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Influence of chromated copper arsenate type-C concentration on the quality of treated fence posts

Influência da concentração de arseniato de cobre cromatado tipo C na qualidade de moirões tratados

Influencia de la concentración de arseniato de cobre cromado tipo C en la calidad de los postes tratados

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Abstract
This study aimed to reduce the concentration of chromated copper arsenate type-C (CCA-C) in preservative treatments of *Eucalyptus* sp. for use in applications with ground contact. The genetic materials used were *Eucalyptus cloeziana* of seminal origin and the clonal hybrid (*E. urophylla* x *E. camaldulensis*), which was derived from six year old crops at commercial plantations. The basic density, moisture content, percentage of sapwood, and wood treatable volume were determined, and the anatomical structures were characterized. The genetic materials were treated with concentrations of 1.2%, 1.3%, 1.4%, 1.5%, and 1.6% active ingredients (AI) of CCA-C. The quality of the preservative treatment was evaluated via penetration and retention tests. The treated wood volume of *E. cloeziana* exhibited 44% irregular partial penetration, 40% vascular penetration, 12% peripheral partial penetration, and 4% no penetration. For the hybrid, the wood volume exhibited 68% total penetration, 24% peripheral partial penetration, and 8% irregular partial penetration. The concentrations of 1.4% AI and 1.5% AI for *E. cloeziana* and of 1.4% AI and 1.6% AI for hybrid reached the minimum retention required by standard.
Keywords: Wood preservation; Preservative penetration and retention; Eucalyptus wood.

Resumo
Este trabalho teve como objetivo reduzir a concentração de arseniato de cobre cromado tipo-C (CCA-C) em tratamentos preservativos de *Eucalyptus* sp. para uso em contato com o solo. Os materiais genéticos utilizados foram *Eucalyptus cloeziana* de origem seminal e o híbrido clonal (*E. urophylla* x *E. camaldulensis*), oriundos de cultivos com seis anos de idade em plantios comerciais. A densidade básica, a umidade, a porcentagem de alburno e o volume tratável da madeira foram determinados e as estruturas anatômicas caracterizadas. Os materiais genéticos foram tratados com concentrações de 1,2%, 1,3%, 1,4%, 1,5% e 1,6% de ingredientes ativos (I.A.) de CCA-C. A qualidade do tratamento preservativo foi avaliada por meio de testes de penetração e retenção. O volume de madeira tratada do *E. cloeziana* apresentou 44% de penetração parcial irregular, 40% de penetração vascular, 12% de penetração parcial periférica e 4% sem penetração. Para o híbrido, o volume de madeira tratada apresentou 68% de penetração total, 24% de penetração parcial periférica e 8% de penetração parcial irregular. As concentrações de 1,4% I.A. e 1,5% I.A. para *E. cloeziana* e de 1,4% I.A. e 1,6% I.A. para híbrido atingiram a retenção mínima exigida pela norma.
Palavras-chave: Preservação da madeira; Penetração e retenção de preservativos; Madeira de eucalipto.

Resumen
Este trabajo tuvo como objetivo reducir la concentración de arseniato de cobre cromo tipo C (CCA-C) en tratamientos preservantes *de Eucalyptus* sp. para uso en contacto con el suelo. Los materiales genéticos utilizados fueron *Eucalyptus cloeziana* de origen seminal y el híbrido clonal (*E. urophylla* x *E. camaldulensis*), provenientes de cultivos de seis años en plantaciones comerciales. Se determinaron la densidad básica, la humedad, el porcentaje de albura y el volumen tratable de la madera y se caracterizaron las estructuras anatómicas. Los materiales genéticos fueron tratados con concentraciones de 1,2%, 1,3%, 1,4%, 1,5% y 1,6% de principios activos (I.A.) de CCA-C. La calidad del tratamiento conservante se evaluó mediante pruebas de penetración y retención. El volumen de madera tratada con *E. cloeziana* presentó 44% de penetración parcial irregular, 40% de penetración vascular, 12% de penetración parcial periférica y 4% sin penetración. Para el híbrido, el volumen de madera tratada presentó 68% de penetración total, 24% de penetración parcial periférica y 8% de penetración parcial irregular. Concentraciones de 1,4% I.A. y 1,5% I.A. para *E. cloeziana* y 1,4% I.A. y 1,6% I.A. para híbridos alcanzó la retención mínima exigida por la norma.
Palabras clave: Conservación de la madera; Penetración y retención de preservativos; Madera de eucalipto.

