



Adhesives from *Anadenanthera peregrina* tannin for plywood production: effect of sodium bisulfite concentration on gluing quality

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Received: 9 October 2025 / Accepted: 26 January 2026 / Published online: 19 February 2026
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Abstract

Applying different salt concentrations during extraction can increase the yield and improve the properties of tannic adhesives. The objective of this study was to develop natural polyphenol adhesives extracted with different concentrations of sodium bisulfite (NaHSO_3) for the production of plywood panels. The polyphenols were extracted from the bark of *Anadenanthera peregrina* with 0, 3, and 5% NaHSO_3 . The adhesives were produced with solids contents of 45% and 6% paraformaldehyde and tested on plywood panels. The adhesives were characterized by FTIR and thermogravimetric analysis. Gravimetric yield, viscosity, gelation time, solids content, and pH of the tannins were measured to evaluate their use as adhesives. The plywoods were evaluated based on the physical, mechanical, and microstructural properties. The tannins extracted from the bark of *A. peregrina*, under different concentrations of NaHSO_3 , reached high Stiasny values; the adhesives produced showed suitable physicochemical properties. However, increasing the concentration of bisulfite resulted in greater water absorption by the panels, restricting their use to indoor environments. Panels bonded with the adhesive extracted with 3% NaHSO_3 stood out for better mechanical properties, especially in static bending, in addition to meeting shear strength requirements. The bisulfite concentration indicates a viable option for increasing tannin yield, making it an environmentally sustainable alternative for the partial or total replacement of commercially formulated phenol–formaldehyde adhesives.

Keywords Bioadhesives · Sulfitation · Natural polyphenols · Wood boards

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Introduction

The wood panel industry primarily uses synthetic adhesives, reacting formaldehyde with compounds such as phenol, urea, and melamine. These adhesives, while offering high performance in terms of strength and durability, are derived from non-renewable sources such as oil and natural gas and are associated with the emission of volatile organic compounds (VOCs), with negative impacts on human health and the environment (Hemmila et al. 2017; Das et al. 2020).

Given the advancement of restrictive legislation regarding the use of formaldehyde, the search for more sustainable and less toxic adhesive alternatives is intensified. Adhesives formulated from natural resources have been gaining prominence for reducing dependence on fossil fuels and mitigating the release of harmful substances. Among these alternatives, tannins stand out as a promising raw material due to their high content of phenolic groups, which confer reactivity potential comparable to synthetic resins (Sarika et al. 2020).

Tannins are secondary metabolites that can be extracted from various parts of the plant, such as leaves, seeds, fruits, and roots. From an industrial perspective, the main source of commercial extraction is tree bark, which provides higher yields and economic viability (Chupin et al. 2015; Azevêdo et al. 2017).

In Brazil, the species *Anadenanthera peregrina* (Fabaceae family), popularly known as angico-vermelho, has great potential for this purpose by presenting fast growth, dense wood, and being widely distributed in the Cerrado biome. The use of bark, generally considered waste, as a source of tannins contributes to the full utilization of the plant, promoting economic and environmental gains (Wu et al. 2020).

Despite the potential of tannins as substitutes for synthetic adhesives, their industrial application still faces limitations, such as high viscosity, structural variability, and low yield in unmodified extracts. Therefore, it is essential to use modification techniques that increase extraction efficiency and improve their adhesive properties. Sulfitation has emerged as one of the most effective methods in this regard, promoting physicochemical changes in tannins through the addition of hot water and inorganic salts, such as sodium bisulfite (NaHSO_3). This modification results in increased yield, reduced viscosity, and greater tannin reactivity, enabling their use in adhesives (Pizzi and Mittal 2017; Martínez et al. 2019).

Although sulfitation is a well-established process, its specific effects on extraction efficiency, chemical structure, and final adhesive performance of tannins from *A. peregrina*, a species known by its high content of phenolic

compounds, have not been systematically investigated. This study aims to fill this gap and to determine whether this species offers a unique advantage over other tannin sources previously studied. In this context, it is hypothesized that there is an optimal NaHSO_3 concentration capable of maximizing tannin yield and adhesive performance while simultaneously minimizing the detrimental effects on the water resistance of the glue line. Based on this premise, the present study aimed to evaluate the quality of plywood panels produced with tannin-based adhesives obtained from the bark of *A. peregrina*, subjected to extraction with different NaHSO_3 concentrations (0, 3, and 5%, relative to the dry mass of the bark), with a view to the partial or total replacement of conventional synthetic adhesives.

Material and methods

Obtainment of raw material

The bark of *Anadenanthera peregrina* was collected from a native forest fragment located in São João Evangelista, State of Minas Gerais, Brazil. The bark was removed using a machete from the trunks of four different trees, ranging in height from 16 to 20 m and with a diameter at breast height (DBH) ranging from 30 to 40 cm. The bark was air-dried, ground in a Willey knife mill, and sieved through 40 and 60-mesh openings. The fraction retained on the 60-mesh sieve was used for the extraction.

Extraction and gravimetric yield of condensed tannin

Tannins were extracted in a concentrated form, based on adaptations of the methodology proposed by Araujo et al. (2020). Distilled water (150 mL) and 10 g of dried bark established a solid/liquid ratio of 1:15 (m/v). Extractions were performed by the addition of sodium bisulfite (NaHSO_3) (0, 3, and 5%). The experiments were conducted in triplicate in a water bath at 70 °C for 3 h. The solid fractions were separated by filtration through a cloth funnel followed by a 200-mesh sieve. The extracts were then filtered through glass crucibles (porosity 2), using a vacuum pump to remove fine particles.

Solids content and total solids yield

Two aliquots of approximately 20 g were collected from each concentrated extract to determine the total solids content. The aliquots were transferred to previously weighed Petri dishes and placed in an oven with forced air circulation (103 ± 2 °C)

until they reached constant mass. The solids content (SC) and the total solids yield (SY) were calculated according to Eq. 1 and 2, respectively.

$$SC = \left(\frac{m_2}{m_1} \right) \times 100 \quad (1)$$

$$SY = \left(\frac{SC * M}{100} \right) \quad (2)$$

where SC = solids content (%); m_2 = dry mass of extract (g); m_1 = wet mass of extract (g); SY = total solids yield (%); M = total mass of concentrated extract (g).

Stiasny reaction and quantification of condensed tannins

The Stiasny index was determined based on Hoong et al. (2010), with modifications. The concentrated extract was weighed (20 g) into flat-bottomed flasks. Ten mL of deionized water, 4 mL of formaldehyde (37% w/w), and 2 mL of concentrated hydrochloric acid (HCl) were added to the same flask. The mixtures were heated under reflux for 30 min. Under these conditions, the tannins react to form insoluble complexes, which were separated by filtration in a glass crucible with (porosity 1). The retained material was oven-dried (103 ± 2 °C) for approximately 24 h. The Stiasny index was then calculated as described in Eq. 3.

$$SI = \left(\frac{A}{B} \right) \times 100 \quad (3)$$

where SI is the Stiasny Index (%); A is the mass of the precipitate, and B is the mass of tannin solids used in the reaction.

The gravimetric tannin yield (%) was obtained from the product of the total solids yield and the respective Stiasny index of each extract. The non-tannin component yield was determined by the difference between the solids yield and the tannin yield.

Extraction of condensed tannins for the formulation of tannic adhesives

After determining the gravimetric tannin yield, new extractions were performed to formulate tannic adhesives, following the same procedure and mass/volume ratio (1:15). After extraction, the material was dried in an oven with air circulation (40 ± 3 °C) until completely dry. It was then macerated in a mortar and pestle until tannin powder was obtained.

Infrared absorption spectrometry analysis

To obtain infrared spectra, the tannins extracted at different NaHSO₃ concentrations were previously ground, sieved

through a 50-mesh, and used to prepare pellets containing potassium bromide (KBr) at a ratio of 1:100 (w/w). Spectroscopic analysis was performed by ATR-FTIR using a Varian 600-IR series FTIR spectrometer coupled to a GladiATR accessory (Pike Technologies). Small amounts of the ground material were applied directly to the crystal, and spectra were obtained in the 4000–500 cm⁻¹ range, with 32 average scans and a resolution of 4 cm⁻¹.

