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GLULAM BONDING QUALITY OF CHROMATED COPPER ARSENATE TREATMENT APPLIED TO *Pinus elliottii* WOOD AND THREE STRUCTURAL ADHESIVES

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ABSTRACT

Glued laminated timber is an alternative gaining prominence in the Brazilian construction field. As a result, industries seek ways to improve the quality of their products, focusing on the wood used, the adhesive, and the manufacturing process. The aims of this study are to evaluate the effect of the preservative treatment chromated copper arsenate (CCA) as a preservative treatment on the gluing quality of *Pinus elliottii* (slash pine) glued laminated timber (Glulam) elements. For this purpose, Glulam was compared with and without CCA treatment, using Cascophen RS 216 -M, Jowat 686,60 and AG 101 adhesives to bond the laminated wood specimens. The glue quality was evaluated through delamination and shear strength tests of the glue lines based on the test method, following the European standard. The delamination results indicated that there were no significant differences between those obtained for the combinations of wood treated with CCA and wood in natura. The results of the shear tests on the glue lines revealed significant differences when comparing the performance of wood treated with CCA to that of untreated (in natura) wood. Specifically, when using Cascophen 216-M and polyurethane AG 101 adhesives, the treated wood demonstrated notably different shear strengths. This indicates that the preservative treatment with chromated copper arsenate alters the bonding effectiveness of these adhesives, highlighting the importance of selecting appropriate adhesive formulations for treated wood to ensure optimal bonding quality. The only species/adhesive combination that showed a delamination limit lower than 4 % was the one that considered natural wood glued with Cascophen 216-M adhesive. To confirm these results is essential a complementary study evaluating the viscosity of the resins used.

Keywords: Adhesive bonding, Chromated Copper Arsenate, delamination, Glued Laminated Timber, Glue line shear, *Pinus elliottii*, preservative treatment.

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INTRODUCTION

Glued laminated timber (Glulam) has shown to be a promising alternative in the segment of industrialized constructions with wooden structures, by using reforested woods (pine and eucalyptus) in its composition (Brito *et al.* 2018, Stringari *et al.* 2020). Glulam is an industrialized product with better mechanical properties and shape adaptability when compared to conventional solid wood pieces with large dimensions (Petrauski *et al.* 2016, Zhan *et al.* 2020).

Glulam industries seek alternatives to improve the quality of their products through the choice of wood species used in the composition, type of adhesive, and quality of the material manufacturing process (Thorhallsson *et al.* 2017). The choice of the correct "species/adhesive" combination in the manufacture of the Glulam element is essential to ensure the structural quality of the element in service (Alade *et al.* 2021). Different structural adhesives are used to manufacture these glued elements, the most common being polyurethanes and resorcinol-based adhesives (Bockel *et al.* 2019, Musah *et al.* 2021, Sun *et al.* 2020, Treu *et al.* 2020).

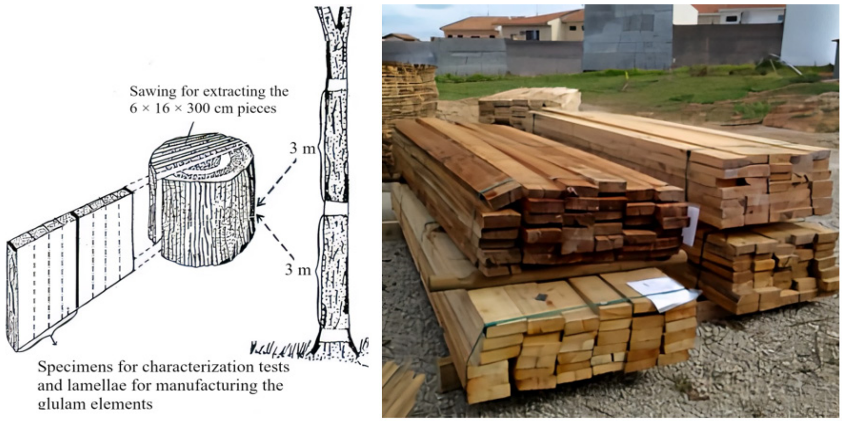
The use of wood and its derived products for structural use requires preservative treatment, which can be done with solutions of chromated copper arsenate (CCA), Chromated copper borate (CCB), and Creosote (Andrade *et al.* 2020, Freitas *et al.* 2010, Lima *et al.* 2020, Molina *et al*. 2011, Oliveira *et al.* 2020, Poleto *et al.* 2020). The preservative treatment is carried out by applying products that prevent the attack of deteriorating agents, those of biological origin (Dias *et al.* 2020). The preservative treatment has the purpose of obtaining longer service life for the material in service (Samani *et al.* 2019).

