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ANALYSIS OF GLUE LINE AND CORRELATIONS BETWEEN DENSITY AND ANATOMICAL CHARACTERISTICS OF *Eucalyptus grandis* × *Eucalyptus urophylla* GLULAM

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ABSTRACT

This study aimed to analyze the glue line thickness effect and its relation with the density and anatomical characteristics of eucalyptus wood. Thus, the thickness of main and secondary glue lines was measured as well as their interaction with the apparent density of elements glued with resorcinol-formaldehyde (RF) and castor oil polyurethane (CP) adhesives. Anatomical wood characterization of *Eucalyptus grandis* × *Eucalyptus urophylla* was performed by correlating glue line thickness. According to normative instruction, specimens were produced for delamination tests. The experiment was conducted in a completely random 2 × 2 design factorial scheme (two classes of wood apparent density and two adhesives). Pearson correlation ($t < 0,01$) was performed among variables. It was found that there was adhesive penetration into vessels and rays of wood. Glue line thickness was higher in woods with density higher than to 580 kg/m^3 glued with RF adhesive. There was a low correlation between wood density and vessel diameter to main and secondary glue lines ($t < 0,01$).

Keywords: Apparent density, castor polyurethane, delamination, laminated wood, resorcinol-formaldehyde.

INTRODUCTION

Glued laminated timber (Glulam) is a structural product obtained by gluing pieces of timber with fibers parallel to each other (Bodig and Jayne 1993). The market still needs to know more information about the mechanical resistance of this product; thus, to understand how Glulam will work it is important to evaluate the behavior of some variables, such as apparent density and timber glued with adhesive.

Wood density provides information to support methods that should be adopted during the gluing process. By its determination, it is possible to correlate adhesion resistance with anatomic elements. Proportion of empty spaces combined with dimensions and arrangement of cellular elements have influence on adhesive mobility and penetration into timber structure as in the resistance of glue line to delamination (Albuquerque and Latorraca 2000, Konnerth *et al.* 2008, Lopes 2008, Singh *et al.* 2008, Stoeckel *et al.* 2013, Gonçalves *et al.* 2019). When low density (higher frequency and diameters of vessel, with high and wide rays), it may allow excessive penetration of adhesive, if the adhesive does not present an ideal viscosity and formation of good glue line (Albuquerque *et al.* 2005, Albino *et al.* 2010).

On the other hand, when species are anatomically unfavorable for gluing, there will be low adhesive penetration and formation of a thick glue line (Albuquerque *et al.* 2005, Tienne *et al*. 2011). Both types of glue lines are undesirable because they reduce the mechanical strength of glued joints causing separation of adjacent layers. Changes on adhesive viscosity, by adding loads or filling, can correct this problem (Lopes 2008).

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Physical-chemical adhesion phenomenon predicts an interaction mechanism between solid surfaces glued by adhesive and the capacity of holding other materials together (Marra 1992). Adhesives are used to join elements by flowing and filling empty spaces between them. Thus, they can reduce distances and create interactions among glued elements (Bianche *et al.* 2017).

It is important to understand the interaction between wood and adhesive because it helps to evaluate gluing quality (Albino *et al.* 2010), especially when it comes to the wood of *Eucalyptus* genus since its anatomical structure presents occurrence of small diameter, presence of tyloses, low frequency, and small width vessels. Other characteristics are also important, such as the frequency of conducting vessels (one of the anatomical parameter that most influences gluing process), followed by frequency, width of rays and condition of the vessels, that may or may not be obstructed by tyloses and gomoresins (Lima *et al*. 2007). This may hinder or prevent the adhesive from penetrating the wood.

Thus, this study aimed to measure the thickness of main and secondary glue lines and to correlate this variable with apparent density, vessel and rays of *Eucalyptus grandis* × *Eucalyptus urophylla* wood.

MATERIAL AND METHODS

Origin and preparation of the material

The wooden planks used came from 11-years-old *Eucalyptus grandis* × *Eucalyptus urophylla* clones from Bahia Wood Products Company, located in the municipality of Nova Viçosa, Bahia State, Brazil. Figure 1 shows the preparation process of the glued laminated timber and the other tests.