Thermogravimetric analysis (TGA)

Thermogravimetric analyses of tannins were performed using a Shimadzu DTG-60AH equipment. The samples were heated from 25 to 900 °C under a nitrogen atmosphere, with a flow rate of 50 mL/min and a heating rate of 20 °C/min. The temperature corresponding to mass loss was determined from the inflection points observed on the derivative thermogravimetric (DTG) curve.

Preparation and evaluation of the properties of tannin-based adhesives

Tannin-based adhesives were prepared by dispersing tannin powder in water, stirring at 4000 rpm, to a total solids concentration of 45%. After 24 h of hydration, 6% paraformaldehyde powder, calculated based on the dry mass of tannin, was added, acting as a binding agent.

In addition to the tannin–formaldehyde adhesive formulations with different concentrations of sodium bisulfite (NaHSO₃), a commercial phenol–formaldehyde-based adhesive was also used for bonding the plywoods, for comparison purposes. The rheological and physicochemical properties of the adhesives, such as viscosity, gel time, solids content, and pH, were evaluated, with five replicates for each parameter. Viscosity was measured with a Ford Cup viscometer (Universal), according to ASTM D1200-10 (ASTM 2010). Gel time was determined according to ASTM D 1084 (ASTM 1997). Solids content was obtained according to ASTM D 1490-01 (ASTM 2013), and pH was measured with a digital pHmeter (Tecnal Tec-3mp).

Production of plywood panels

Veneers of *Pinus oocarpa* (18 years old) from a plantation located at the Federal University of Lavras (UFLA). The veneers were obtained with a nominal thickness of 2 mm, and subsequently cut to dimensions of 480 × 480 mm. After production, the veneers were conditioned indoors and then dried in an oven with forced air circulation until they reached a moisture content of 4%. The veneers with the best visual and structural quality were selected for panel assembly.

The panels were manufactured with five veneers arranged in cross-layers, with final dimensions of 300 × 300 × 10 mm,

using a 320 g/m² adhesive grammage (double line). For each bioadhesive formulation and for the control (commercial adhesive), three panels were produced. The pressing cycle adopted consisted of a temperature of 150 °C, a specific pressure of 4 MPa, and a pressing time of 10 min, using an automatic hydraulic press (Fig. 1).

After pressing, the panels were stored in a climate-controlled room (22 ± 2 °C and $65 \pm 3\%$ relative humidity) until the mass stabilized. The test specimens were then cut, and the experimental design for plywood production was presented in Table 1.

Characterization of plywood panels

The physical properties evaluated included apparent density, dry weight moisture, and total water absorption, according to the procedures established in NBR 9485 (ABNT 2011b), NBR 9484 (ABNT 2011a), and NBR 9486 (ABNT 2011c), respectively. Regarding the mechanical properties, the modulus of rupture (MOR) and modulus of elasticity (MOE) under static bending (parallel and perpendicular directions) were determined according to standard NBR 9533 (ABNT 2012b). The dry shear test at the glue line was performed according to standard NBR 12466–1 (ABNT 2012a), while the percentage of wood failure was determined by visual evaluation of the fracture area after the shear test, in accordance with the procedure described in EN 314–1 (CEN 1993). The wood-adhesive interface was analyzed by scanning electron

Table 1 Treatments and composition of plywoods

Treatment	Bissulfite (%)	N° of plywoods	N° of veneers
T0	0	3	5
T3	3	3	5
T5	5	3	5
PF	-	3	5

PF Phenol–formaldehyde.

microscopy (SEM) using a FIB-SEM TESCAN VEGA3 microscope operating at an accelerating voltage of 10 kV. For this purpose, specimens with dimensions of 1.0×1.0×0.5 cm (length×width×thickness) were collected from the region after dry shear testing. The samples were fixed to aluminum stubs with double-sided carbon tape and metallized with a thin layer of gold for observation.

Data analysis

The averages obtained for the quantification of tannins, the physical–chemical analyses of the adhesives, and the physical–mechanical properties of the plywood panels were subjected to ANOVA and Scott-Knott test at a 95% probability level to evaluate the performance of adhesives formulated with tannins at concentrations of 0, 3, and 5% NaHSO₃. All statistical analyses were performed using Sisvar software, version 5.8.

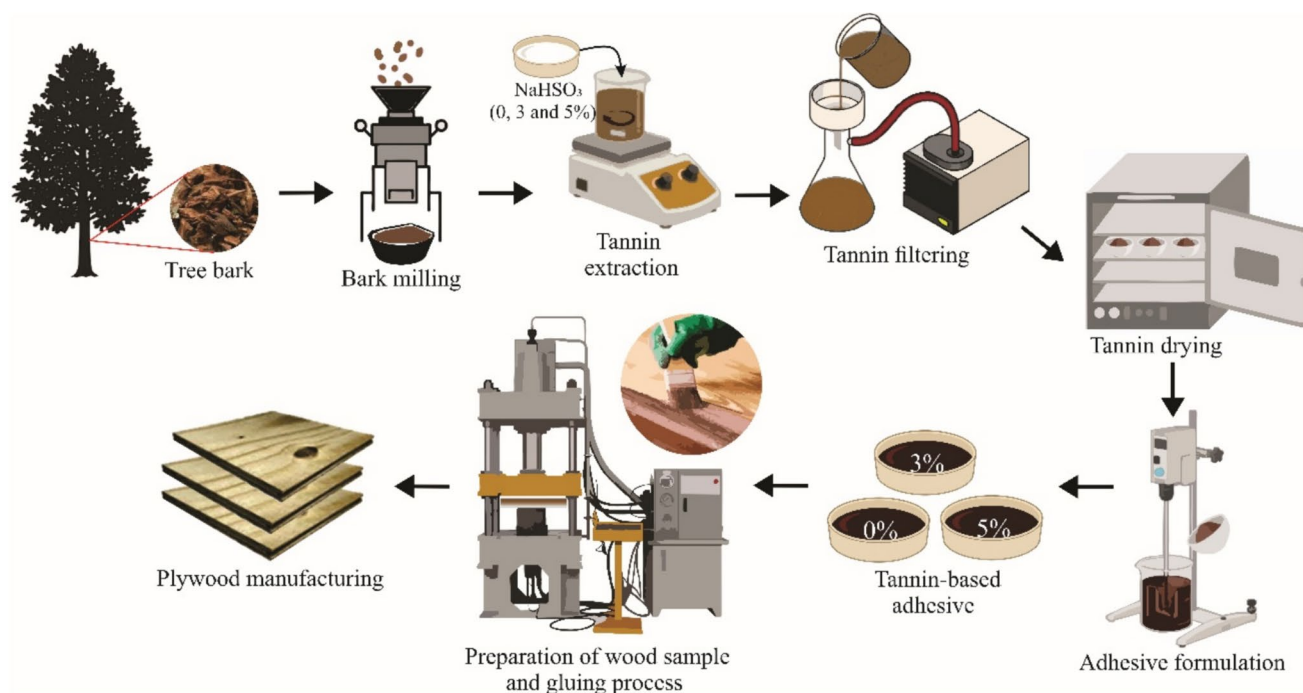


Fig. 1 Adhesive and plywood panel production

Results and discussion

Quantification of condensed tannins from *A. peregrina* bark

The average values in total solids yield (SY), Stiasny index (SI), condensed tannin content (CTT) and non-tannic compounds (NTC) were presented in Table 2.

Total solids yield (SY) represents the percentage of solids extracted from plant species. For *A. peregrina*, SY values ranged from 11.36 to 31.55%, with statistically significant differences between treatments. As shown in Table 2, a progressive increase in SY was observed with increasing sodium bisulfite (NaHSO_3) concentration. This behavior is consistent with the description by Pizzi and Mittal (1994), who reported a directly proportional relationship between salt addition and increased tannin extraction. The presence of salt favors the solubilization of tannins due to the increased water solubility resulting from the opening of the heterocyclic ring of the flavonoid units.

In the study by Sartori et al. (2018), when extracting tannins from the bark of two *Eucalyptus urophylla* hybrids with the addition of 5% sodium sulfite (Na_2SO_3), they observed a significant increase in extraction yield compared to extraction with pure water. Similarly, in the present study, using 5% NaHSO_3 resulted in an almost threefold increase in SY compared to water extraction. Souza et al. (2020) found a 15.1% yield for *Acacia mangium* tannins extracted with pure water, while Araujo et al. (2020) reported a yield of 25.5% for *Myrcia eximia* tannins extracted in pure water and 35.0% with 3% Na_2SO_3 , values higher than those verified in the present work.