However, with the current use of different types of reforested woods associated with different structural adhesives to manufacture Glulam elements and considering the need to ensure the increase of the service life of wood structures in general, this study is justified. Therefore, this research aims to evaluate the influence of the use of CCA in the bonding quality of Glulam elements made with pine wood compared to the response of Glulam elements made with in natura wood (without CCA treatment).

MATERIALS AND METHODS

In the present study, the Glulam elements were made with slash pine (*Pinus elliottii* Engelm.) wood belonging to resistance class C30, treated with CCA, and with in natura wood (not treated with CCA). The CCA treatment in autoclave used the full-cell process, i.e., an initial vacuum was applied to drain the volume of air existing in the dry wood before the treatment, which was later replaced by a preservative solution. The CCA retention considered was 6,5 kg/m³, as ABNT NBR 9480-09 (2009) required.

For the assembly of the Glulam elements and the characterization specimens, the wood lamellae were obtained from different pieces of wood with dimensions 6 cm × 16 cm × 300 cm (Figure 1). From each piece, wooden lamellae were extracted with dimensions 2 cm × 5 cm × 120 cm. To comprehensively assess the mechanical properties and behavior of the materials, eighteen specimens were then prepared from each lamella: six for the compression test parallel to the fibers, six for the shear test parallel to the fibers, and six for determining moisture content and density. This structured approach to specimen preparation and testing was rigorously aligned with the specifications and standards outlined in ABNT NBR 7190-3 (2022). By strictly following these guidelines, our methodology ensures the consistency, accuracy, and reliability of our tests, allowing for the precise evaluation of the Glulam elements under investigation.



(a) Scheme to extract the planks from the trunk (b) Planks ready to extract the lamellae and specimens for characterization.

Figure 1: Slash pine (*Pinus elliottii* Engelm.) wood for manufacturing the Glulam elements.

From a total of 180 wooden lamellae produced, 50 % (90 lamellae) were submitted to the preservative treatment with CCA, while the remaining 50 % had no treatment with CCA. The wooden lamellae were submitted to the mechanical grading process, based on Annex B of ABNT NBR 7190 (1997) and visual grading according to ASTM D245-19 (2019). All the wooden lamellae and characterization specimens were dried until they reached a moisture content of 12 %. After reaching the moisture content of 12 %, the lamellae were placed separately in transparent polyethylene bags, where they remained for 30 days for the homogenization of the mass of water throughout its volume. At the time of gluing, the wooden lamellae had a moisture content of 12 %.

Three types of structural adhesives were used for gluing the lamellae, considering a total of six different species/adhesive combinations (Figure 2): two polyurethane adhesives (Jowat 686.60 - monocomponente defined as "PUmono" in this work and AG 101 - bicomponent defined as "PUbi") and a resorcinol-based adhesive (Cascophen RS-216-M - bicomponent, defined as "Res").

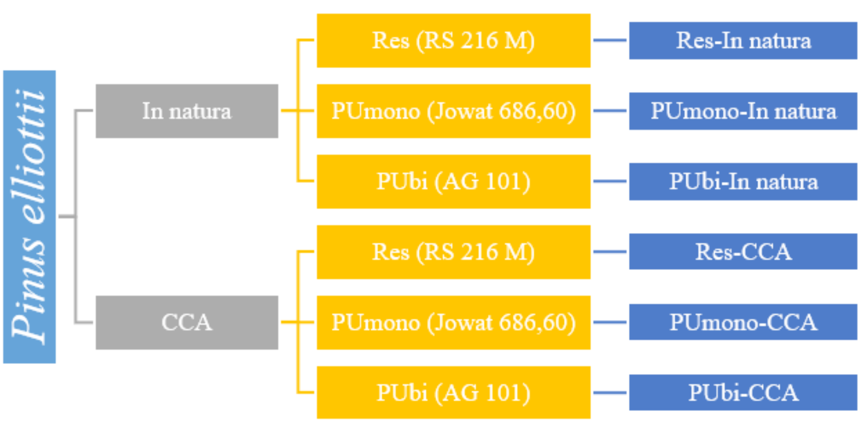


Figure 2: Species/adhesive combinations considered for slash pine (*Pinus elliottii* Engelm.) lamellae.