A total of 40 wooden planks provided by the company were placed in a covered environment until reaching the local equilibrium humidity. After that, the planks were visually classified and separated by desired dimensions to produce Glulam blocks.

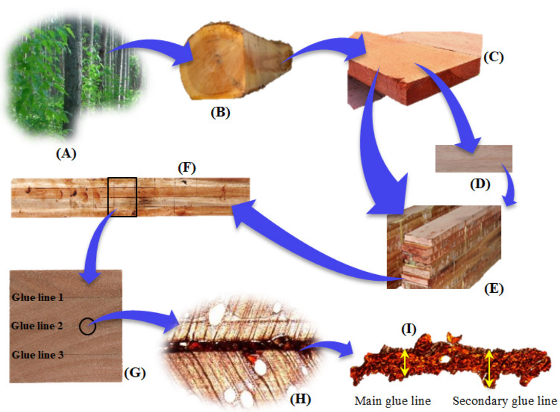

Figure 1: Method of obtaining wood boards and specimens. Commercial eucalypt plantation (A), Wood primary processing (B), Wooden sawn (C), Test specimen for determination gravity in wood (D), Glulam blocks (E), Glulam ready and clean (F), Specimen for delamination test and measuring the glue line (G), Lamina illustrating the glue lines (H), Main and secondary glue line measurement (I).

The characterization of the wood physical properties (moisture content and apparent density or specific gravity) was performed according to Brazilian Standard - NBR 7190, Brazilian Association of Technical Standards - ABNT NBR 7190 (1997), when using the same specimens. To perform the tests, four specimens were removed from each board, totaling 160 specimens, which were sectioned in dimensions of 3 cm × 2 cm × 5 cm (radial × tangential × longitudinal).

Adhesive and glued elements

There were tested two commercial adhesives, a thermoset Cascophen RS-216-M based on resorcinol-formaldehyde (RF), and a thermoplastic bi-component polyurethane based on castor oil (Imperveg® AGT 1315). Instructions of how to use the adhesives, as well the extender and catalyst choice were made based on recommendations from the manufacturer. For Cascophen RS-216-M application was added 20 % of FM-60-M catalyst. The Imperveg® AGT 1315 was applied in the proportion 1:1,2 of one part of component A (prepolymer) and 1,2 part of component B (polyol).

To produce Glulam, the boards were segregated according to their apparent density into two groups: G1) apparent density less than 580 kg/m^3; and G2) equal or higher than 580 kg/m^3. After the segregation groups, boards were sectioned and transformed into 56 lamellae with dimensions 21 cm × 21 cm × 2,5 cm (length × width × thickness).

Each group had 28 lamellae, resulting in 14 Glulam blocks. Seven Glulam blocks were used for each adhesive. A spatula was used to apply 300 g/m^2 to each pair of boards in a single glue line. They were joined and pressed in hydraulic press (capacity 15 tonnes) for 48 hours at a pressure of 1,0 MPa and a temperature of 20 °C (ABNT NBR 7190 1997).

Wood anatomical characterization

The vessels (diameter and frequency) and rays (height, width, and frequency) measurements were made from wood samples with 1,0 cm × 1,5 cm × 2,0 cm (radial × tangential ×longitudinal), made by a manual sliding microtome (Leica SP9000).

Wood laminas were photomicrographied with image analyzer software (AxioVision Rel. 4.5 2014). In addition, it was performed microscopic identification with 10x magnification lens (Axio Ziezz, Scope.A1) (IAWA 1989).

Visualization of the wood-adhesive interface

The study of the wood-adhesive interface was carried out based in Albino *et al.* (2010). The timber laminas came from cubical joints of a 0,5 cm × 0,5 cm × 0,5 cm, in a manual sliding microtome. Each cube had samples collected in the glue line.