Tannins are phenolic compounds with high reactivity toward formaldehyde, a characteristic that highlights their potential to formulate or modify wood adhesives. The Stiasny Index (SI) represents the percentage of condensed tannins in the extract and is obtained through the reaction with formaldehyde and HCl. This index is widely recognized as the main parameter for evaluating the feasibility of using tannic extracts in adhesives (Chupin et al. 2013).

As shown in Table 2, all SI values were statistically similar and met the minimum limit of 65% established by Yazaki and Collins (1994) as a reference for industrial use. Higher SI values are desirable because they indicate a higher

proportion of condensed tannins, which are responsible for polymerization during adhesive curing, and also reflect a lower presence of non-tannic compounds (Carneiro et al. 2007).

In the study by Araujo et al. (2020), an increase in SI was observed for *Myrcia eximia* with the addition of 3% Na_2SO_3 compared to water extraction. However, in the present study, variations in NaHSO_3 concentration did not result in statistically significant differences in SI, indicating stability of condensed tannin content across different treatments.

Paes et al. (2013), when extracting tannins from *A. colubrina* bark, obtained SI values lower than those verified in this study for *A. peregrina*, with averages of 68.3% for extraction in pure water and 64.52% with 3% Na_2SO_3 . According to Mota et al. (2017), both species have similar chemical compositions, which suggests that the differences in SI values may be related to seasonal or structural factors, such as the collection period and the position of the bark on the trunk. Paes et al. (2010) highlighted the influence of phenophases and trunk height on SI variation, reinforcing the importance of these factors in the extraction process.

The condensed tannin content quantifies the active fraction of tannins present in the analyzed extracts. In this study, values ranged from 9.82 to 25.43%, with the highest average observed in the 5% NaHSO_3 treatment. This result is similar to the values reported for *Acacia mearnsii* (average of 28%), a species widely used in commercial tannin production in Brazil (TANAC 2025). Efficient tannin extraction depends on a balance between salt concentration and extraction time: very low salt concentrations combined with short extraction times are insufficient for large-scale extractions, while long periods combined with high concentrations can compromise tannin quality and increase the presence of non-tannic compounds.

For optimal utilization of tannins during extraction, it is essential to achieve a balanced relationship between high contents of condensed tannins and low percentages of non-tannic compounds. The presence of these residues can be detrimental to adhesive production, as they promote the formation of weaker glue lines and increased viscosity, which are limiting factors for performance in wood panel bonding (Carneiro et al. 2009). Similar behavior has been reported for commercial tannin sources such as *Acacia mearnsii*, in which sulfitation with sodium bisulfite increases tannin solubilization and extraction yield but may also promote the co-extraction of carbohydrates and other non-phenolic compounds when higher concentrations are applied (Pizzi and Mittal 2017). In the present study, although increasing the concentration of the extracting salt enhanced tannin yield, it also led to a concomitant increase in impurity contents. Extractions performed with 3 and 5% NaHSO_3 resulted in higher non-tannin levels compared to extraction with pure water, indicating that the quantitative gain

Table 2 Gravimetric yield of tannins extracted from the bark of *A. peregrina*

Treatments	SY (%)	SI (%)	CTT (%)	NTC (%)
T0	11.3 ± 4.3 a*	86.5 ± 5.1 a	9.8 ± 4.0 a	1.5 ± 1.9 a
T3	24.7 ± 3.9 b	79.5 ± 5.7 a	19.6 ± 2.9 b	5.1 ± 1.4 b
T5	31.5 ± 2.8 c	80.6 ± 4.9 a	25.4 ± 2.1 c	6.1 ± 2.0 b

*Averages followed by the same letter in the column do not differ statistically by the Scott-Knott test at 95% probability

in extraction efficiency may be accompanied by limitations in chemical reactivity and adhesive performance of the extracts.

From a quantitative and functional standpoint, the extraction performed with 3% NaHSO_3 represents an optimal compromise between yield and tannin quality for *Anadenanthera peregrina*. Although higher NaHSO_3 concentrations promoted additional increases in extraction yield, these gains were not accompanied by proportional improvements in the Stiasny index. In contrast, the extract obtained with 3% NaHSO_3 exhibited a comparatively high Stiasny index, indicating a higher proportion of reactive condensed tannins relative to non-tannin components. This behavior is consistent with observations reported for *Acacia mearnsii* tannins, in which moderate levels of sulfitation typically enhance extractability and reactivity, whereas excessive sulfite addition results in diluted phenolic content and reduced adhesive performance (Pizzi 2017; Pizzi and Mittal 2017). Therefore, while the general sulfitation paradigm observed in commercial tannins is confirmed, the identification of 3% NaHSO_3 as the optimal condition for *A. peregrina* highlights a species-specific balance between extraction efficiency, chemical reactivity, and potential applicability in wood adhesive formulations.

FTIR of condensed tannins extracted from *A. peregrina* bark

The large-scale use of tannins in industrial processes requires their characterization using non-destructive, rapid,

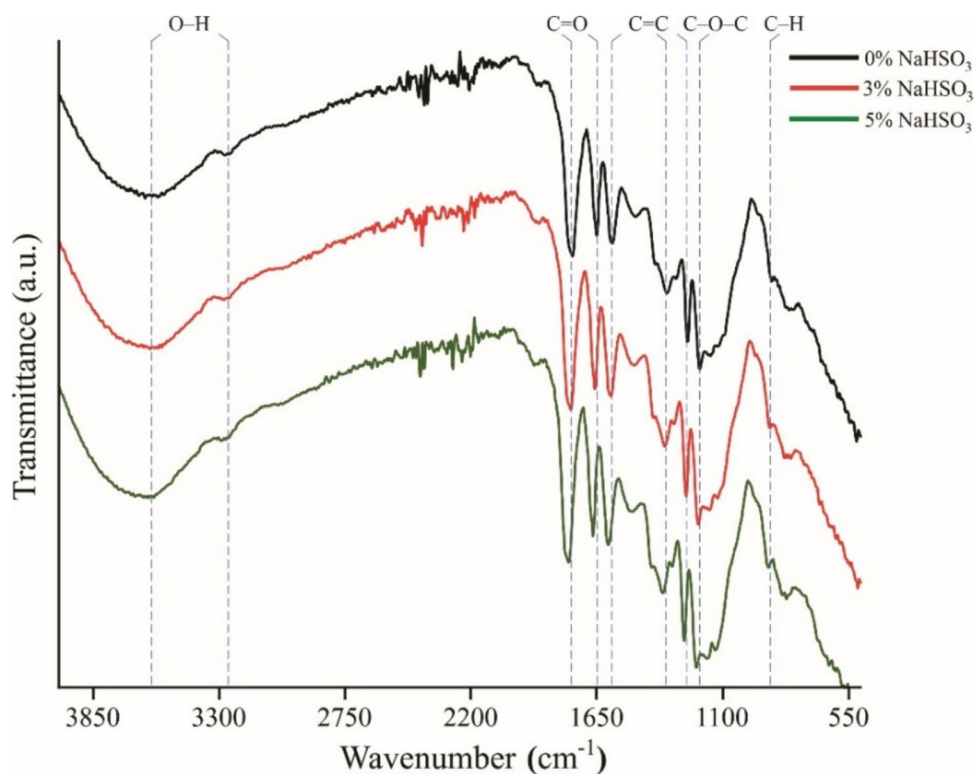
and reliable methods which provide accurate information on their physicochemical properties. In this context, FTIR has emerged as one of the most widely used techniques, offering consistent data in a simplified format, with high spectral resolution and short analysis times (Ricci et al. 2015; Gierczak et al. 2017). Figure 2 presents the FTIR spectra of *A. peregrina* tannins, extracted with 0, 3, and 5% NaHSO_3 , over the spectral range from 4000 to 500 cm^{-1} .

When analyzing the results, it can be stated that, in general, all spectra retain characteristic bands of the tannin matrix, indicating that the basic structure of the material was preserved after sulfitation. However, relevant variations in the intensity and definition of specific bands highlight the presence of sulfonated groups in the chemical structure of the macromolecules.