1. Introduction

Native forest woods are used in civil construction and rural buildings because they have natural resistance and durability. However, the use of this raw material source without proper replacement has led to scarcity. Therefore, it has been necessary to replace native woods with other rapidly grown materials from planted forests (Paes et al., 2001; Raminelli et al., 2019; Vivian et al., 2020).

Despite their abundance and rapid growth, reforestation woods have low natural durability, and treatment with preservatives is necessary to increase their resistance to attack by xylophagous organisms (Vivian et al., 2020).

In Brazil, the market for treated wood remains smaller than the pulp and paper, reconstituted panels, timber, and biomass energy industries. The production of wood intended for preservative treatment reached an estimated 1.4 million m^3 in 2018. The main products were fence posts, poles, and crossties (IBÁ, 2019).

Chromated copper arsenate type-C (CCA-C) is considered the best preservative and the most used in many countries. In the United States, it dominated the wood market between 1970 and 2004. Since then, its use was restricted to industrial applications, and its retail trade was prohibited. In Brazil, it is applied to approximately 90% of treated wood because it has lower leaching and better field performance when compared to other preservatives available on the market (Lebow, 2010).

Several technical standards define quality parameters for product marketing, and they highlight the penetration and retention performance of the preservative (Hunt; Garratt, 1967). The Brazilian Technical Standard NBR 9480 (ABNT, 2009) states that the preservative should penetrate 100% of the sapwood, and the retention should be at least 4.0 kg m^{-3}, 6.5 kg m^{-3}, or 9.6 kg m^{-3} for components that do not have contact with the ground (construction wood), those that have contact with the ground (fence post), and utility poles and crossties, respectively.

Several studies have evaluated the quality of wood treated with CCA-C. Fence posts made of *Eucalytpus urophylla* clones aged nine years treated with 1.5% active ingredients (A.I.) and 1.7% AI of CCA-C and compressed at 12 kgf cm^{-2} for 60 min achieved deep and regular fence post penetration (Amaral, 2012). In addition, Schneid et al. (2013) produced poles of approximately 18-year-old *E. saligna* and *E. cloeziana* treated with 2.0% AI of CCA-C and exposed them to 60 min, 90 min, and 120 min of pressures of 10 kgf cm^{-2} and 12 kgf cm^{-2}. They observed that only *E. saligna* poles treated with a pressure of 12 kgf cm^{-2} for 120 min reached the minimum retention (9.6 kg A.I. m^{-3}) required by NBR 9470 (ABNT, 2009).

Valle et al. (2013) investigated the penetration and retention in *E. urophylla* wood aged 5.3 years, treated with 2.0% A.I. CCA-C, and exposed to 90 min of 12 kgf cm^{-2} pressure. These procedures resulted in deep and regular penetration for all of the pieces treated with CCA-C and average retention above the minimum (6.5 kg A.I. m^{-3}) required by the Brazilian standard NBR 9480 (ABNT, 1986) (Valle *et al.* 2013). In their investigation of the retention of CCA-C in fence posts of *Corymbia torelliana* (11 years old) and *E. grandis* × *E. urophylla* (9 years old) treated with a concentration of

2.0% A.I. of CCA-C and exposed to 60 min under pressure, Lopes et al. (2017) noted that the average retention for both genetic materials was higher than the minimum requirement by the standard (6.5 kg A.I. m^{-3}).

To meet minimum retention requirements, higher CCA-C concentrations are used during treatment, which increases the price of treated wood because treatment usually constitutes 50% of the total production cost. As such, the preservative treatment is particularly costly. Research that aims to reduce the preservative concentration while meeting the requirements of relevant standards is essential both economically and environmentally. Therefore, the present study aimed to reduce the concentration of CCA-C in preservative treatments of *Eucalyptus* sp. for use in applications with ground contact.

2. Methodology

The genetic materials used were *Eucalyptus cloeziana* of seminal origin and the clonal hybrid (*E. urophylla* × *E. camaldulensis*), which was derived from six year old crops at commercial plantations. For each genetic material, 25 fence posts of 2.20 m in length and from 8 cm to 10 cm in diameter were collected randomly from the drying stacks of a forestry company's yard in Martinho Campos, Minas Gerais, Brazil.