A total of 28 cubes were prepared, 14 cubes for each group of apparent density (seven cubes for adhesive) resulting in 56 timber laminas. For each cube 30 measurements were taken, which means, 15 for the main glue line and 15 for the secondary ones.

Thickness measurements were done in the main and secondary glue lines, through glue line width with a 5x objective and image analyzer software.

Delamination test on blocks of glued laminated timber

Delamination test was executed arranging the specimens inside an autoclave. After that, glue lines were exposed to stresses that came from vacuum and pressure effects. That is possible due to 6 days period of moistening and drying cycles, as steps 1 to 4 adapted from the American Institute of Timber Construction - AITC (2007) (Figure 2). Each cycle lasted 48 hours, out of a total three ones.

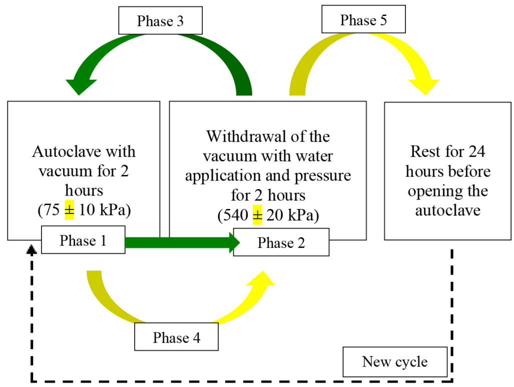

Figure 2: Scheme of moistening cycle for delamination test on Glulam specimens. Adapted from AITC (2007).

At the end of the third cycle, it was possible to tell, how Glulam would behave outside. This was done by measuring the delamination percentage gained in the two specimens top faces. It was possible to observe the percentage of delamination, by the relation between maximum opening length and the total length of the glue line.

Samples were placed outside in natural environmental conditions to dry out for 36 hours at 28 °C ± 2 °C of temperature, aiming to reduce their weight, no more than 5 % to 6 % of the initial weight range. For each specimen, it recommended that the total glue line delamination should not exceed 10% of total length at the specimen top, to Glulam approval for exterior use (AITC 2007).

Statistical data analysis

The experiment was made in a completely randomized design following 2 × 2 factorial scheme, where apparent density and adhesive had two levels. Results were submitted to variance analysis - ANOVA ($F < 0,05$). Priori the ANOVA, the normality requirements by Kolmogorov-Smirnov test ($p < 0,05$) and homogeneity of variances by Cochran test ($p < 0,05$) ware performed. For anatomical characteristics, it was also performed a descriptive statistical analysis.

Pearson correlation coefficient ($t < 0,01$) was used to find out the relationship between variables (apparent density, adhesive type, main glue line, secondary ones, vessel diameter, height, and width of rays, and number of cells). Correlations were realized when considering the ratings 0,1 - 0,3 weak, 0,4 - 0,6 moderate, 0,7 - 0,9 strong correlation, as Dancey and Reidy (2006).

RESULTS

Physical and anatomical wood characteristics

The *Eucalyptus grandis* × *Eucalyptus urophylla* showed an apparent average density (ρ) of 590 kg/m^3 and an average moisture content of 9,34 %. The Figure 1 shows the anatomical characteristics of the studied wood.

Table 1: Average and descriptive numbers of measured parameters in the wood rays of *Eucalyptus grandis* × *Eucalyptus urophylla* wood.

Parameters evaluated	**Density (kg/m³)**	
	< 580 kg/m³ (Group 1)	≥ 580 kg/m³ (Group 2)
Height of rays (µm)	234,72 (63,91)	254,58 (73,69)
Width of rays (µm)	13,33 (2,93)	16,18 (4,75)
Cells number in height	14 (6)	15 (3)
Frequency (rays mm⁻¹)	13 (2)	16 (2)

Value in parentheses refers to standard deviation.

Wood-adhesive interface and delamination test

By the photomicrographs analyses, it was possible to observe the adhesive penetration in the wood anatomical structure. A more visible glue line was found in the laminated timber glued with RF adhesive, due to its reddish color (Figure 3c and Figure 3d).