The spectral band between 3600 and 3200 cm^{-1} is attributed to the stretching vibrations of hydroxyl (O–H) groups, present in both phenolic and aliphatic structures. This region shows changes in intensity and band broadening with increasing NaHSO_3 concentration, because the introduction of sulfonate groups promotes stronger intermolecular interactions with water molecules (Socrates 2004; Ricci et al. 2015).

According to Soto et al. (2005), peaks located between 1400 and 2000 cm^{-1} demonstrate the aromatic nature of the tannin structure. The region from 1800 to 1680 cm^{-1} is particularly relevant for qualitative analysis, as it may indicate the presence of flavonoids or oxidation products of

Fig. 2 FTIR spectrum of *A. peregrina* tannins extracted in different concentrations of NaHSO_3



hydroxyl groups present in flavanol molecules, as a consequence of the extraction process. The bands between 1760 and 1700 cm^{-1} correspond to the stretching of $\text{C}=\text{O}$ bonds, while the band close to 1717 cm^{-1} is attributed to $\text{C}=\text{O}$ bonds, associated with the interaction of catechin with the polymer matrix (Torreggiani et al. 2008). The variations in the intensity of these bands after sulfitation indicate chemical modifications of these compounds, without compromising their aromatic structure.

The bands located between 1620 and 1400 cm^{-1} are associated with stretching vibrations of $\text{C}=\text{C}$ bonds in aromatic rings, presenting peaks of varying intensities (from strong to medium). The intensity of these bands may be influenced by the stretching of the C4–C8 interflavonoid bond, observed during the condensation process in proanthocyanidins. The band 1626 cm^{-1} , in particular, may be related to the catechin monomer (Kim et al. 2007).

The region between 1450 and 900 cm^{-1} , of great structural complexity, is considered one of the most informative for the characterization of substituents in aromatic rings. This range presents typical bands resulting from the combination of aromatic C–H bendings and C–O and C–OH deformations (Özacar et al. 2008; Torreggiani et al. 2008).

For sulfonated tannins, the spectral region between 1000 and 1300 cm^{-1} is fundamental, as it is attributed to $\text{C}=\text{C}$ stretching vibrations characteristic of the $\text{CR}_2\text{--CHR--CR}(\text{SO}_3^{2-})$ structure. In Fig. 2, a more intense band can be observed at 1252, 1262, and 1266 cm^{-1} for tannins extracted with 0, 3, and 5% NaHSO_3 , respectively, which is due to the opening of the pyran ring during tannin sulfitation (Hoong et al. 2009). The comparison of the infrared signals and their relative intensities indicates that the proportion of heterocyclic C-ring opening in the flavonoid unit depends on the concentration used for tannin extraction. Thus, it is observed that pyran ring opening during extraction appears to be more significant when 5% of the extracting salt is used.

The region from 1225 to 950 cm^{-1} is related to in-plane bending of aromatic C–H bonds. The bands observed in this range are sensitive to the number and position of substituents, indicating the presence of monomeric constituents in tannins. Further, the range between 900 and 740 cm^{-1} is associated with oscillations of O–H groups of aromatic alcohols, in addition to out-of-plane deformations of the aromatic ring (Ricci et al. 2015).

Table 3 presents a summary of the main bands observed in this study for *A. peregrina* tannins.

Thermogravimetric analysis of tannins extracted from *A. peregrina* bark

Figure 3 presents the thermograms of tannins extracted with 0, 3, and 5% NaHSO_3 , highlighting the thermal behavior of

Table 3 Summary of FTIR spectrum characteristics of tannins from *A. peregrina*

0% NaHSO_3	3% NaHSO_3	5% NaHSO_3	Bandwidth assignment
3553	3551	3590	O–H stretching
3255	3252	3256	O–H stretching
1759	1769	1771	C=O stretching
1698	1703	1707	C=O stretching
1649	1657	1663	Aromatic C=C stretching
1621	1629	1631	C=C stretching
1581	1587	1593	C=C stretching
1478	1493	1499	Aromatic C=C stretching
1342	1348	1360	C=C stretching vibrations
1252	1262	1266	C–O–C stretching of the pyran ring groups
978	979	983	In-plane C–H bending

the extracts. The TG curve (Fig. 3a) shows the progressive mass loss with increasing temperature, while the DTG curve (Fig. 3b) highlights the most intense degradation events through the peaks corresponding to the highest weight loss rates.

Figure 3a indicates that all tannin extracts exhibit similar overall thermal degradation profiles, although differences in mass loss intensity and peak positions are observed depending on the NaHSO_3 concentration. The first mass loss event occurs below approximately 120–150 °C and is mainly attributed to the evaporation of free and weakly bound water, as well as residual low-molecular-weight volatile compounds. This event is clearly evidenced by the intense negative DTG peaks observed in this temperature range, particularly near 100 °C (Fig. 3b), indicating a high rate of mass loss associated with moisture removal rather than chemical degradation of the tannin structure.

A second and more gradual mass loss stage is observed between approximately 200 and 350 °C, which can be associated with the onset of thermal decomposition of less stable functional groups and side chains of condensed tannins. In this region, the DTG curves show broader and less intense peaks, indicating a lower degradation rate compared to the moisture loss stage.

The main structural thermal degradation of the tannin matrix occurs predominantly between 350 and 500 °C, corresponding to the breakdown of phenolic structures, cleavage of interflavonoid bonds, and degradation of the aromatic backbone typical of condensed tannins (Pena et al. 2009; Araujo et al. 2020). In this temperature interval, the TG curves reveal that the extract obtained without NaHSO_3 (0%) exhibits a higher total mass loss, indicating lower thermal stability compared with the samples extracted using 3 and 5% NaHSO_3 .

The DTG curves further support this behavior, as the samples containing NaHSO_3 present broader and slightly shifted degradation peaks toward higher temperatures, particularly in the 350–500 °C range, suggesting delayed

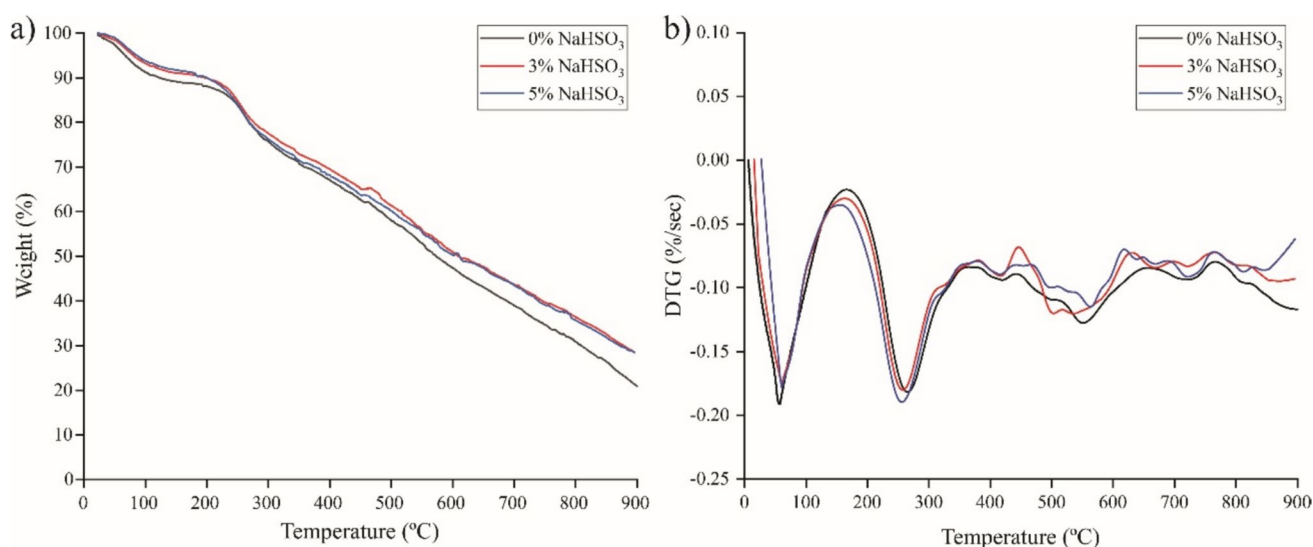


Fig. 3 Thermal degradation of tannins extracted in 0, 3 and 5% NaHSO₃. **(a)** weight loss with increasing temperature; **(b)** first derivative of TGA (DTG)

thermal decomposition. This shift indicates that the addition of bisulfite influences the chemical nature of the extracted tannins, possibly favoring fractions with higher molecular weight or increased structural condensation, which enhances thermal stability (Chupin et al. 2015). Above 500 °C, a slow and continuous mass loss is observed for all samples, which can be attributed to the gradual decomposition of more thermally stable carbonaceous structures and char formation.