From each fence post, 3 cm (base and top) were discarded to avoid impurities and cracks. A 5-cm portion was also removed at one of the ends to determine the basic density according to NBR 11941 (ABNT, 2003) and the moisture content as NBR 7190 (ABNT, 2010), and the sapwood percentage, treatable wood volume, vessel diameter, and frequency were measured.

To determine the percentage of sapwood and treatable wood volume, cross measurements of the total diameter and the heartwood diameter were performed on each disk with the aid of a ruler with a precision of 1 mm. The length of the fence posts was also measured. From these measurements, the percentage of sapwood and the treatable volume of fence posts were calculated. It was not possible to visually differentiate the heartwood from sapwood in *E. cloeziana*.

To determine the diameter and frequency of the vessel, temporary slides were made with histological sections of the analyzed material. The measurement of anatomical characteristics was performed according to the IAWA Committee (1989), using an Olympus BX41 microscope with an attached camera and WincellPRO image analysis software.

The resultant fence posts containing the same characteristics were treated with CCA-C in an autoclave. The product used was LIFEWOOD 60 composed of 28.5% hexavalent chromium (CrO_3), 11.10% bivalent copper (CuO), 20.40% pentavalent arsenic (As_2O_5), and 40% water, supplied by Koppers Performance Chemicals Brasil Comércio de Preservantes LTDA. For each preservative treatment, five fence posts of each genetic material were selected for quality testing. Each batch of fence posts was treated with a certain A.I. concentration (Table 1).

Table 1. Concentrations of active ingredients used in the preservative treatment.

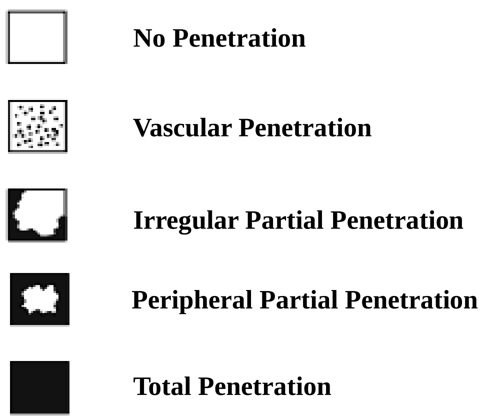
Genetic Material	Batch	Concentration of Active Ingredients (%)
Eucalyptus cloeziana	1	1.2
	2	1.3
	3	1.4
	4	1.5
	5	1.6
Clonal hybrid	1	1.2
	2	1.3
	3	1.4
	4	1.5
	5	1.6

Source: Authors.

The fence posts were inserted into normal production at the forestry company, which consisted of: i) 20 min of vacuuming to reach 515 mmHg and 30 min to maintain 515 mmHg; ii) injection of the preservative under 12 kgf cm^{-2} of pressure for 45 min; iii) 10 min of vacuuming to reach 200 mmHg. After treatment, the logs were transported to the outdoor storage yard and covered.

The treated fence posts were sampled by removing 5 cm discs from the centers of the sampled posts. The quality of the treatment was evaluated in accordance with NBR 6232 (ABNT, 2013), wich is responsible for standardizing the penetration and retention testes of the preservative in the wood. The penetration test was performed with colorimetric reactions, in which freshly cut discs were sprayed with a solution of chromoazurol S provided by Wood Biodegradation Laboratory of the Federal University of Espírito Santo, Brazil on all transverse surfaces. After drying, the surfaces were photographed and, based on the penetration patterns reported by Sales-Campos et al. (2003), they were grouped by three independent evaluators, and the average was calculated (Figure 1).

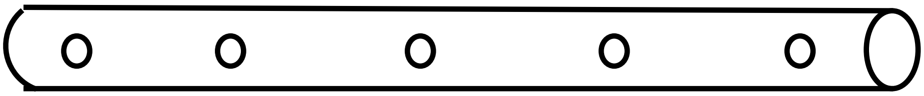
Figure 1. Penetration patterns by Sales-Campos *et al*. (2003).



Source: Sales-Campos et al. (2003).

