

Articles

Association of tannin with pyroligneous liquor as a natural preservative for *Eucalyptus* sp. wood

Associação do tanino ao licor pirolenhoso como preservante natural para a madeira de *Eucalyptus* sp.

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ABSTRACT

This study aimed to evaluate the potential use of tannin and pyroligneous liquor as a natural preservative for *Eucalyptus* sp. wood. Three trees, approximately 10 years old, were felled, and logs were removed and specimens were prepared for application of 4 treatments (control, pyroligneous liquor, liquor and tannin, and chromated copper borate). The treated and untreated logs were exposed to soil in a decay field for a period of 120 days. Chemical analyses: mass loss, penetration, retention and leaching of preservative, visual classification of specimens and static bending tests were performed. The specimens were installed in a decay field and subsequently collected for analysis and determination of properties. The analyses of bulk density at 12% moisture content, static bending, solubility in sodium hydroxide, preservative absorption, and solubility in cold water demonstrated that there was interaction between the factors and statistical differences between and within the treatments. It was concluded that the treatment of *Eucalyptus* sp. with pyroligneous liquor and tannin, in comparison to the control and to the woods treated only with pyroligneous liquor, was efficient mainly for the property of resistance to static flexure and bulk density, showing itself to be a potential preservative product.

Keywords: Decay field; Wood durability; Natural preservatives; Mechanical resistance

RESUMO

Neste trabalho objetivou-se avaliar o potencial uso do tanino e do licor pirolenhoso como preservativo natural da madeira de *Eucalyptus* sp. em exposição ao solo em um campo de apodrecimento durante um período de 120 dias. Foram abatidas três árvores com cerca de 10 anos, das quais foram retiradas toras e confeccionados os corpos de prova para aplicação de 4 tratamentos (testemunha, licor pirolenhoso, licor e tanino e borato de cobre cromatado). Foram realizadas análises químicas e de perda de massa; penetração, retenção e lixiviação do preservativo, classificação visual dos corpos de prova e ensaios de flexão estática. Os corpos de prova foram instalados em campo de apodrecimento e posteriormente coletados para as análises e determinação das propriedades. As análises da densidade aparente a 12% de umidade, resistência à flexão, solubilidade em hidróxido de sódio, absorção do preservativo e solubilidade em água fria demonstraram que houve interação entre os fatores e diferenças estatísticas entre e dentro dos tratamentos. Concluiu-se que o tratamento de *Eucalyptus* sp. com licor pirolenhoso e tanino, em comparação à testemunha e às madeiras tratadas apenas com licor pirolenhoso, foi eficiente principalmente para a propriedade de resistência à flexão estática e densidade aparente a 12% de umidade, mostrando-se um potencial produto preservativo.

Palavras-chave: Campo de apodrecimento; Durabilidade da madeira; Preservativos naturais; Resistência mecânica

1 INTRODUCTION

Due to its high adaptability and rapid growth, *Eucalyptus* spp. is currently the most widely used genus for reforestation purposes in Brazil and other countries such as India, South Africa, Argentina, Portugal, Angola, Spain, China, France, and Japan. It is also widely used in construction, pole production, laminating, plywood, pallets, room decorating, the automotive industry, and charcoal production (Arantes et al., 2024; Moraes et al., 2024).

Among the more than 700 species of *Eucalyptus*, many have high natural durability wood, maintaining their properties for a long period. At the same time, wood has characteristics that make it stand out from other materials. However, since it is an organic material, all wood is subject to deterioration by fungi, bacteria, insects, weathering, fire, and other chemical, physical, and biological agents (Giorgi et al., 2020; Crisp et al., 2024).

To mitigate this biotic or abiotic deterioration process and increase the useful life of the wood, it is common to use chemical preservatives to treat *Eucalyptus* spp. wood,

such as creosote oil, chromated copper arsenate (CCA), and chromated copper borate (CCB). Despite their proven effectiveness in protecting wood, using such preservatives can damage the environment and the health of producers and consumers of treated wood. For this reason, the aim is to develop technology capable of generating products that are less aggressive and harmful to the environment, known as natural preservatives (Järvinen et al., 2022).

