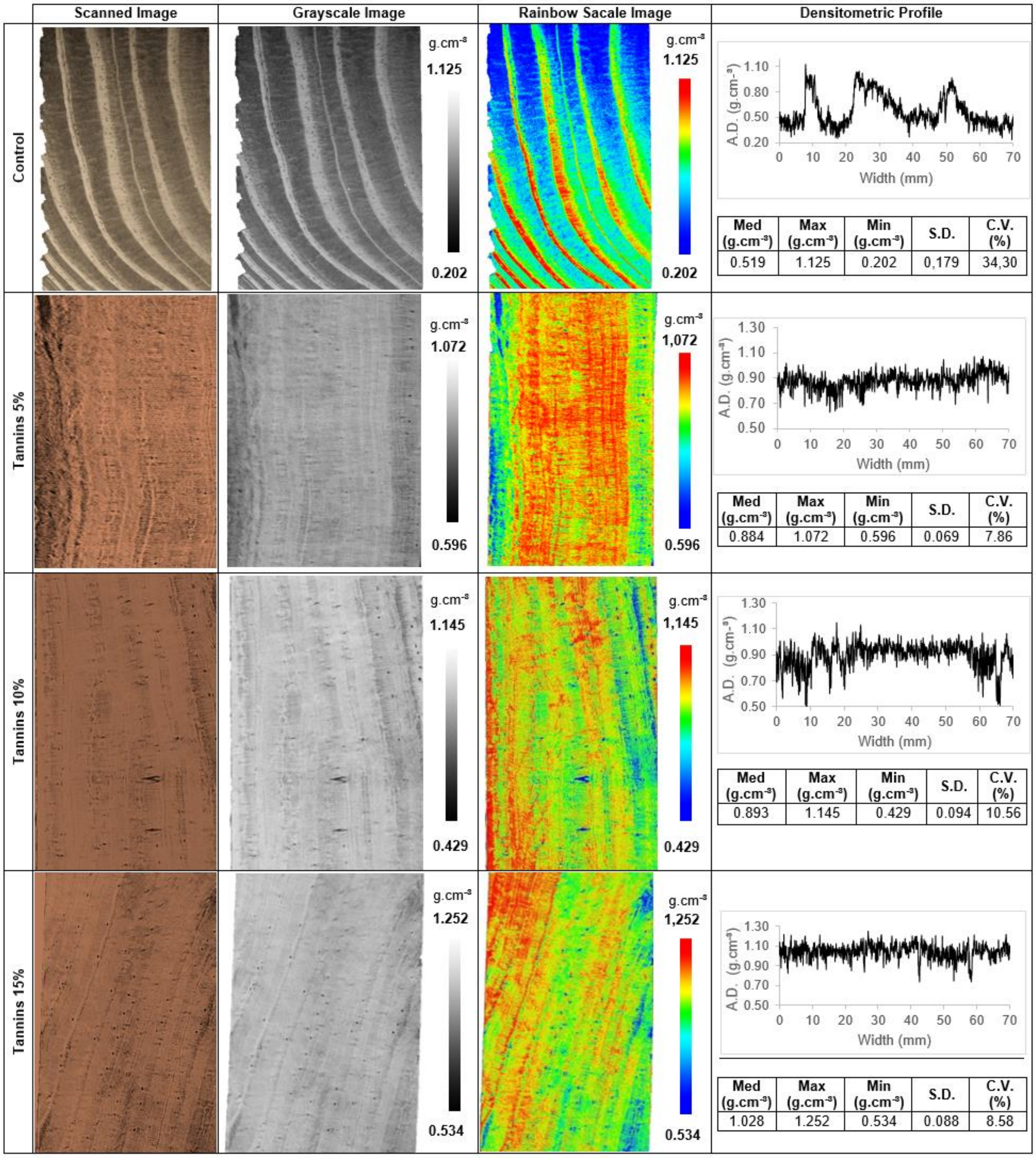
Fig. 2. Average bulk density of *Pinus elliottii* wood *in natura* (control) and modified by preservative treatment with tannins at 5, 10, and 15% and thermomechanical densification. Means followed by the same letter do not differ by the Tukey test (p>0.05).