For the retention test, 5 equidistant increments were removed from each fence post (Figure 2). They were then oven-dried at 103 ± 2 °C for 6 h. They were weighed (dry mass obtained through a precision scale) and the length and diameter (0.01 mm) were measured (using a digital caliper) for volume determination to calculate the anhydrous density (g cm^{-3}). Subsequently, the incremental positions were ground and sifted. The 40 mesh and 60 mesh samples were oven-dried (103 ± 2 °C) until reaching a constant weight. Then, they were transferred to plastic cuvettes and mounted with Mylar film TF-160 manufactured by Premier Lab Supply located in the city of Port St. Lucie, FL, USA, for X-ray analysis. CCA-C retention and the concentration of each active ingredient (copper, chromium, and arsenic) of the product in the analyzed samples were obtained by the Lab-X 3500 spectrometer manufactured by Oxford Instruments located in the city of Witney, Oxon, UK, using the anhydrous density as the input variable. CCA-C retention and the concentrations of chromium, copper, and arsenic were indicated by the spectrometer readings.

Figure. 2. Places in the fence posts where the increments were removed.



Source: Authors.

The physical parameters, anatomical parameters, and CCA-C penetration data were subjected to descriptive analysis. The CCA-C retention data were subjected to analysis of variance (ANOVA) and, when significant, the regression equations were fitted for the variables as a function of the CCA-C concentrations. The correlation between CCA-C retention and wood characteristics was also investigated.

Before the ANOVA, the normality of the data was verified *via* Kolmogorov-Smirnov test, and the homogeneity of the variances was verified *via* Cochran test. Statistical analyses were performed using the R software, version 3.2.4. (R Core Team, Vienna, Austria).

3. Results and Discussion

3.1 Analysis of wood characteristics

The minimum, average, and maximum values of the anatomical and physical parameters for the studied woods are presented in Table 2.

Eucalyptus cloeziana had the lowest average moisture content and the highest basic density, while the hybrid had the highest moisture content and the lowest basic density. The average moisture values found for both materials are by the recommendations of authors such as Santini (1988).

It was not possible to distinguish heartwood and sapwood in *Eucalyptus cloeziana*. Regarding the diameter of the vessels, the results found in this study for both materials are in agreement with results observed by other authors such as Evangelista (2011). However, to the frequency of vessels, the results found here for the analyzed materials differ from the results of the cited author. This difference was probably due to the particularities of the materials, such as, for example, the water regime of the place where the evaluated genetic materials were planted.

Table 2. Values of the physical and anatomical parameters for *E. cloeziana* and the Clonal Hybrid.

Parameters	*Eucalyptus cloeziana*			Clonal Hybrid		
	Min.	Avg.	Max.	Min.	Avg.	Max.
Percentage of Sapwood (%)	N.D.	N.D.	N.D.	38.37	48.41 (7.80)	72.73
Treatable Volume (m^3)	N.D.	N.D.	N.D.	0.003	0.004 (0.0007)	0.006
Vessel Diameter (μm)	33.92	102.87 (19.04)	150.46	31.05	123.80 (22.58)	170.32
Vessel Frequency (vessels × mm^{-2})	27.00	39.13 (7.36)	58.00	17.00	25.79 (4.78)	37.00
Moisture Content (%)	14.50	16.20 (1.10)	18.80	14.70	19.20 (2.42)	24.70
Basic Density (g cm^{-3})	0.492	0.602 (0.05)	0.703	0.414	0.468 (0.04)	0.596

The values in parentheses are the standard deviations (SD); N.D. = Not Distinct. Source: Authors.

3.2 Penetration test of the CCA-C in wood

The wood volume of *E. cloeziana* exhibited 44% irregular partial penetration, 40% vascular penetration, 12% peripheral partial penetration, and 4% no penetration, which was in accordance with the patterns reported by Sales-Campos et al. (2003). These results are similar to those found by Chagas et al. (2015) in *Tectona grandis* wood. However, these results differed from those observed by Schneid et al. (2013) who used greater times and different pressure values in *Eucalyptus saligna* and *Eucalyptus cloeziana* wood poles.

For the clonal hybrid, the wood volume exhibited 68% total penetration, 24% peripheral partial penetration, and 8% irregular partial penetration, which was similar to the results obtained by Amaral (2012) and Valle et al. (2013) for a clone of *E. urophylla*.