Furthermore, the toxicity present in these compounds, especially CCA, has led to their use being regulated and even restricted in several countries. The main reason for this is the leaching of products considered carcinogenic and/or toxic to the environment present in these components, such as arsenic, which through leaching is capable of contaminating both soil and surface water (Morais et al., 2021).

Natural preservatives are products obtained from plant extracts that aim to increase wood's durability without chemical additives. In Brazil, only one commercial wood preservative product with proven fungicidal action uses a renewable natural resource, tannin, as its active ingredient (IBAMA 2024). In recent years, research has been published in several countries on the effectiveness of tannin as a biocide and boron fixative in the formulation of wood preservatives (Thevenon et al., 2010; Tondi et al., 2012).

Tannin ($C_{76}H_{52}O_{46}$) is an organic compound found in various plant species whose insecticidal and antifungal action helps protect against pathogens and deteriorating agents, giving wood greater natural resistance. Studies using tannin-based extractives extracted from *Acacia podalyriifolia* G. Don have obtained significant results regarding their effectiveness in controlling termites in *Pinus* sp. specimens (Souza; Demenighi, 2017; Militão et al., 2021).

Pyroligneous liquor, composed mainly of pyroligneous acid and tar resulting from the condensation of gases from the wood carbonization process, i.e., charcoal production, is a potential preservative product. Pyroligneous liquor is a natural product that, despite having various uses such as fungicidal activity, is normally discarded in

the charcoal production process and is released into the environment in the form of smoke, causing pollution and waste. Therefore, its use as a wood preservative can contribute to the solution of two environmental problems: the preservation of wood with natural products and the reduction of pollution resulting from its disposal in the charcoal production process (Hu et al., 2022).

Some treatment methods widely used in wood preservation industries are Bethell and Burnett, in which wood logs are stacked in chambers and temperature and pressure are applied (Castro et al., 2021). This method is effective but not very accessible to small and medium-sized farmers who do not have the equipment and sufficient demand to justify the necessary investment

Few studies in the scientific literature evaluate the performance of natural products in wood preservation such as the use of pyroligneous liquor and tannin (Leng et al., 2024; Moraes et al., 2025). Therefore, this research aimed to evaluate the potential use of pyroligneous liquor and tannin as natural preservatives and their influence on the physical and mechanical properties of *Eucalyptus* sp. wood.

2 MATERIALS AND METHODS

The experiment was conducted at the Wood Technology Laboratory of the UFT—Federal University of Tocantins, Gurupi Campus, state of Tocantins, Brazil, located at 11°43' S and 49°04' W. According to the Köeppen classification, the climate is tropical (Aw), with an average annual rainfall of 1617 mm and an average monthly temperature between 19°C [66,2°F] and 36°C [96,8°F] (Alvares et al., 2013).

The experiment was conducted using a completely randomized statistical design (DIC) with a 4 x 2 factorial, in which four wood treatments were separated (without preservative, with pyroligneous liquor, with pyroligneous liquor + tannin, and CCB) at two exposure times (0 and 4 months).

Wood from a 10-year-old *Eucalyptus* sp. clonal plantation in the southern region of Tocantins was used. Three trees with an average height of 25 meters (82'02") and

an average diameter of 31.6 cm (1'037") were felled. Logs measuring around 50 cm of length (1'64") were removed from the base logs, from which 80 specimens measuring 20 x 30 x 50 mm (width x height x length, respectively) were made to determine the bulk density at 12% moisture content of the wood based on the NBR 7190 standard (ABNT, 1997) and with dimensions of 20 x 20 x 300 mm (width x height x length) for the static bending test, following the COPANT 555 standard (COPANT, 2007).

The specimens were used to analyze mass loss, penetration, retention, leaching, and visual classification before and after exposure in the rotting field. The penetration and retention analyses of the preservatives were carried out following standard EN 1250-2 (EN, 1995), and the visual classification of attacks by xylophagous organisms was carried out following standard AWWA E1-17 (AWWA, 2017).