For the modified woods, there was a significant difference between treatments, with a 10% increase in bulk density for modified woods treated with the highest concentration of tannins (15%), compared to woods treated with the lowest concentrations (5 and 10%). The treatment with acacia tannins may be responsible for the increase in the bulk density of the modified woods, since, the composition of the tannin extract consisting of tannins and high molecular weight non-tannin substances (Pizzi 1983) can influence the increase in mass of the preserved wood, due to the tendency to impregnate the tracheids and rays that make up the anatomical structure of pine wood, at the concentration and immersion time that were applied in study (Tondi *et al.* 2013).

In addition to the increase in density, another characteristic of modified wood is greater homogeneity. Through the analysis of the densitometric profile and X-ray images (Fig. 3) it is possible to observe the distribution of apparent density in *Pinus elliottii* wood *in natura* and modified by preservative treatment with tannins at 5, 10, and 15% and thermomechanical densification.

The grayscale X-ray image of *Pinus elliottii* in natura wood (control sample) showed regions with dark gray tone, whose apparent density is lower and can reach up to 0.202 g.cm^{-3} and regions with light gray tone, with apparent density of 1.125 g.cm^{-3}. In the grayscale X-ray images of the wood modified by tannin preservative treatment and by thermomechanical densification, the predominance of regions in light gray tones are characteristic of the higher density of the modified samples in relation to the control, whose maximum density was 1.072; 1.145; and 1.252 g.cm^{-3}, for the treatments with tannins at 5, 10, and 15%, respectively.

Similar behavior occurs in the rainbow scale images, in which the lower density regions of the wood are blue and the higher density regions are red. In the control sample, the tonal variation between areas of lower and higher density alternate following the changes of early (lower density – blue color) and late (higher density – red color) wood. In the rainbow scale images of the modified samples, this wood variation is not clear, and there is a predominance of red, yellow and green shades resulting from the higher density of the modified wood in relation to the *Pinus in natura* wood.



Med ($g.cm^{-3}$)	Max ($g.cm^{-3}$)	Min ($g.cm^{-3}$)	S.D.	C.V. (%)
0.519	1.125	0.202	0,179	34,30

Med ($g.cm^{-3}$)	Max ($g.cm^{-3}$)	Min ($g.cm^{-3}$)	S.D.	C.V. (%)
0.884	1.072	0.596	0.069	7.86

Med ($g.cm^{-3}$)	Max ($g.cm^{-3}$)	Min ($g.cm^{-3}$)	S.D.	C.V. (%)
0.893	1.145	0.429	0.094	10.56

Med ($g.cm^{-3}$)	Max ($g.cm^{-3}$)	Min ($g.cm^{-3}$)	S.D.	C.V. (%)
1.028	1.252	0.534	0.088	8.58

Fig. 3. X-ray images and densitometric profiles of *Pinus elliottii* wood *in natura* (control) and modified by preservative treatment with tannins at 5, 10, and 15% and by thermomechanical densification. Mean: average bulk density; Max: maximum bulk density; Min: minimum bulk density; S.D.: standard deviation; C.V.: coefficient of variation

In the pine wood *in natura*, the apparent density variations occur due to the differentiation of the early and late wood that are formed with the growth in diameter of the tree. In conifers, the regions of latewood are formed during the period of slow/unfavorable growth of the tree and have a characteristic thicker tracheid cell wall and smaller cell lumens (Trianoski *et al.* 2013; Marini *et al.* 2021), resulting in higher density in these areas. The initial wood is formed during the period of favorable growth of the tree, the tracheids have thinner cell wall and larger lumens (Trianoski *et al.* 2013), and its characteristic is the formation of regions of lower density in the wood. In modified wood this variation of the lignin is not clear due to the compression of the wood by the thermomechanical densification process, which results in the joining of the lignin and higher concentration of lignocellulosic mass per volumetric unit, forming a more homogeneous material with higher bulk density (Laine *et al.* 2016; Pertuzatti *et al.* 2018; Silva 2019).

In the densitometric profile of pine wood *in natura* (control), the regions of latewood (of higher density) are characterized by density peaks, which reached the maximum bulk density of 1.125 g.cm^{-3} (Fig. 3). In regions of earlywood containing tracheids with thinner cell wall and larger lumens, the density is lower, being represented in the profiles by the lowest valleys.