The basis weight used for the Res adhesive was 240 g/m². For both polyurethane adhesives (PUmono and PUbi), a basis weight of 200 g/m² was used. Adhesives were applied only in a single face of lamella (double glue line).

When available, the viscosities allowed for the adhesives were those indicated by the manufacturers' catalogs. According to Motta *et al.* (2014), the viscosity value of the Res adhesive, according to information from the manufacturer, ranges from 500-1000 Pa s. For the PUmono adhesive the viscosity ranges between 10200 ± 2500 m Pa s. However, neither the literature nor the manufacturer's catalog provided the viscosity value of the PUbi resin.

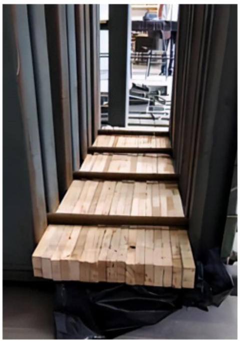
The pressing of the beams lasted 72 hours to ensure the complete curing of the adhesives. The bonding pressure of the beams was applied through a floor press, with pressure control using a torque meter.

The gluing pressure applied to the Glulam beams was 1,2 MPa (according to standard project ABNT NBR 7190-3 (2022), for woods with a density equal to or higher than 500 kg/m³). The average apparent density obtained in the wood characterization, referring to a standard reference moisture content of 12 %, according to ABNT NBR 7190 (1997), was 547 kg/m³ (Table 1).

The torque applied to each screw of the floor press for gluing pressure control was equal to 35,41 N m. Figure 3 shows the details of assembling of Glulam beams by using the floor press with bonding pressure gauged by torque meter.

(a) Application of the gluing pressure with torque meter

(b) Lamellae after drying in polystyrene bags



(c) Glulam beams at rest in the floor press

Figure 3: Fabrication of the Glulam beams.

Thirty-six Glulam beams with dimensions 10 cm× 5 cm × 120 cm were produced, 18 beams treated with CCA, and the other 18 in natura. Sixteen specimens were taken from each beam (seven for the delamination tests and another seven for the bond line shear strength tests), excluding the two specimens from the extremities (Figure 4). For each species/adhesive combination (Figure 2), 42 specimens were considered (21 for the delamination tests and 21 for the shear bond strength tests). The lamellae with better visual quality and higher modulus of elasticity in bending were positioned at the extremities of the beams, while in the central part of the beam, on the neutral line, were positioned the lamellae with lower mechanical and visual quality.

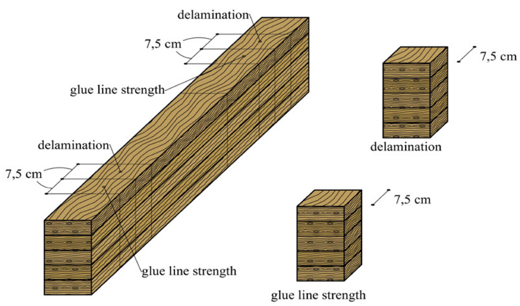


Figure 4: Removal specimens for delamination and shear strength tests on the glue line.

The delamination and shear strength tests of the glue lines were carried out based on the recommendations of the test method ABNT NBR 7190-3 (2022) for the external environment, based on the European standard

EN 14080-13 (2013).

Delamination tests

The delamination specimens initially had their masses determined. Firstly, the samples were immersed in water inside an autoclave at a temperature of 20 ºC (Figure 5a). Afterward, a vacuum of 80 kPa was applied for 30 minutes. Subsequently, the specimens were submitted for 2 hours to a pressure of 550 kPa. A metal grid was positioned over the specimens inside the autoclave, ensuring submersion in water during the delamination tests. After a single vacuum-pressure cycle (Figure 5b), the samples were placed in a drying chamber at a temperature of 70°C (Figure 5c). Inside the drying chamber, the airflow was at a velocity of 2 m s^{-1} and relative humidity was approximately 8 %. The specimens were 50 mm apart during the drying, with the cut cross-sections positioned parallel to the airflow. The specimens continued in the chamber until specimen's mass reached a value between 0 % and 10 % above the initial mass obtained before the autoclave process. The drying process of the specimens in the chamber lasted between 19 and 22 hours.

(a) Specimens before the vacuum and pressure process, (b) Specimens after the vacuum and pressure process (c) Drying of the specimens in the chamber.

Figure 5: Details of the drying process.