The chemical components used to formulate the CCB solution were obtained from the Federal University of Tocantins-UFT warehouse and agricultural product stores. The solution was prepared according to the standard NBR 9480 (ABNT, 2009) using a 2.0% concentration of the preservative solution (Embrapa, 2004). This protocol recommends the addition of acetic acid to the water to acidify the solution and facilitate the dilution of the other components. In addition, potassium dichromate, boric acid, and copper sulfate were added in the recommended proportions until complete dilution.

The pyrolysis process obtained the pyroligneous liquor at the Laboratório de Tecnologia e Utilização de Produtos Florestais I-UFT (Forest Products Technology and Utilization Laboratory I-UFT) during the carbonization of *Eucalyptus* wood for charcoal production. To treat this compound, 10% concentrated liquor was used with 8 liters of water. The tannin used was donated by the company TANAC—Unidade Tanino de Montenegro, based in the state of Rio Grande do Sul. It is obtained from species of the *Acacia* genus.

For the composition of the treatment with pyroligneous liquor and tannin, 5% tannin and 5% pyroligneous liquor diluted in the same amount of water used in the

composition of the treatments described above were used. The method used in this research was immersion, in which the wood is immersed for a period of 14 days until the product is sufficiently absorbed.

All the specimens were sent to the oven at 60°C for acclimatization for 12 hours, and then the temperature was raised to 103°C (217,4°F) until they were completely dry. Immediately after complete drying, the specimens were subjected to the treatments and remained immersed in the preservative solutions for 21 days. After this period, the specimens were returned to the oven until completely dry and then taken to the open field. It should be noted that, following standard EN 350 (EN, 2016), the untreated specimens were used as witnesses to analyze the wood's natural durability.

The specimens were chopped, crushed, and ground for the chemical analysis. Therefore, the particles used were those that passed through the 40-mesh sieve and were retained on the 60-mesh sieve according to the standard pre-established by the Technical Association of the Pulp and Paper Industry (TAPPI, 1999). Solubility in cold water and NaOH was quantified before and after exposure to the decay field according to the standards established by Wastowski (2018). To do this, the particles were weighed and immersed in NaOH in a water bath for one hour and in cold water for 48 hours in the other test, filtered, and dried to determine these parameters (TAPPI, 1999).

The flexural strength analyses were conducted at the Instituto Federal do Tocantins, campus de Palmas, Brazil (Federal Institute of Tocantins's Palmas campus Materials Testing Laboratory) using the 4.500 Kgf capacity EMIC Universal Testing Machine. Five samples of each treatment were tested before and after exposure to assess mechanical strength.

The decay field was set up in an open area with little vegetation cover (Figure 1). According to each treatment, the samples were mounted on grids (some in direct contact with the soil and others arranged on top of them, i.e., without contact with the soil) and remained exposed for around 120 days.

Figure 1 – Rotting field and arrangement of specimens



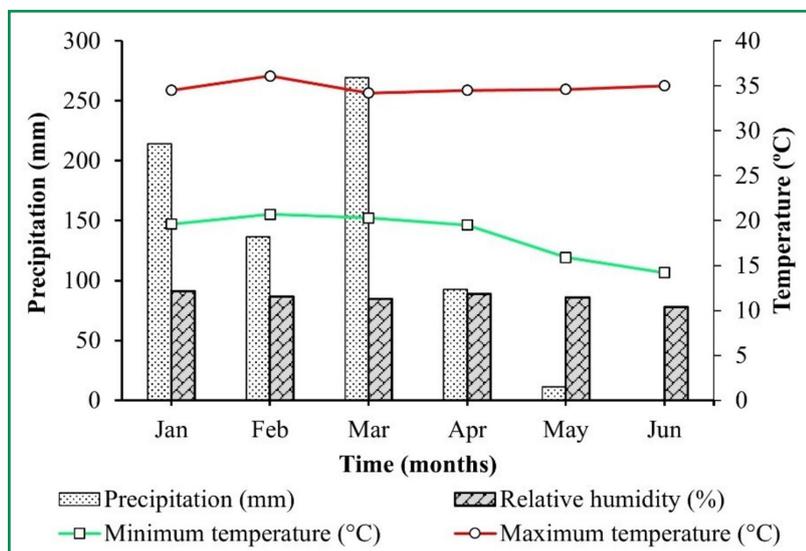
Source: Authors (2024)