After delamination test, Glulam blocks of *Eucalyptus grandis* × *Eucalyptus urophylla* did not present changes that compromised the integrity of the wood-adhesive interface. Moisture, pressure and temperature variation were satisfactory to guarantee structural element integrity, as cited by Fiorelli and Dias (2005). After visual analysis of glue lines conditions, specimens did not present delamination slits; which means 0 % delamination.

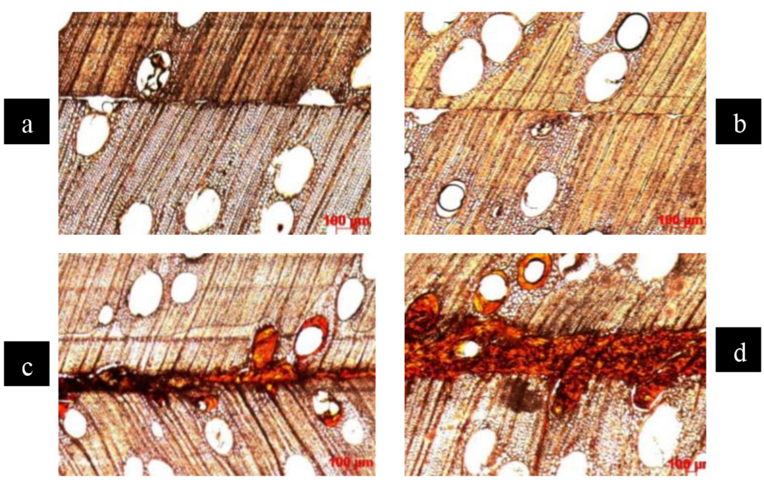

Figure 3: Glue line in the transverse surface of elements glued with castor polyurethane (CP) and resorcinol-formaldehyde (RF). (a) CP in the wood lamina of Group 1 (< 580 kg/m³); (b) CP in Group 2 (≥ 580 kg/m³); (c) RF in of Group 1 (< 580 kg/m³); (d) RF in of Group 2 (≥ 580 kg/m³).

Main and secondary glue lines and with apparent wood density

Based on F test, the effects of apparent density, adhesives, and their interaction were significant to thickness of the main glue line. The interaction between apparent density and adhesives was unfolded and analyzed (Table 2).

Table 2: Average values for thickness of main and secondary glue lines.

Density (kg/m^3)	Main glue line (µm)		Secondary glue line (µm)	
	Adhesive			
	CP	RF	CP	RF
< 580 (Group 1)	3,17Ab	125,22Ba	51,77Ab	278,18Ba
≥ 580 (Group 2)	1,70Ab	217,77Aa	71,79Ab	432,83Aa

Averages with uppercase (vertical) and lowercase letters (horizontal) for each glue line do not differ (F test, $p < 0,05$); CP: castor polyurethane adhesive; RF: resorcinol-formaldehyde adhesive.

To CP adhesive, the contrast between average apparent density levels was statistically zero. Thus, Glulam elements in Group 1 and 2 with CP adhesive showed no significant difference for both glue line thicknesses.

Pearson's correlation on studied variables

The main glue line thickness showed a strong and significant correlation ($t < 0,01$) with adhesive (0,739) (Table 3). Correlation is a statistical analysis that measures the association between variables. Thus, it was possible to say that the behavior presented by glue line thickness was related to the adhesive.

Table 3: Pearson's correlation coefficients based on the average of studied variables for the *Eucalyptus grandis* × *Eucalyptus urophylla* Glulam.