In the densitometric profiles of modified wood, the density peaks are characterized by greater homogeneity of the samples, with less variation between the areas of low and high apparent density, compared to the control sample. The lower coefficients of variation compared to the control sample (34.30%) also show the greater homogeneity of the modified woods (Fig. 3). Homogeneity can be a positive factor in the wood processing process, as it ensures similar physical and mechanical characteristics throughout the piece, resulting in lower defect propensities (Roszyk *et al.* 2020). In addition, the higher density of modified woods can qualify them for the production of furniture, flooring, and construction uses (Ulker *et al.* 2012; Pertuzzatti *et al.* 2018).

Mechanical Properties

The average values of static flexural strength, compression parallel to the grain, and Janka hardness of untreated and tannin extract treated and thermomechanically densified pine woods are presented in Table 1.

Table 1. Mechanical Properties of *Pinus elliottii* wood *in natura* (control) and Modified by Preservative Treatment with Tannins at 5%, 10%, and 15% and Thermomechanical Densification

Parameter	**Static bending**		**Parallel compression**	**Janka hardness**
	MOR (MPa)	MOE (MPa)	(MPa)	(Kgf.cm^{-2})
Control	67.59 b $^{(21.80)}$	2213.84 b $^{(19.46)}$	29.80 c $^{(5.13)}$	319.13 c $^{(14.57)}$
Tannins 5%	118.49 a $^{(39.98)}$	4821.25 a $^{(11.79)}$	52.95 b $^{(22.33)}$	572.50 b $^{(12.09)}$
Tannins 10%	74.03 b $^{(36.09)}$	4638.40 a $^{(14.63)}$	74.78 a $^{(16.54)}$	722.23 a $^{(19.53)}$
Tannins 15%	92.44 ab $^{(41.01)}$	4926.77 a $^{(15.32)}$	82.07 a $^{(14.88)}$	714.20 ab $^{(18.07)}$

MOR: modulus of rupture, MOE: modulus of elasticity. Means followed vertically by the same letter do not differ by the Tukey test (p>0.05) for the same variable. Values in parentheses refer to the coefficient of variation (%)

The modulus of rupture of the modified wood preserved with 5% tannins differed from the control pine wood (*in natura*), with an increase of 75%. For the modulus of elasticity, there was an average increase of 120% in modified wood compared to the control treatment. Thermomechanical densification is responsible for improving the mechanical performance of wood, resulting in increment in strength due to the disruption of the cell wall and alignment of cellulose nanofibers that increase hydrogen bonding and strengthen the wood structure (Song *et al.* 2018).

There was an increase in the parallel compressive strength of the modified pine woods, and the highest average increment, of 169% occurred for the woods treated with tannins at concentrations of 10 and 15% (Table 1), relative to the control. The rearrangement of anatomical elements after compaction, which result in a more homogeneous structure with a lower proportion of voids and a greater amount of lignocellulosic mass per unit volume, generating a material with a greater capacity to resist mechanical loads (Ulker *et al.* 2012; Pertuzzatti *et al.* 2018; Silva 2019). However, treatments with different concentrations of tannins can also contribute to the increase in parallel compressive strength, especially at higher contents, such as at concentrations of 10 and 15%. Increasing the content of extractives within the cell wall of tracheids can influence better mechanical properties in the transverse direction of the wood, such as compressive strength and hardness (Gündüz *et al.* 2011; Sommerauer *et al.* 2019).

In the modified wood treated with tannins at 10% the increment in hardness was 126.3% compared to wood without any treatment (control). With the thermomechanical densification process, parameters such as closing time and press temperature can influence the increase in hardness and density of the wood (Laine *et al*. 2013b). However, tannin treatment also has a positive impact on the process, since it was observed in the treatment of wood of the species *Fagus sylvatica* with preservative based on tannin copolymers, there was an increase in hardness of 45, 25, and 12% in the axial, radial and tangential directions, respectively (Sommerauer *et al.* 2019).

Biological Test with Termites

The results of the biological resistance to dry wood termite attack (*Cryptotermes brevis*) of untreated and tannin extract treated and thermomechanically densified pine woods are presented in Table 2. There was a 55% increase in the percentage of termite mortality for the tannin treatment at a concentration of 15% (Table 2) compared to untreated pine wood (control).