During the exposure period, inspections were carried out to check for the presence of xylophagous and/or fungal agents in the area where the specimens were exposed. Weather conditions in the region during the exposure time were checked on the website of the Instituto Nacional de Meteorologia (National Institute of Meteorology), station A019, which is close to the experiment site (Figure 2).

Before and after each exposure period, the specimens were removed, inspected, dried at 103°C (217,4°F) until 0% dry, and subjected to analysis of bulk density at 12% moisture content, static flexural strength, mass loss, solubility in NaOH and cold water (leaching) and visual classification using the same procedures described above.

Statistical analysis was carried out using the Shapiro-Wilk test, followed by analysis of variance (ANOVA). For multiple comparisons of means using the SISVAR program, Tukey's test at the 5% significance level was used (Ferreira, 2011). Pearson's correlation coefficient was determined according to the treatment averages of the variables analyzed. The graphs were generated with the "ggplot2" package (Wickham, 2016), and the statistical analyses and results depend on the "agricolae" package (Mendiburu, 2017).

Figure 2 – Meteorological conditions of the period correspond to the exposure time of the specimens



Source: Authors (2024)

3 RESULTS AND DISCUSSIONS

Figure 2 shows the visual classification of the witness specimens - untreated (Figure 3-A), treated with CCB (Figure 3-B), pyroligneous liquor (Figure 3-C), and liquor and tannin (Figure 3-D). Traces indicating attack by xylophagous agents can be seen along the entire length of the specimens, mainly termites. In addition, more visible dark spots were noted on the untreated wood (witness), indicating the existence and greater preference for fungi.

At the end of the exposure time, the untreated wood showed a loss of around 8% in bulk density. On the other hand, the samples treated with pyroligneous liquor + tannin and those treated only with pyroligneous liquor showed a similar loss in density (5.25% and 5.20%, respectively), while those treated with CCB showed a loss of 1.35%. Segundinho et al. (2021) analyzed the bulk density of *Eucalyptus grandis* × *Eucalyptus urophylla* hybrids and found similar values to those observed in this study in treated and untreated samples, as well as Lopes et al. (2023) and Carvalho et al. (2023) when analyzed the density of *Corymbia* and *Eucalyptus* clones and *Eucalyptus* sp., respectively.

Figure 3 – Visual classification of the treated and witness (untreated) specimens after the last exposure time: A - untreated specimens; B - treated with CCB; C - treated with pyroligneous liquor and tannin; and D - treated only with pyroligneous liquor



Source: Authors (2024)

Table 1 shows the bulk density, flexural strength, and NaOH solubility values of treated and untreated *Eucalyptus* sp. wood.

The untreated wood had a greater loss of bulk density, which may indicate greater deterioration due to attack by xylophagous agents and/or severe weather conditions, such as the severe drought and high temperatures in the study region in the last two months of exposure.

According to Ogawa and Yazaki (2018), acacia tannins are composed of polyflavanones with molecular weights ranging from 300 to 3,000 g/mol, depending on the degree of polymerization. This high molecular weight may have influenced the observed density, as these polymers occupy the void spaces of the wood and contribute to an increase in density of the samples treated with pyroligneous liquor and tannin. However, the compounds found in pyroligneous acid have a low molecular weight (Jindo et al., 2022). However, it does contain tar, which is a highly viscous substance. After drying, this viscosity is retained in the wood, which may also affect the density of samples treated with this product.