Overall, although the most intense DTG peaks are observed at lower temperatures due to moisture loss, the effective structural degradation of tannins occurs at higher temperatures, mainly between 350 and 500 °C. The presence of NaHSO₃ positively affects thermal stability, which is particularly relevant for applications such as adhesives and other materials subjected to elevated processing or service temperatures.

Physicochemical properties of tannic adhesives and phenol–formaldehyde

Table 4 presents the physicochemical properties of adhesives formulated with different concentrations of NaHSO₃ and a commercial phenol–formaldehyde adhesive. The PF

treatment presented the highest solids content (75.68%), significantly higher than the other treatments, which ranged from 36.11 to 39.84%. This result is consistent with the industrial formulation of phenolic adhesives, which are prepared with a high concentration of solid resins to ensure greater bonding efficiency and final product strength.

Gonultas (2018) cited that tannin-based adhesives can form an efficient glue line even at lower solids contents. This occurs because, at higher concentrations, viscosity tends to increase, which can hinder the application and penetration of the adhesive into the substrate. This trend is confirmed by the values obtained in the present study, which observed that adhesives with higher solids contents consequently presented higher viscosities.

Carneiro et al. (2009), when formulating tannic adhesives extracted with 3% Na₂SO₃, reported a solids content of 45.5%, pH 3.2, gel time of 133.3 s, and viscosity of 290 cP. In the present study, different values were obtained. Although the tannins were extracted from the same plant species, the difference in the extracting salt used, as well as factors such as the time and region of bark collection, may have influenced the results. Furthermore, bark processing may also have contributed to these variations,

Table 4 Physicochemical properties of adhesives used in plywood production

Treatments	Solids Content (%)	pH	Gel Time (s)	Viscosity (cP)
T0	39.84 ± 3.1 a*	5.73 ± 0.8 a	262.2 ± 21.0 a	336.946 ± 29.8 b
T3	36.11 ± 2.1 a	5.75 ± 0.9 a	315 ± 52.0 b	106.470 ± 19.0 a
T5	36.69 ± 2.5 a	4.53 ± 1.0 a	382.2 ± 41.2 b	101.849 ± 25.5 a
PF	75.68 ± 2.9 b	11.4 ± 1.2 b	466.7 ± 29.3 c	748.396 ± 38.4 c

T0 = 0% NaHSO₃; T3 = 3% NaHSO₃; T5 = 5% NaHSO₃; PF = Phenol–Formaldehyde. *Averages followed by the same letter in the column do not differ statistically by the Scott-Knott test at 95% probability.

since aspects such as material particle size and environmental conditions directly affect the quality and yield of tannin extraction (Hoong et al. 2009; Chaves et al. 2021).

The pH of tannin-based bioadhesives shows their acidity profile, in contrast to the typically basic nature of phenol–formaldehyde-based adhesives. According to Iwakiri and Trianoski (2020), the pH of adhesives should be between 2.5 and 11, as extreme values can compromise bond quality. In the present study, the lowest pH values were observed in tannin adhesives formulated with NaHSO_3 . Zhang et al. (2010) point out that excessively acidic pHs can generate a corrosive environment along the bond line, which can accelerate panel degradation. According to Brosse and Pizzi (2017), pH significantly influences the gel time of adhesives, while Dunky (2021) report that viscosity tends to increase at higher pHs, depending on the type of tannin used.

The gel time of the tannic adhesives was shorter than that of the commercial phenol–formaldehyde. This can be attributed to the molecular structure of tannins, which, because they are high molecular weight compounds, have a molecular growth rate higher than the rate of bond formation, favoring faster gelation and curing, in addition to a shorter shelf life compared to synthetic phenolic adhesives (Brosse and Pizzi 2017). Souza et al. (2020), when comparing adhesives formulated with *Acacia mangium* and phenol–formaldehyde, observed a gel time of 240 s for acacia and 787 s for the phenolic adhesive, a value significantly higher than that found in the present study.

The shortest gel time was obtained in the bioadhesive formulation extracted with pure water. As the NaHSO_3 concentration increased, the gel time also increased. However, this behavior was inversely proportional to viscosity, since higher bisulfite concentrations resulted in bioadhesives with reduced viscosity. This effect may be related to the breaking of intermolecular bonds between tannins by the reducing action of bisulfite, which reduces the viscosity of the mixture.

According to Pizzi (1994), the use of salts for tannin extraction for adhesives production promotes the opening of the C-ring of the condensed tannin structure, resulting in the introduction of polar phenolic hydroxyl groups. This structural modification increases the solubility of the extracts in aqueous media and, consequently, reduces their viscosity. Similar results were reported by Noreljaleel et al. (2020), who observed a reduction in the viscosity of adhesives based on *Acacia* tannins extracted with NaHSO_3 . The authors attributed this behavior to the introduction of bisulfite groups at the C2 carbon of the tannin monomer ring, promoting the cleavage of the ether bond in the heterocyclic C-ring. This structural disruption led to a decrease in the length of the polymer chains, directly contributing to the reduction in the viscosity of the extract.

The addition of NaHSO_3 significantly alters the physicochemical properties of tannin-based adhesives, modulating

their acidity, reactivity, and fluidity. This modulation can be exploited for developing more sustainable alternative adhesives, adjusting the formulation according to the requirements of the production process and the desired physical and mechanical performance of the panels.

Physical properties of the plywoods

The apparent density of the panels ranged between 0.74 and 0.79 g/cm^3 , with no statistically significant differences between treatments (Fig. 4a). This suggests that the partial replacement of the phenol–formaldehyde (PF) adhesive with tannins associated with different concentrations of NaHSO_3 did not significantly influence the compaction of the panels during pressing. The maintenance of density between treatments indicates that the adhesives formulated with tannins presented adhesion and curing capacities compatible with PF, at least concerning the structural consolidation of the panel.

Regarding moisture content, values ranged from 4.66% (T3) to 5.99% (PF), also with no statistical differences between treatments (Fig. 4b). The slight reduction in moisture content in T3 may be associated with lower water retention by the adhesive with 3% NaHSO_3 , which may reflect greater efficiency in the polymerization reaction or lower hygroscopicity of the adhesive system. However, as no statistically significant differences were observed between treatments, it is not possible to state that the addition of tannins or sodium bisulfite influenced moisture retention in the panels. Even so, all treatments presented values below the maximum limit of 12% established by ABIMCI (2019), which is relevant from a technical point of view, since moisture contents above this limit can compromise the physical and mechanical performance of the panels.

The plywood density is related to several factors, such as the forest species used, the moisture content of the veneers, as well as the temperature and pressure applied during pressing. According to Bekhta et al. (2020), when high pressures are not used, the density of plywood can be approximately 5% higher than that of the original wood. The panels produced in this study had an apparent density approximately 40% higher than that of the original wood.

Internal bond strength depends largely on the adhesive quality and the density of the lower-density layer in the panel (Pizzi 2019). In the study by Carvalho et al. (2016), when comparing synthetic adhesives (UF and PF) with adhesives formulated with tannins from *Stryphnodendron adstringens* and *Acacia mangium* for plywood production, obtained averages between 0.510 and 0.527 g/cm^3 , lower density values than those found in this research.