	D_{ap}	A_{dhe}	LP	LS	DV	H_{ray}	L_{ray}	N_{Cel}
D_{ap}	-	$0,000^{ns}$	0,131**	0,125**	0,197**	$0,105^{ns}$	$0,235^{ns}$	$0,1394^{ns}$
A_{dhe}		-	0,739**	$0,070^{ns}$	0	0	0	0
LP			-	$0,060^{ns}$	$0,100^{ns}$	$-0,100^{ns}$	$-0,050^{ns}$	$-0,080^{ns}$
LS				-	$-0,003^{ns}$	$0,023^{ns}$	0,175**	$-0,019^{ns}$
D_V					-	0,221**	$-0,249^{ns}$	$-0,104^{ns}$
H_{ray}						-	0,361**	0,503**
L_{ray}							-	0,608**
N_{Cel}								-

D_{ap}: Apparent density; A_{dhe}: Adhesive; LP: Main glue line; LS: Secondary glue line; D_V: Diameter of the vessels; H_{ray}: Height of rays; L_{ray}: Width of rays; N_{cel}: Number of cells.
** Significant correlation ($t < 0,01$); ns: non-significant correlation ($t > 0,05$).

DISCUSSION

Physical and anatomical characteristics of wood

These characteristics have an influence on adhesive penetration (Marra 1992), pressure need, pressing time and adhesive cure (how long it needs to be ready for use), making its determination an indispensable factor to produce Glulam elements (Segundinho *et al.* 2015).

The largest vessel diameters were found on Group 2 timber boards samples (138,56 µm), value 7,57 % higher than Group 1. Results found in this study were lower than reported by Queiroz (2002) and Boschetti *et al.* (2015), respectively, 125,5 µm and 116 µm, for the same hybrid trees.

In this study, we used trees with 11 years old. Young to mature wood transition happens gradually between 7 to 14 years of growth for this specie (Adamapoulos 2011). Possibly, wood samples came from a more mature tree, with cells of thick wood walls and reduced size of its cellular elements. This fact could explain the contrast between reduced values for vessels found in this work and those mentioned in literature.

Group 1 wood samples (lighter boards) presented 30,70 % superior vascular frequency when compared to Group 2, respectively 7,82 and 7,76 vessels/mm^2. The hybrid used in the research was provided in a batch, for this reason, it is possible that some timber boards originally came from trees with different ages. Age is an important factor when it comes to the adult wood formation because it allows higher thickening of cell walls, vessels, and parenchyma. Thus, it may have influenced the measured difference in board anatomical elements (Gonçalves *et al.* 2009). The higher vascular frequency for Group 1 blocks of wood was compensated by a smaller vessel diameter size. Woods with smaller vascular diameters have a higher vascular frequency (Oliveira *et al.* 2012).

Group 2 timber boards showed 7,80 % higher rays height than boards from Group 1, respectively 254,58 µm and 234,72 µm. Rays width in wood from Group 2 was 17,61% higher than Group 1 and the number of cells was 8,53 % higher in Group 2. Different anatomical behaviors on *Eucalyptus* genus can be explained by silvicultural treatments, plant growth and tree age (Gonçalves *et al.* 2004, Oliveira *et al.* 2012).

Adhesive-wood interface, delamination and glue line analysis

When conditions like wettability, roughness (even if minimally), and the cleanliness the surface are present, the penetration of the adhesive occurs more deeply into wood structure (Chandler *et al.* 2005). To be able to create a strong bonding between an adhesive and the substrate, it is required enough resin to penetrate into wood components, which means that the adhesive must have satisfactory mobility (Chandler *et al.* 2005). Thereby, glued elements possibly had ideal conditions on the surface to create a strong connection (Albino *et al.* 2010) and the amount of used adhesive was enough.

There was adhesive penetration into rays and vessel elements, which was possible due to perforations on their walls that allowed the inter-cells communication. These characteristics can help adhesive acquiring by the structure and in the anchoring formation between both (Albino *et al.* 2010).

Adhesive penetration into ray cells may have happened through transfer cells since they are made of parenchymatic cells and they work as conducting elements in the radial direction. Parenchyma cells have a modified wall to allow transportation, which is generally short-distance (Albino *et al.* 2010). The specie anatomical condition, high or low density, reflects on adhesive penetration (Albuquerque and Latorraca 2000, Kamke and Lee 2007).