Table 1 – Average values for bulk density, flexural strength, and NaOH solubility of *Eucalyptus* wood, untreated and treated with liquor, liquor and tannin, and CCB

Parameters <i>Eucalyptus</i> sp. wood	Treatment	Time (months)		Losses (%)	Pr>Fc Interaction
		0	4		
Bulk density (Kg/m ³)	No treatment	561.01 aC (5.31)	519.76 bC (1.78)	7.94	*
	Liquor	589.45 aA (5.68)	560.05 bB (8.43)	5.25	
	Liquor and tannin	584.74 aAB (4.78)	555.84 bBC (5.30)	5.20	
	CCB	574.43 aB (6.70)	566.77 bA (3.91)	1.35	
Static bending (MPa)	No treatment	97.31 aA (1.66)	63.24 bC (11.46)	53.87	*
	Liquor	93.65 aB (6.01)	67.07 bB (3.05)	39.63	
	Liquor and tannin	75.74 bC (6.59)	66.16 bB (6.08)	14.48	
	CCB	96.51 aA (4.66)	76.47 bA (15.17)	26.21	
NaOH solubility (%)	No treatment	14.33 bD (0.70)	23.95 aB (0.43)	67.13	*
	Liquor	23.47 bB (2.32)	24.05 aB (0.69)	2.47	
	Liquor and tannin	24.50 bA (0.31)	25.69 aA (1.3)	4.86	
	CCB	21.57 aC (0.61)	21.98 aC (1.93)	1.90	

Source: Authors (2024)

In where: Averages followed by the same lowercase letter in the column and uppercase letter in the row do not differ statistically from each other according to the Tukey test at the 5% probability level. * Significant at 5% probability level ($0.01 \leq p < 0.05$). Values in brackets correspond to the coefficient of variation (%).

The modulus of elasticity (MOE) and modulus of rupture (MOR) tests indicated the mechanical strength of this study. In this case, the wood's resistance to deformation (EOM) and rupture (MOR) when subjected to a predetermined force is evaluated. The untreated wood showed 97.31 MPa at time 0 and 63.24 MPa at the

end of the exposure time, showing a strength loss of 53.87%. The other treatments had significantly lower losses than untreated wood, especially those treated with liquor + tannin, whose values fell by 14% compared to the first time (75.74 MPa in month 0 and 66.16 MPa in month 4).

Also, it was observed that among the evaluated treatments, the samples treated with pyroligneous liquor combined with tannin and CCB showed lower loss of resistance compared to those treated with liquor alone. It is noteworthy, however, that the samples treated with liquor and tannin initially exhibited lower values for this property, which can be attributed to the high reactivity of tannin in acidic environments.

According to Osman (2012), tannins extracted from *Acacia nilotica* exhibit high reactivity at low pH, promoting accelerated autocondensation reactions at elevated temperatures. This process results in the formation of dense and highly cross-linked polymer networks that, although contributing to protection against degrading agents, may compromise the elasticity of the wood, thus explaining the initial reduction in flexural strength observed in this treatment.

Bulk density at 12% moisture content strongly correlates with the mechanical properties of *Eucalyptus* wood, so the higher the density, the greater its strength (Marini et al., 2022). The proposed treatments bear this out since the bulk density at 12% moisture content between the treatments decreased over time, consequently reducing the resistance. Since solubility in NaOH makes it possible to assess how badly the fungal attack has damaged the wood, it can be seen that the untreated specimens showed a 14.33% loss in solubility in the first month, reaching 23.95% at the end of the experiment, which shows a loss of 67.13%. On the other hand, the samples treated with pyroligneous liquor + tannin had 23.47% and 24.50% solubility in the first month and 24.05% and 25.69% in the fourth month, respectively. This shows that the treated wood had a low preference for fungi over the exposure time.

Table 2 shows the average preservative absorption and solubility values in cold water. The specimens treated with liquor, liquor tannin, and CCB absorbed 41.79%,

27.97%, and 21.31% of the preservative solutions to which they were exposed, respectively. There was greater absorption in the samples treated with liquor and low absorption in the samples treated with CCB.