The insufficient penetration in *E. cloeziana* may have been due to its higher vessel frequency per square millimeter, as its vessels showed a smaller average diameter (Table 2). The reuse of preservative solution increases impurities in the CCA-C solution, which can produce a large number of particles that make penetration difficult.

3.3 Retention test of the CCA-C in wood

For *E. cloeziana*, the lowest (4.3 kg m^{-3}) and the highest retention values (7.5 kg m^{-3}) were observed at concentrations of 1.3% and 1.5% A.I. of CCA-C, respectively. For the clonal hybrid, the lowest (4.6 kg m^{-3}) and the highest retention values (7.4 kg m^{-3}) were observed at concentrations of 1.2% and 1.4% A.I. of CCA-C, respectively. For *E. cloeziana*, the lowest variability in retention values was observed at a concentration of 1.3% A.I. of CCA-C, and the greatest variability was observed at a concentration of 1.5% A.I. of CCA-C. For the clonal hybrid, 1.6% and 1.3% A.I. of CCA-C concentrations had the lowest and highest variability, respectively (Table 3).

Table 3. Average Values of Retention at Different Concentrations of Active Ingredients for *E. cloeziana* and Clonal Hybrid.

Genetic Material	CCA-C Retention (kg m^{-3})/Concentration of Active Ingredients (%)				
	1.2	**1.3**	**1.4**	**1.5**	**1.6**
Eucalyptus cloeziana	5.8 (0.36)	4.6 (0.20)	6.5 (0.37)	6.5 (1.01)	5.8 (0.63)
Clonal hybrid	4.9 (0.21)	5.8 (0.42)	6.8 (0.43)	6.2 (0.34)	6.6 (0.17)

The values in parentheses are the standard deviations (SD). Source: Authors.

For *E. cloeziana*, the lowest average retention (4.6 kg m^{-3}) was observed in fence posts treated with 1.3% A.I. of CCA-C concentration, and the materials treated with 1.4% and 1.5% A.I. of CCA-C showed the highest average retention (6.5 kg m^{-3}) and reached the minimum retention required by NBR 9480 (ABNT, 2009) for use in applications with ground contact. For other concentrations, the fence posts did not meet the required minimum for applications with ground contact. These materials should be used in structures without ground contact that require a minimum retention of only 4.0 kg m^{-3}. These results differed from the data obtained by Lopes et al. (2018) for 11 year old *Corymbia torelliana* treated with 2.0% A.I. of CCA-C and exposed to pressure for 60 min. However, the conditions employed by Lopes et al. (2018) increase the cost of preservative treatment.

For the clonal hybrid, the lowest average retention (4.9 kg m^{-3}) was observed in fence posts treated with 1.2% A.I. of CCA-C and the highest average retention (6.8 kg m^{-3}) was observed for fence posts treated with 1.4% A.I. of CCA-C. The fence posts treated with 1.4% and 1.6% A.I. of CCA-C reached the minimum prescribed by NBR 9480 (ABNT, 2009) for use in applications with ground contact. Wood treated with the other concentrations should be used in applications without ground contact. These results differed from the data obtained by Valle et al. (2013) on *E. urophylla* wood treated with 2.0% A.I. of CCA-C and exposed to 90 min of pressure and the results found by Arantes et al. (2017) who treated *Eucalyptus urophylla* x *Eucalyptus grandis* fence posts with 1,7% A.I. of CCA-C.