When the adhesive has corrected viscosity and finds in substrate ideal conditions of surface and microscopic structure, the product created from this gluing must have equal or superior characteristics than the sum of individual characteristics from the materials that it was made from.

Adhesive penetration depth into the wood may have helped in the resistance show by the specimens in the delamination test. Another factor that could explain this behavior are the inherent characteristics of used adhesives that have high resistance to humidity, which makes them able to be used outdoor (Lay and Cranley 1994, Pizzi 1994).

In this study, the resulting numbers for main and secondary glue line thickness were different from those found in species like *Eucalyptus cloeziana* according to Segundinho *et al.* (2017). This is associated with a longer time and lower pressure applied in the present work, respectively 48 hours and 1,0 MPa.

RF adhesive presented opposite behavior to polyurethane adhesive in both glue lines formed, showing a contrast between the average apparent density levels, statistically different from zero ($F \leq 0,05$).

Highest thickness averages to main and secondary glue lines were found in Group 2, 42,50 % and 35,73 % higher than Group 1 averages for RF (Table 2). These numbers are higher than those reported in previous studies for double glue lines 150 g/cm^2 - 52,13 µm and 60,23 µm, respectively (Segundinho *et al.* 2017).

Some variables are very important to the timber gluing process, among them, adhesive amounts to be applied in each specie, wood lamina thickness, pressure and pressing time (Chandler *et al*. 2005, Iwakiri *et al.* 2005, Nascimento *et al.* 2013).

There was a significant difference between glue lines glued with different adhesives. The highest averages were found in those elements glued with RF adhesive, in both groups. In Group 1, RF Glulam elements presented 97,47 % averages (main glue line) and 81,39 % (secondary glue line) higher than those glued with polyurethane castor adhesive. To Group 2, the averages were also 99,22 % and 83,41 %, respectively, higher than polyurethane glued elements.

Anatomical characteristics are the reason why Group 2 showed higher values because vessels with larger diameters, high and wide rays provided ideal conditions to adhesive mobility and penetration. However, their distinct viscosity can explain the difference found between adhesives.

The higher the viscosity showed by the adhesive, higher its difficulty of spreading, which is caused by the lower flowability, resulting in less adhesive penetration on the capillary structure of the wood (Bianche *et al.* 2017).

Correlation between physical and anatomical wood characteristics

When adhesive does not show ideal viscosity, there may be difficulties getting into porous structure of the wood, because of the reduction or obstruction of empty spaces. This could lead to a thicker main glue line formation and lower depth penetration of the adhesive, which was not observed in this study.

Main glue line presented inverse and not significant correlation to ray characteristics, which means that these variables are not demanding when it comes to gluing. The necessary penetration of the adhesive to anchor in the macro and microscopic regions of the wood will be facilitated if the species shows characteristics such as higher porosity, large diameters vessels, and high and wide rays, as observed in the thickness data of the glue line (Table 1).

A wood increase age causes a reduction of the porosity, consequently, the wood will have a greater contact area and an increase in the mechanical resistance to the worth which it is submitted.

The height of rays had a moderate correlation with cell number, rays width and number of cells, respectively 0,503 and 0,608, both under significant ($t < 0,01$). Since the rays are formed by parenchyma cells and have an indeterminate length, it can be said that, increasing the height and width of rays will increase their capacity to create new storage cells (Burger and Richter 1991), which can explain the correlation between these variables.

CONCLUSIONS

The anatomical structure wood in this study allowed the adhesive penetration into vessels and rays;

The apparent density difference showed by the two groups gave distinction to the variability of the anatomical elements presented by timber boards;

The adhesives used had satisfactory characteristics, what was proved by the delamination test;

Tested adhesives are qualified for structural uses since variations of humidity, pressure, and temperature did not promote weakness of the glue line;

There was a significant interaction between glue line thickness and apparent density. Woods of higher apparent density glued with resorcinol-formaldehyde adhesive responded better to this interaction;

Rays height and width had a moderate correlation with cell numbers. This characteristic qualifies the wood as favorable to gluing, since larger rays will contribute to the adhesive permeability.

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