The percentage of total delamination of each specimen was obtained by Equation 1 based on Figure 6.

$$D_t = \frac{L_o}{L_t} 100 \quad (1)$$

From Equation 1, D_t is total delamination, L_o is the sum of the length of the opening of all the glue lines of the cross-sectional face, and L_t is total length of all the glue lines of the cross-sectional face.

For all delamination tests, the glue line opening (delamination) was evaluated by measuring the glue lines of the front and back faces of the sample taken from the Glulam element (without considering the delamination of the lateral glue lines). The delamination limit considered for coniferous woods, according to the ABNT NBR 7190-3 (2022) test method, was 4 %.

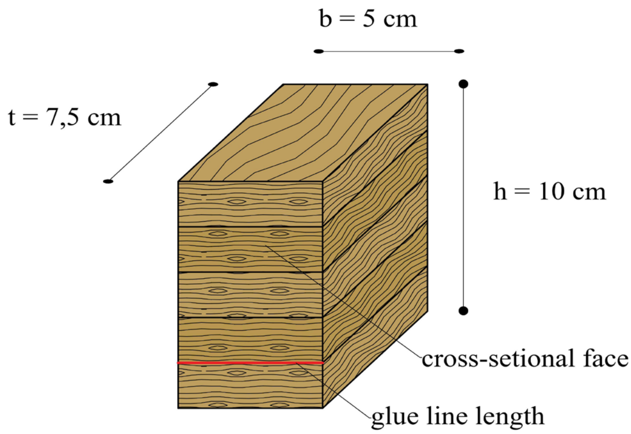
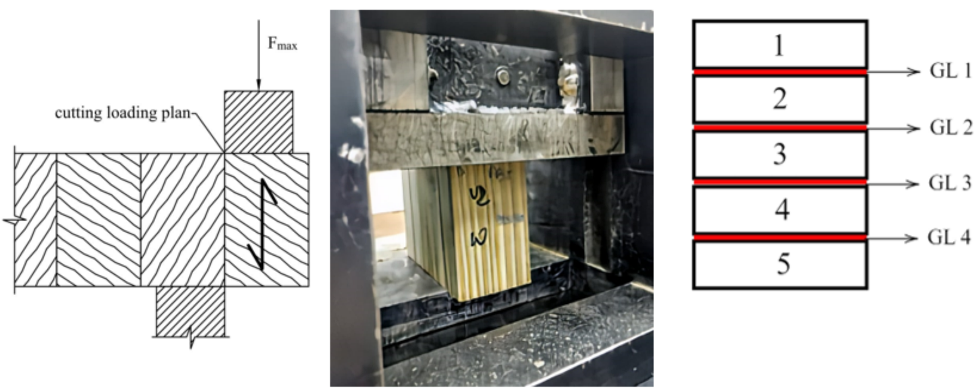


Figure 6: Cross-section considered in the delamination evaluation.

The shear strength of the glue lines and the percentage of wood failure in the sheared area were registered. The shear strength for the glue lines was obtained using Equation 2. The four glue lines (named GL1 to GL4, Figure 7c) for each of the specimens evaluated were ruptured by shear. A metallic device was attached to the EMIC testing machine for the shear tests (Figure 7b). The percentage wood failure after glue lines' shear tests, were quantified using ImageJ software in the bonding regions.

$$f_v = \frac{F_{v,max}}{bt} \quad (2)$$

From Equation 2 f_v is the shear strength of the glue line, $F_{v,max}$ is maximum shear load applied to the rupture lamella, and b and t are the width and height of the specimen (Figure 7).



(a) Schematic for the glue line shear test (b) Shear specimen in the metal testing device (c) Identification of the glue lines for the tests

Figure 7: Details of the glue line shear test.

The results of delamination and shear strength of the glue lines were submitted to statistical analysis. Initially, the normality of the data was verified using the Anderson-Darling test. Subsequently, an ANOVA variance analysis was performed using Tukey's test and p-values were analyzed. Thus, it was verified if there were significant differences between the means obtained for a significant level $p < 0,05$.

RESULTS AND DISCUSSION

In the following items, the main results of the wood characterization, delamination, and shear bond strength tests are presented for the species/adhesive combinations, considering both CCA treated and untreated (in natura) woods. The main mechanical and physical properties of pine wood obtained from the experimental characterization in the laboratory are presented in Table 1.

Table 1: Average results of the wood properties characterization (MC=12 %).