The low absorption observed is due to the application method used (Dip-treatment). However, it is important to note that treatments with CCB as well as less aggressive preservatives such as micronised copper have a low absorption depending on the method and intensity of application. This limitation can have a negative impact on the long-term effectiveness of the treatment and reduce the durability and overall efficiency of wood protection against biodegrading agents (Nguyen et al., 2013; Moraes et al., 2025).

Regarding solubility in cold water, the samples of untreated wood, treated with pyroligneous liquor, and treated with liquor and tannin, showed 5.61%, 5.63%, and 4.65% losses at time 0. At the end of the fourth month, both were 5.06%, 5.19%, and 3.97%. The specimens treated with CCB initially obtained 7.90% and, at the end of the exposure time, had 3.41% losses.

Solubility in cold water favors the loss of preservative components that have not been sufficiently absorbed during treatment. As can be seen with untreated wood, there was little difference between the exposure times, with only total extractives leaching. On the other hand, CCB showed high leaching in the latter. This can be explained by the low impregnation of the CCB components, which remained superficial and were lost during the exposure process. Thus, the results presented allow us to conclude that the untreated wood showed greater deterioration due to the site's soil and climate conditions, as did the samples treated with CCB.

High rainfall rates were observed during the first three months of exposure, which favored the loss of surface components in the rain. The month of March saw rainfall of around 270 mm, which, combined with the low impregnation of the CCB, contributed significantly to leaching, reducing protection and making the treated wood subject to attack by xylophagous agents. Another reason for this situation is the

presence of boron in the composition of CCB, which is less resistant than the arsenic component of CCA and is more prone to leaching in treated wood exposed to severe weather because it does not bind to the wood structure during treatment (Moreschi, 2013; Baharuddin et al., 2022).

The low absorption and, consequently, high leaching of the preservative treatment is highly detrimental to the durability of the treated wood. The rapid loss of the solution's components compromises the treatment's efficiency. Despite being less harmful than CCA, CCB is still toxic to the environment, so the leaching of this component represents an environmental liability due to the risk of soil and groundwater contamination (Souza; Demenighi, 2017).

Table 2 - Absorption of the preservative and cold water solubility of treated and untreated *Eucalyptus* sp.

Parameters of <i>Eucalyptus</i> sp. Wood	Treatment	Time (months)		Pr>Fc Interaction
		0	4	
Preservative absorption (%)	No treatment	-	-	
	Liquor	41,79 A (5,62)	-	
	Liquor and tannin	27,97 B (3,49)	-	*
	CCB	21,31 C (5,71)	-	
Cold water solubility (%)	No treatment	5,61 aB (8,38)	5,06 aA (2,05)	
	Liquor	5,63 aB (4,12)	5,19 aA (2,97)	*
	Liquor and tannin	4,65 aC (1,61)	3,97 bB (2,55)	
	CCB	7,90 aA (8,25)	3,41 bB (1,57)	

Source: Authors (2024)

In where: Averages followed by the same lowercase letter in the column do not differ statistically from each other using the Tukey test (5%). * significant at 5% probability level ($01 \leq p < 0.05$). The values in brackets correspond respectively to the coefficient of variation (%).

4 CONCLUSIONS

It was concluded that:

- The treatment of *Eucalyptus* sp. wood with pyroligneous liquor + tannin, compared to untreated wood and wood treated only with pyroligneous liquor, was effective mainly for the properties of bulk density and flexural strength;

- The wood treated with pyroligneous liquor + tannin was superior to all the other treatments in terms of static flexural strength and solubility in cold water;

- Wood treated with CCB showed greater leaching of the components than the other treatments;

- Pyroligneous liquor, combined with tannin, is a potential preservative product that can sustainably replace the usual preservatives such as CCB, which is considered more toxic and polluting to the environment than that of natural origin;

- Higher concentrations of pyroligneous liquor and tannin are recommended to increase their preservative potential, resulting in better performance in the physical and mechanical characteristics of *Eucalyptus* wood.

It is important to emphasise that the results obtained in the present study refer to a four-month exposure period. Therefore, this information should be carefully considered when making comparisons with other studies.

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