According to Hunt et al. (2018), increased density is generally associated with higher volumetric swelling and shrinkage coefficients, as well as stiffness and strength

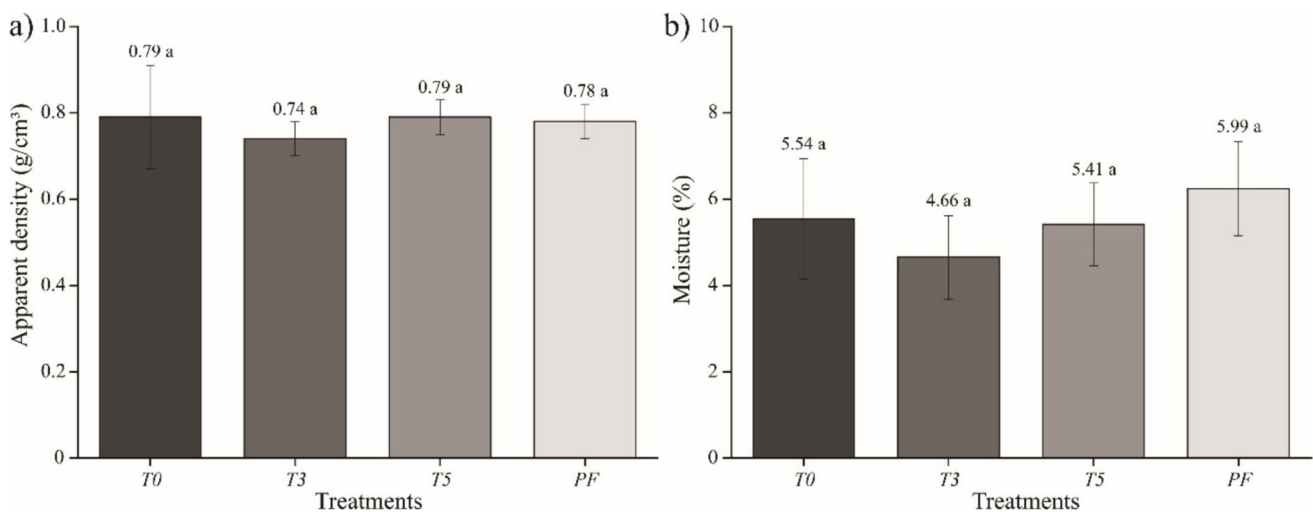


Fig. 4 (a) Apparent density and (b) moisture content of plywood. *Averages followed by the same letter do not differ significantly according to the Scott-Knott test at the 95% probability level

properties, resulting in higher internal stresses in response to moisture variations. Furthermore, wood density directly influences adhesive penetration: less dense woods tend to generate hungry glue lines, while denser woods can hinder adhesive penetration (Iwakiri and Trianoski 2020).

Therefore, since no significant differences were observed between the density and moisture content of the panels bonded with tannin adhesives and those bonded with the commercial adhesive, it can be inferred that the panels produced with tannin-based adhesives presented suitable properties for structural applications.

Figure 5 presents the water absorption results of the plywood panels after 2 and 24 h of immersion. It can be seen that the tannin treatments modified with 3% (T3) and 5% (T5) NaHSO_3 presented higher absorption values compared to the tannin treatment extracted with pure water (T0) and the commercial phenol–formaldehyde adhesive, especially after 2 h of immersion. This suggests that extraction with NaHSO_3 of tannins, while it can improve certain chemical properties, tends to increase the hydrophilicity of the adhesive, favoring greater water absorption in the first time.

After 24 h of immersion, the same behavior is observed: treatments T3 (48.67%) and T5 (49.12%) keep the highest absorption values, significantly higher than those of treatments T0 (38.41%) and PF (38.58%). T0 and PF do not differ from each other, as well as T3 and T5, demonstrating that the panels with tannins extracted with NaHSO_3 were more susceptible to water penetration over time.

This result may be related to the degree of cross-linking of the polymer network formed by the adhesives. The phenol–formaldehyde adhesive is known for forming a highly cross-linked and water-repellent three-dimensional structure, which explains its good performance against water

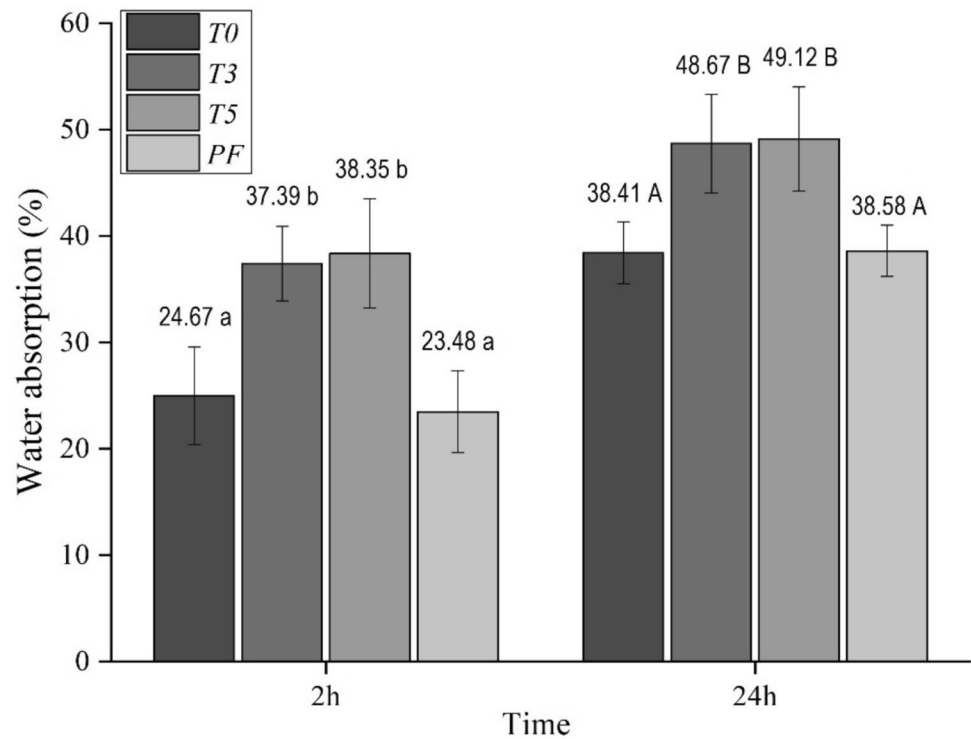
absorption. On the other hand, the sulfited tannins may have undergone secondary reactions that reduced the cross-link density or introduced more polar groups, increasing their affinity for water. According to Pizzi (1979), sulfitation increases the solubility of adhesives; however, in the absence of efficient cross-linking, this process can compromise the water resistance of the panels, making them more susceptible to moisture absorption.

In terms of performance, both T0 and PF demonstrated more stable behavior and resistance to water penetration, making them more suitable for applications where exposure to moisture is a critical factor. Comparing with the results obtained by Setter et al. (2021), it is observed that the treatment with phenol–formaldehyde adhesive after 24 h showed greater water absorption (62.76%) than the PF treatment evaluated in this study (38.58%). According to Bekhta et al. (2020), water absorption is related to the panels density. Panels with higher density tend to have a smaller number of pores and, consequently, lower water absorption. In this study, the increased water absorption in the panels is closely related to the structural changes induced by NaHSO_3 in the tannin molecule. The sulfitation process promotes the opening of the C-ring in condensed tannin structures, resulting in the introduction of sulfonic groups and an increase in free phenolic hydroxyls. These groups are highly polar and hydrophilic, which enhances the adhesive's affinity for water and, consequently, reduces the moisture resistance of the glue line in the produced panels (Pizzi 1994).

Mechanical properties of plywoods

Figure 6 shows the average values of the Modulus of Elasticity (MOE) and Modulus of Rupture (MOR) obtained in

Fig. 5 Water absorption of plywood. *Averages followed by the same letter do not differ significantly by the Scott-Knott test at the 95% probability level



the static bending of plywoods produced with tannin-based adhesives extracted with different concentrations of NaHSO_3 (T_0 , T_3 and T_5) and commercial adhesive based on phenol-formaldehyde (PF), evaluated in the directions parallel and perpendicular to the fibers.

The panels bonded with PF adhesive presented the highest MOE values, both in the parallel (13,293.5 MPa) and perpendicular (3549.7 MPa) directions, demonstrating the excellent mechanical performance associated with the high strength of the phenolic adhesive. Among the tannin-based

treatments, the 3% NaHSO_3 concentration (T_3) promoted the greatest increase in MOE in the parallel direction (10,911.3 MPa), achieving performance comparable to that of the commercial adhesive. This result can be attributed to the optimized reactivity of the sulfonated tannins at this concentration level, which favors the formation of a more rigid polymeric network in the adhesive matrix.