Wood	$f_{c0,m}$ (MPa)	$f_{v0,m}$ (MPa)	E_{c0} (MPa)	$E_{M,m}$ (MPa)	$\tilde{n}$ (kg/m^3)
Pinus elliottii	43,01	9,36	11970	10759	547

$f_{c0,m}$ = mean value of the fiber parallels compressive strength; $f_{v0,m}$ = mean value of the fiber parallel shear strength; $E_{c0,m}$ = mean value of the fiber parallels compressive modulus of elasticity; $E_{M,m}$ = mean value of the laminated modulus of elasticity; $\tilde{n}$ = bulk density at 12 %.

In this study, we have demonstrated that the application of CCA effectively preserves the inherent mechanical properties of slash pine (*Pinus elliottii* Engelm.). This finding aligns with the research conducted by Almeida *et al.* (2019), emphasizing the relevance of our results to the construction industry, particularly in the production of Glulam. A critical measure of this preservation is the characteristic compressive strength parallel to the fibers ($f_{c0,k}$), which laboratory testing determined to be 30,11 MPa. This measurement classifies the CCA-treated slash pine (*Pinus elliottii* Engelm.) within the strength class C30, according to the standards set by ABNT NBR 7190 (1997).

The significance of this classification extends beyond the mere preservation of wood's structural integrity; it signifies the CCA treatment's capacity to maintain the wood's suitability for demanding structural engineering applications without compromise. This insight is crucial for advancing the utilization of treated wood in construction, as it assures engineers and architects of the material's reliability and performance within specified structural classes. Additionally, our findings contribute to the broader understanding of how preservative treatments can be employed to enhance the durability and longevity of wood products while sustaining their mechanical performance.

Delamination

Table 2 compares the average delamination results obtained among the species/adhesive combinations considered. All combinations showed a tendency towards statistical normality.

Table 2: Mean delamination results and comparisons by Tukey's test.

Combination	L_t (mm)	L_o (mm)	D_t^* (%)
Res-In natura	236,30	5,22	2,26c
PUmono-In natura	398,35	112,29	28,17b
PUbi-In natura	399,43	240,18	60,06a
Res-CCA	402,72	33,83	8,41c
PUmono-CCA	389,17	148,43	38,05b
PUbi-CCA	395,47	239,49	60,66a

*Different letters indicate significant difference (p < 0,05).

The lowest percent delamination was obtained for the species/adhesive combinations that did not use CCA treatment. The only combination that met the maximum delamination limit of 4 % was the combination Res-In Natura. The percentages of delamination between the CCA-treated and untreated wood did not show significant differences. However, the average results of the percentages of delamination of treated wood were higher when compared with the results of untreated wood. These results agreed with the results obtained by Azambuja and Dias (2006). Table 3 presents the average results and comparisons of the shear strengths of the glue lines obtained for the species/adhesive combinations analyzed with and without CCA treatment.

Table 3: Comparison of the mean shear strengths of the glue lines using ANOVA and Tukey's test.

Combination	GL1	GL 2	GL 3	GL 4
Res-In natura	5,94a	4,95b	4,29b	4,99b
PUmono-In natura	7,75a	5,04b	4,87b	6,98a
PUbi-In natura	5,55a	3,36b	3,28b	7,00a
Res-CCA	8,40a	5,47b	6,99b	8,16a
PUmono-CCA	7,29a	5,74a	6,49a	7,81a
PUbi-CCA	9,11a	6,74c	6,76ab	7,03bc

*Different letters indicate significant difference ($p < 0,05$).

The variability observed between the glue line strengths is attributed to the fact that each set of lamellae has different visual and mechanical ratings and different apparent densities. Denser woods tend to present greater penetration difficulty for the adhesive. The combinations referring to the central glue lines (GL2 and GL3) showed lower mean shear strengths than the end lines (GL1 and GL4), and these differences were statistically considerable. For combination 5, the strengths of the center glue line (GL2 and GL3) did not differ statistically from the strengths of the end lines (GL1 and GL4). Figure 8 shows the overall comparison between the species/adhesive combinations with and without CCA treatment.