Table 2. Biological Resistance to Termite Attack of *Pinus elliottii* wood *in natura* (control) and Modified by Tannin Treatment at 5%, 10%, and 15% and Thermomechanical Densification

Parameter	Control	Tannins 5%	Tannins 10%	Tannins 15%
Mortality (%)	49.58 b $^{(18.16)}$	50.83 b $^{(34.17)}$	62.08 ab $^{(18.26)}$	76.67 a $^{(17.82)}$
Wear score	3.0 a $^{(28.45)}$	2.0 b $^{(39.01)}$	1.7 bc $^{(48.99)}$	1.2 c $^{(67.76)}$
Number of holes	3 a $^{(38.97)}$	2 ab $^{(41.83)}$	2 ab $^{(27.39)}$	1 b $^{(54.77)}$

Means followed by the same letter in the same row are statistically equal by Tukey's Test (p>0.05). Values in parentheses refer to the coefficient of variation of the samples.

The tannin treatment had an impact on the reduction of damage and the number of holes caused by termites. Tannins have toxicity to microorganisms and insects, and in their composition protocyanidins (which are polymers of flavan-3-ol and/or flavan-3,4-diol) are the termiticidal compounds with the ability to inactivate termite enzymes (Ohara *et al.* 1994; Monteiro *et al.* 2005; Ogawa and Yazaki 2018), favoring mortality and low level of wood damage. However, part of the composition of tannin extracts consists of non-tannin substances, formed by carbohydrates, hydrocolloid gums, and amino acid fractions (Pizzi 1983; Missio *et al.* 2017), which can reduce the complete efficiency (100% of average termite mortality, without wood damage) of the preservative substance in termite control.

In the raw pine wood, the damage is considered severe (level 3); and for the modified woods, there was a decrease in the level of damage to moderate in woods treated with tannins at 5 and 10%, and superficial for those treated with tannins at 15%.

In the *Pinus* wood *in natura* the damage is considered severe, with the presence of holes that exceed the thickness of the wood, galleries and superficial cavities; and for the samples treated with the 5 and 10% tannin concentrations the level of damage is moderate, with holes and cavities mainly in the region of the junction between the woods that form the sample (area where, probably, there is greater susceptibility to deterioration). For the wood treated with 15% tannins, the level of damage is superficial, with fewer holes and cavities.

In the wood modified with tannins at a 15% concentration, samples were found in which the mortality of termites was 100%, with no surface damage. This behavior, together with the lower number of holes and damage caused to the wood modified with the 15% tannin concentration, may show the efficiency of the preservative treatment for densified wood against *Cryptotermes brevis* termites.

CONCLUSIONS

1. Wood modification with preservative treatment with tannins (5, 10, and 15%) and thermomechanical densification improved the physical, mechanical and biological parameters of the caused *Pinus elliottii* wood, revealing itself as a potential technological route for the production of high performance lignocellulosic materials.
2. The modification provided an increase in the apparent density. With the X-ray densitometry technique the greater homogeneity of the modified samples was evidenced.
3. There was an increase in the mechanical strength of the modified woods, more evident for the woods treated with tannins (10 and 15%) in relation to the untreated woods. The parallel compressive strength had the highest average increase, 169%.
4. An increase in biological resistance to dry wood termites (*Cryptotermes brevis*) was observed mainly for the wood treated with the 15% tannin concentration, where termite mortality was higher (54%) and damage was lower compared to untreated wood.
5. The impregnation of tannin extracts into the modified wood improved its quality, demonstrating potential and efficiency in its use as a preservative.
6. Further studies should be carried out to evaluate the leaching resistance of the treatment with tannins in thermomechanically densified wood when in contact with moisture, and the modified wood resistance to rotting fungi.

ACKNOWLEDGEMENTS

This work was supported by the Brazilian agencies “Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq)” and “Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES)”, and by the Portuguese agency “Fundação para a Ciência e a Tecnologia (FCT)” through funding of the Forest Research Centre (UID/AGR/00239/2019 & UIDB/00239/2020) and CLEANPELL (EXPL/CTM-PAM/0850/2021). Solange Araújo acknowledges funding from FCT for their research contracts, DL 57/2016/CP1382/CT0018.

Competing Interests Declaration

No potential conflict of interest was reported by the authors.

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Article submitted: June 28, 2022; Peer review completed: August 6, 2022; Revised version received and accepted: November 6, 2022; Published: November 22, 2022.
DOI: 10.15376/biores.18.1.641-652