In the perpendicular direction, treatment T_3 (3006.9 MPa) showed intermediate performance, superior to T_0 (2432.3 MPa) and T_5 (2017.7 MPa), suggesting that the

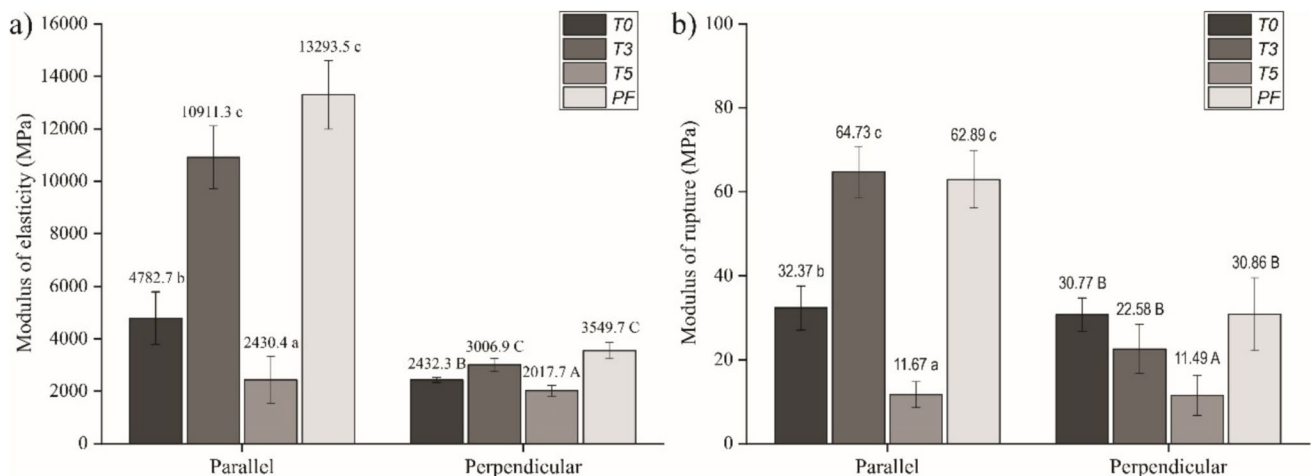


Fig. 6 (a) Modulus of elasticity and (b) rupture with static bending in the parallel and perpendicular direction of the grain. *Averages followed by the same letter do not differ significantly by the Scott-Knott test at the 95% probability level

action of sulfite may not be linear in different load application directions. In general, using NaHSO_3 contributed to improving the stiffness of the panels, especially in the direction parallel to the fibers.

Regarding MOR, the commercial adhesive (PF) showed values of 62.89 MPa in the parallel direction and 30.86 MPa in the perpendicular direction. Among the tannin-based adhesives, treatment T3 stood out with values of 64.73 MPa and 22.58 MPa, respectively, being statistically equivalent to PF. This suggests that the addition of 3% NaHSO_3 significantly improves in rupture strength, possibly due to forming a more cohesive and resistant polymer network. In contrast, the T5 treatment resulted in inferior performance, especially in the parallel direction (11.67 MPa), which may indicate excess sulfite, compromising the structural integrity of the adhesive.

Based on the results, the panels bonded with tannins extracted in the presence of 3% NaHSO_3 presented greater stiffness and lower elasticity among the concentrations evaluated. This treatment was the only one that met the minimum values required by ABNT (1986) standard, which establishes, for the modulus of elasticity (MOE), values of 5223 MPa (parallel) and 1485 MPa (perpendicular), and, for the modulus of rupture (MOR), 30.9 MPa (parallel) and 14 MPa (perpendicular).

Carvalho et al. (2016), when using 3% Na_2SO_3 in the extraction of tannins from barbatimão, obtained average MOR and MOE values in the parallel direction of 46.03 MPa and 3877.93 MPa, and in the perpendicular direction of 29.25 MPa and 2018.21 MPa, respectively. Souza et al. (2020), in turn, used *Acacia mangium* tannins extracted in pure water to formulate adhesives, recording average MOR values of 59.40 MPa (parallel) and 32.70 MPa (perpendicular), and MOE of 6359 MPa and 1836 MPa, respectively. In the present study, the adhesives formulated with tannin extracted with 3% NaHSO_3 (T3) showed superior performance to those of the cited works, with average MOR and MOE values in the parallel direction of 64.73 MPa and 10,911.34 MPa, and in the perpendicular direction of 22.58 MPa and 3006.95 MPa. It is noteworthy, however, that only the average perpendicular MOR value was lower than that recorded by Carvalho et al. (2016).

Thus, the results demonstrate that the addition of NaHSO_3 at concentrations 3% improves the mechanical properties of the panels, while excessive concentrations (5%) can compromise performance. In comparison, the adhesive T3 proved to be competitive with the commercial adhesive PF, indicating potential application in plywood panels with suitable mechanical performance. The main objective of the shear test is to evaluate the quality of the glue line, which is essential for classifying the plywood according to its end use: interior, intermediate, or exterior. Tannin and phenol–formaldehyde-based adhesives were evaluated for shear strength and failure rate in dry wood (Fig. 7).

The results indicate significant differences between treatments, highlighting the influence of the adhesive formulation on the integrity of the glue line (Fig. 7a). The commercial PF adhesive presented the highest shear strength (8.43 MPa), a value statistically superior to the other treatments, demonstrating its efficiency in forming cohesive and strong bonded joints. This performance is attributed to the high reactivity of formaldehyde with phenolic groups, resulting in a highly crosslinked and delamination-resistant polymer matrix.

On the other hand, adhesives T0, T3, and T5 presented significantly lower strengths, ranging from 1.37 to 2.31 MPa, with no statistical differences between them. These results are in agreement with data obtained by Lopes et al. (2021), who, when extracting tannins from *Mimosa tenuiflora* bark using solutions with 0 and 5% NaHSO_3 , observed a reduction of approximately 10% in shear stress between panels produced with tannic adhesives extracted at 0% and those extracted at 5% sulfite. In the present study, the reduction was more pronounced, reaching approximately 40%. This may be related to the excessive sulfitation of the tannin phenolic groups, which compromises the formation of effective crosslinks in the adhesive matrix, resulting in more fragile bonds.

The sulfitation process of tannins allows modulation of adhesive viscosity; however, very low or very high values should be avoided. As shown in Table 4, treatments T3 and T5 resulted in reduced average viscosities, which may have contributed to the formation of starved glue lines, partially compromising the shear strength of the panels. Thus, although adhesive T3 produced a panel with excellent composite stiffness and bending strength, the bond line itself represents the weak point responsible for the low shear performance, likely due to rheological issues and inadequate glue-line penetration. Nevertheless, the results indicate that, despite the observed limitations, the tannins used demonstrated adhesive potential. The formulation employed in this study was sufficient to achieve adequate bond-line strength, meeting the minimum requirements established by standards such as EN 314–2 (1993), which requires values above 1.0 MPa for indoor applications.

Figure 8 shows the glue line of the plywood glued with tannic adhesives under different concentrations of NaHSO_3 and plywood glued with the phenol–formaldehyde formulation. Since the panels glued with phenol–formaldehyde presented excellent mechanical properties, observing Fig. 8d, it is possible to infer that their viscosity was adequate, not causing a thick glue line.