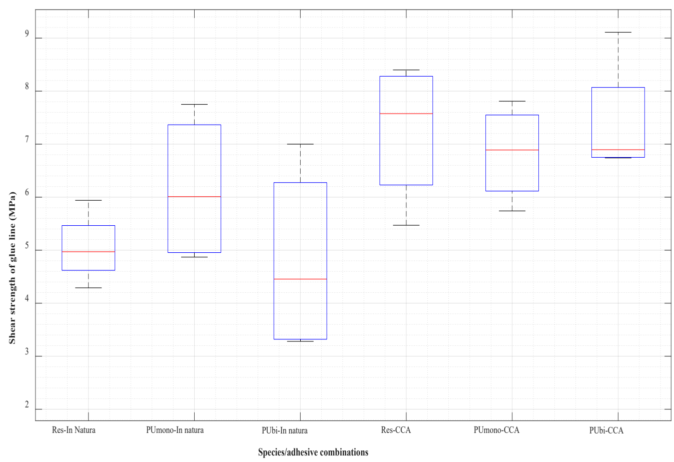


Figure 8: Boxplot for species/adhesive combinations considering the overall average of shear strengths obtained for all glue lines of the beams.

The comparison between pairs of combinations (Res-In natura/CCA; PUmono-In natura/CCA and PUbi-In natura/CCA), in Figure 8, indicated the existence of relevant differences between pairs Res-In natura/CCA and PUbi-In natura/CCA. However, there were no relevant differences between combinations PUmono-In natura and PUmono-CCA.

Table 4 shows the average percentage wood failure (WF) obtained for the specimens in the glued regions for each species/adhesive combination analyzed. Higher percentages of failure in wood indicate better bonding quality. In bonding wood pieces with structural adhesives, the highest percentage of failure is expected occurs in wood and not in the adhesive. There was a tendency to increase the percentage wood failure for the species/adhesive combinations that used treated wood and polyurethane adhesives when compared to species/adhesive combinations that used woods without CCA treatment.

Table 4: Results of the average percentage wood failure.

Combination	WF* (%)
Res-In natura	80,05a
PUmono-In natura	83,75a
PUbi-In natura	78,26a
Res-CCA	76,11a
PUmono-CCA	84,90a
PUbi-CCA	79,12a

*Different letters indicate significant difference ($p < 0{,}05$).

Based on Table 4, the best failure responses occurred for Combinations PUmono-In natura and PUmono-CCA. However, none of the combinations showed relevant differences between wood treated and untreated.

It is worth mentioning that the viscosity of the adhesive is also an important factor to be considered when studying the shear strength of the glue line because the higher the viscosity, the higher the resistance to movement and, consequently, the lower the ability of the adhesive to penetrate the wood. High viscosity values are undesirable since they impair the bonding quality due to the difficulty in uniformly distributing the adhesive over the wood (difficulty in spreading). On the other hand, low viscosity adhesives may result in an excessively absorbed glue line on the wood. Therefore, one should pay attention to these extreme viscosity values. In research developed by Motta *et al.* (2014), for the application of Res adhesive, 20 % FM-60-M catalyst and 5 % ethyl alcohol were added to improve adhesive penetration into the wood. In the present study, the viscosities of the adhesives were not experimentally quantified, and the general values recommended by the manufacturers were used.

CONCLUSIONS

The delamination tests for all the species/adhesive combinations analyzed in this study showed no significant differences between the combinations of wood treated with CCA and those without preservative treatment (in natura).

Only the combination of untreated with CCA and glued with the Res adhesive, showed a delamination rate below 4 %, indicating good glue quality.

The shear strength of the glue lines' showed no significant difference between PUmono-In natura and PUmono-CCA, referring to pine wood glued with the Jowat 686,60 adhesive.

The combinations of Res-In natura and Res-CCA (bonded with Cascophen RS216-M adhesive), as well as PUbi-in natura and PUbi-CCA (bonded with AG 101 adhesive), showed significant differences in shear strength of the glue line between in natura wood and CCA treated wood.

For all combinations analyzed, there were no significant differences in the percentage of wood failure between in natura wood and CCA-treated wood. However, there was a tendency to increase this percentage when using CCA treatment in wood associated with polyurethane adhesives.

For future studies, it is necessary to evaluate the viscosity of the adhesives used when gluing the lamellae to clarify the results presented here. It is essential, to consider the use of solvents in the adhesives examined in this study, to improve penetration into the wood.

Authorship contributions

N. A. B.: Conceptualization, methodology, investigation, formal analysis, writing - original draft. M. H. M. M.: Conceptualization, methodology, investigation, formal analysis, writing - original draft. K. A. O.: Formal analysis, writing - review & editing. C. A. B. O.: Formal analysis, writing - review & editing. J. C. M.: Conceptualization, supervision, funding acquisition, project administration. A. L. C.: Conceptualization, supervision, funding acquisition.

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