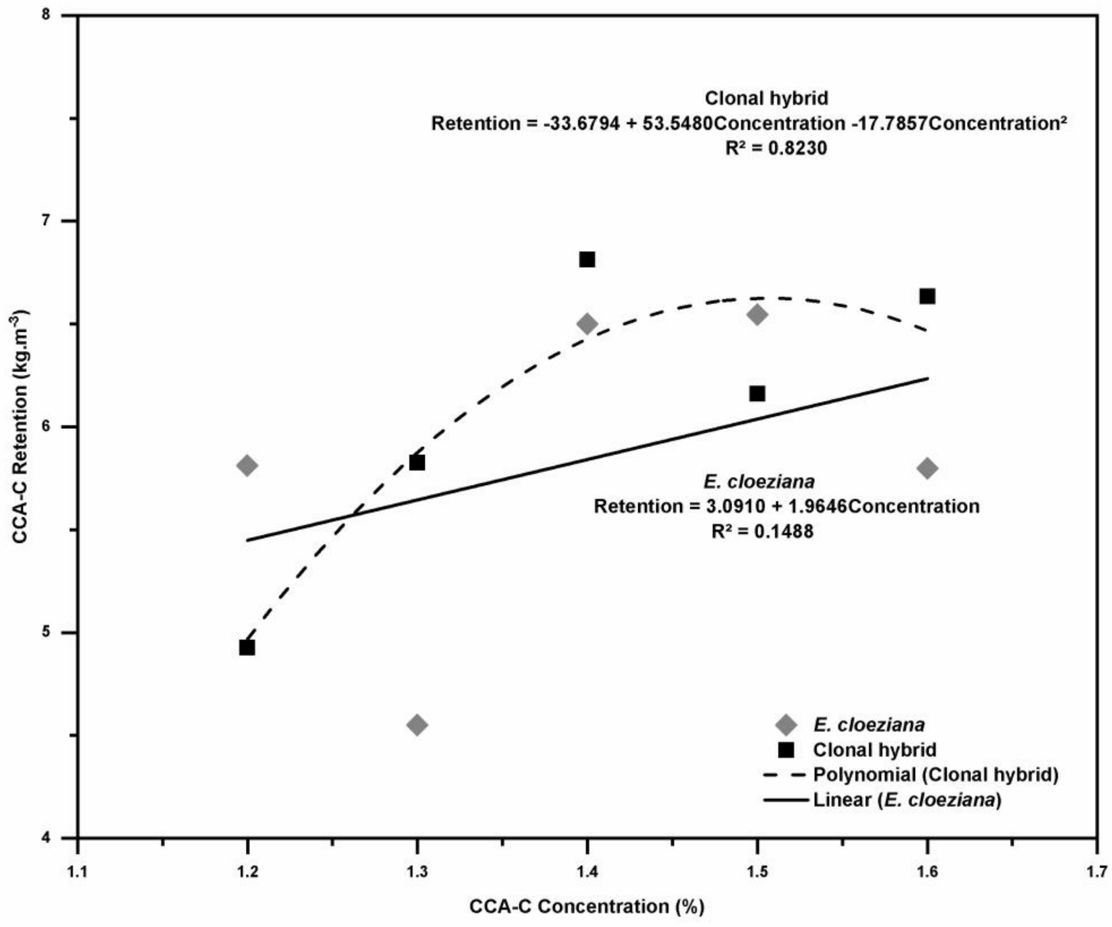
For both genetic materials, the retentions below the minimum stipulated by NBR 9480 (ABNT, 2009) may have been due to poor distribution or penetration of preservative in the fence posts.

3.4 Functional relation between retention and concentration of the CCA-C

When the equations fitted for *E. cloeziana* were analyzed, the linear model (Figure 3) was found to be significant, and it showed the best data behavior. The determination coefficient (R^2), indicated that approximately 15% of the total retention variation could be explained by variation in the CCA-C concentration.

The fitted equations for the clonal hybrid showed that the relationship between retention and concentration exhibited better behavior with the quadratic model (Figure 3). The determination coefficient indicated that 82% of the total variation in the CCA-C retention could be explained by variation in the CCA-C concentration.

Figure 3. Scatter plot of the retention variation as a function of CCA-C concentrations.



Source: Authors.

Analysis of the correlations between retention and moisture content and between retention and wood basic density for *E. cloeziana* and the clonal hybrid found no significant correlations among the variables. However, for the clonal hybrid, correlation coefficients were 0.44 and 0.47 when the retention was associated with the percentage of sapwood and with the treatable volume, respectively.

4. Conclusion

For the *E. cloeziana* fence posts treated with concentrations of 1.4% and 1.5% A.I. of CCA-C, the retentions were satisfactory for use in applications with soil contact according to NBR 9480 Standard (ABNT, 2009). In addition, for the clonal hybrid, woods treated with 1.4% and 1.6% A.I. of CCA-C also meet the requirements for use in applications with ground contact as per the standard. The other concentrations used reached the minimum stipulated for use in structures without contact with the ground according to the standard.

It is recommended to carry out tests with xylophagous organisms using the same genetic materials used in this study and with the same treatment parameters to evaluate the toxicity conferred on wood by the preservative products to these organisms. Moreover, it is recommended preservative treatment in woods with greater moisture and diameter variation to observe the influence of these variables on solution retention.

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