Regarding wood failure, the percentage values are presented in Fig. 7b. According to ASTM (2018), for bonding dry laminate joints and other non-structural joints, a minimum of 60% visible wood failure in the shear plane is required, which indicates effective adhesion and good interaction at the adhesive-wood and adhesive-adhesive interfaces. In general, the higher the percentage of wood

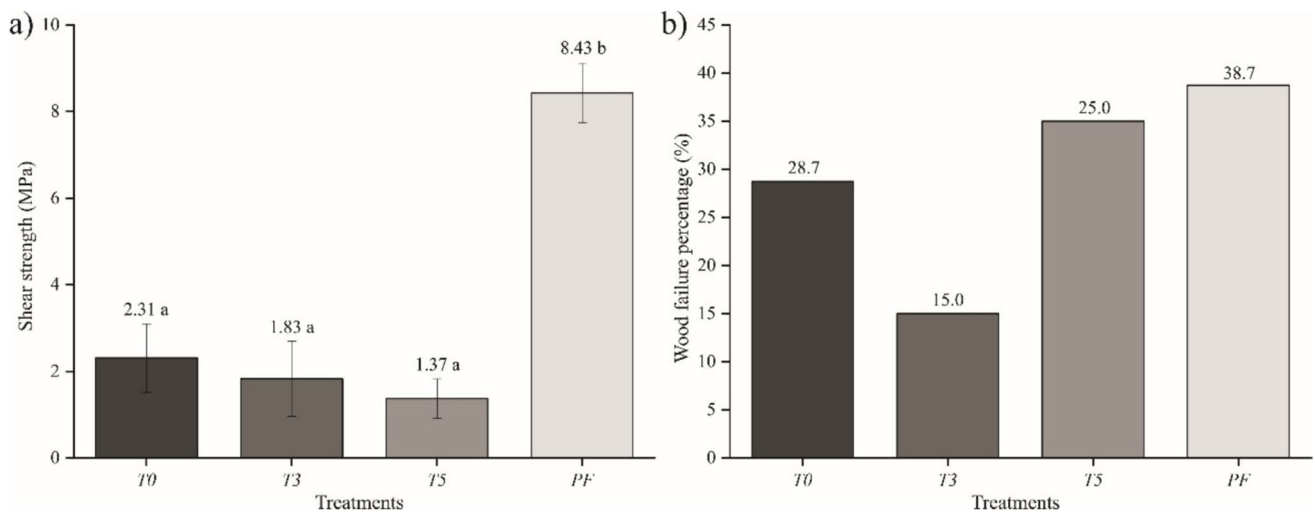
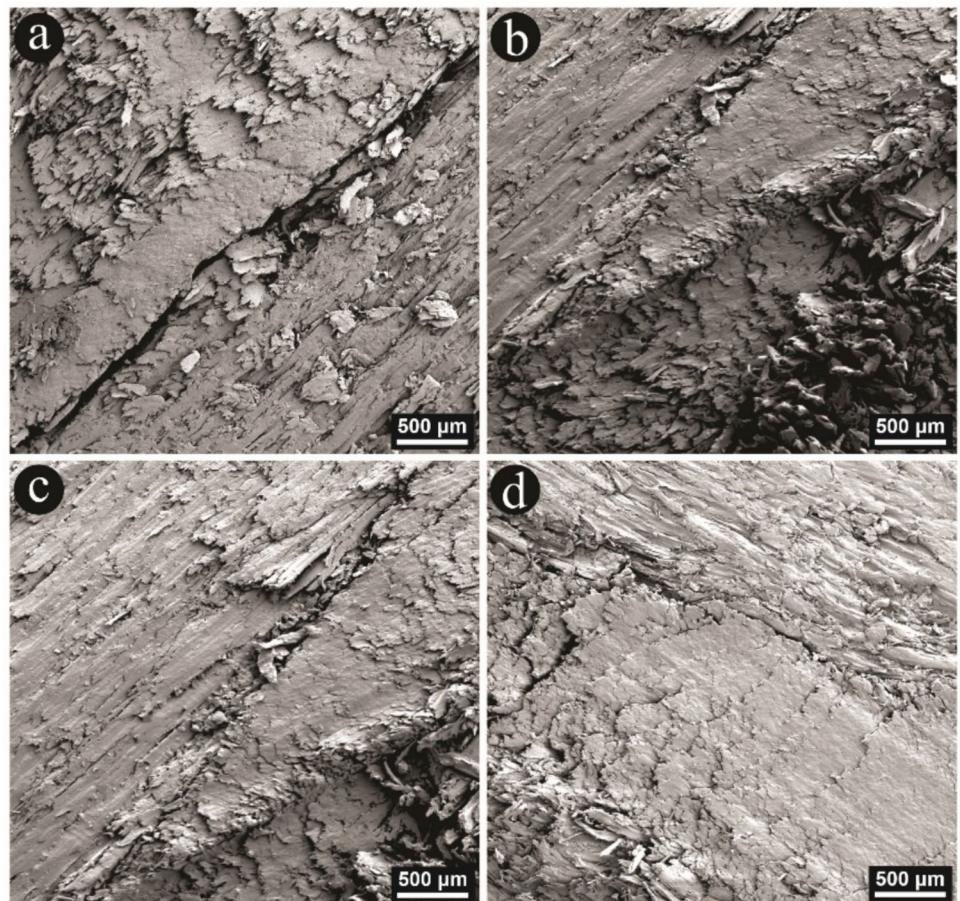


Fig. 7 (a) Shear strength and (b) Percentage of failure in dry conditions. *Averages followed by the same letter do not differ significantly by the Scott-Knott test at the 95% probability level

failure, the better the bond quality, as it means that the failure occurred preferentially in the substrate (wood) and not at the glue line.

None of the adhesives evaluated in this study met the minimum 60% limit established by ASTM (2018), indicating that glue line strength is still below the ideal for more demanding structural applications. However, when

Fig. 8 Fractured wood surface after dry shear test. **a)** plywood glued with tannic adhesive extracted in 0% NaHSO_3 ; **b)** plywood glued with tannic adhesive extracted in 3% NaHSO_3 ; **c)** plywood glued with tannic adhesive extracted in 5% NaHSO_3 and **d)** plywood glued with phenol–formaldehyde



considering the criteria of the European standard EN (1993), wood failure values are not decisive for bond approval, as long as shear strength values are within the minimum specified limits. Therefore, all treatments met the requirements of the aforementioned standard, even with low wood failure rates, suggesting technical feasibility for applications in indoor or non-structural environments, as long as the use limits defined by the standard are respected.

Therefore, although some tannin treatments demonstrated satisfactory mechanical properties (MOE and MOR), the shear strength results indicate limitations of these adhesives without additional modifications to the formulation or curing strategy. Complementary studies with co-adhesives, catalysts or hybrid systems may be necessary to enable the technical use of tannin adhesives as a replacement for conventional synthetic adhesives.

Conclusion

The study demonstrates that tannins extracted from the bark of *A. peregrina* have high potential for the formulation of bioadhesives, exhibiting high Stiasny indices that corroborate their industrial viability. Chemical modification via sulfitation proved to be promising, as the addition of sodium bisulfite (NaHSO_3) significantly increased extraction yield and reduced adhesive viscosity, facilitating handling and application. Among the tested concentrations, the treatment with 3% NaHSO_3 stood out as the most balanced, meeting the requirements of ABNT NBR 9531 and EN 314–2 standards for static bending mechanical properties and shear strength. However, the higher water absorption observed in the sulfitated panels indicates increased adhesive hydrophilicity, which restricts the use of these panels to indoor environments. In summary, the *A. peregrina* tannin adhesive at 3% represents a sustainable alternative for replacing commercial phenolic resins. To enable more demanding structural applications, future studies should investigate the use of co-adhesives or crosslinking agents to enhance moisture resistance and bond-line cohesion without compromising system flowability.

Acknowledgements The authors would like to thank the Wood Science and Technology Program (PPGCTM) from Federal University of Lavras (UFLA) for providing research materials and infrastructure. We also thank the National Council for Scientific and Technological Development (CNPq, process 150494/2024-6), Minas Gerais Research Foundation (FAPEMIG), and the Coordination for the Improvement of Higher Education Personnel (CAPES) for their financial support for researcher.

Author contribution All authors contributed to the study conception and design. Michele Lopes Medina: Conceptualization, Methodology and Writing—original draft. Felipe Gomes Batista: Writing – review & editing. Ana Carolina Corrêa Furtini, Carolina Aparecida dos Santos and Caroline Junqueira Sartori: Investigation, Methodology, Data

Curation and Formal analysis. Thaís Brito Sousa and Mário Vanoli Scatolino: Validation, Revision and Translation. Lourival Marin Mendes, Fábio Akira Mori and José Benedito Guimarães Junior: Supervision, funding acquisition and Resources. All authors read and approved the final manuscript.

Funding The Article Processing Charge (APC) for the publication of this research was funded by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) (ROR identifier: 00x0ma614). The research was funded by CNPq, CAPES and FAPEMIG.

Data availability All information was constructed by the authors and can be published by the journal.

Declarations

Ethics approval and consent to participate The authors' participation was authorized during the construction of the study.

Consent for publication All authors agree with the publication of the article in this journal.

Competing interests The authors confirm that there is no conflict of interest regarding the submission.

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