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ARTÍCULO

Utilization of nanotalc modified adhesives in plywood panels

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Abstract:
As an alternative for the partial replacement of synthetic adhesives are the modifications that occur still during their synthesis, to improve and adhere new properties. Among the possible materials used in nanoscale, talc is a mineral material of natural origin and a promising raw material due to its low cost, lightness, natural hydrophobicity. Due to the scarcity of studies with the insertion of nanotalc in adhesives, this study aimed to produce plywood panels bonded with phenol-formaldehyde adhesive nanomodified with talc to evaluate its physical-mechanical resistance when compared to conventional synthetic adhesives. To carry out this study, three *Pinus oocarpa* trees with 28 years of age were used. Different concentrations of talc were used in the formulation with the phenol-formaldehyde adhesive, being the treatments 0; 0,05; 1; 1,5 and 2 % of talc (mass/mass) in relation to the adhesive, with three panels per treatment, totaling 15 panels. The pressing time was 8 minutes with temperature of 160 ºC and pressure of 1 MPa. The physical-chemical characteristics of the lignocellulosic material and of the plywood panels were determined. The quality of the adhesive produced was demonstrated by its resistance to water absorption with the insertion of up to 2 % talc to the adhesive, reducing it considerably. From 1 % talc inserted, the plywood panels had their mechanical characteristics superior to those glued with pure phenol-formaldehyde adhesive. However, as the study is innovative in nature, further research should focus on the application and quality evaluation of other talc nanomodified adhesives on different types of wood panels.

Keywords: Elastic modulus, nanotechnology, *Pinus oocarpa*, synthetic adhesives, wood adhesives, wood-composites, wood panels.

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Introduction

An effective bonding process during the manufacturing of wood panels is crucial to ensure product quality. For adhesion to occur, the physical and chemical phenomenon establishing the connection between solid surfaces, there must be a good wood-adhesive-wood interaction. Therefore, the choice of adhesive for bonding wood products is fundamental (Pizzi *et al*. 2020).

Prevalent adhesives in wood panel manufacturing are derived from non-renewable sources such as phenol and urea-formaldehyde (Young *et al*. 2019). Faced with the oil crisis in the 1970s and growing environmental concerns, research has explored the partial or total replacement of phenolic adhesives with bioadhesives that exhibit comparable or superior physical-mechanical characteristics to conventional ones (Kumar and Leggate 2022, Gu *et al.* 2019). Several studies support the feasibility of reducing the use of synthetic adhesives through the incorporation of nanoscale reinforcing structures (Zidanes *et al*. 2023, Nicolao *et al*. 2022, Huang *et al.* 2023, Shirmohammadli *et al.* 2018). The use of nanoparticles in adhesives tends to improve their specific properties such as mechanical reinforcement, wood surface adhesion, moisture resistance, panel dimensional stability, thermal, barrier, chemical properties, among others, and has received great interest in research for various applications (Zhou and Du 2020, Ahmadi-Dehnoei and Ghasemirad 2021, Antov *et al.* 2021b).

Among the benefits mentioned, talc, a natural mineral material, becomes a promising raw material for use on a nanoscale due to its low cost, lightweight, and natural hydrophobicity. For this reason, its use has been favored in various sectors such as improving the ductility of materials, nano-reinforcement in polypropylene (Yousfi *et al*. 2013, Savini *and* Oréfice 2020).

In view of the above, this research aims to contribute information on the characterization of phenol-formaldehyde adhesives reinforced with different concentrations of talc nanoparticles, thus enabling the development of superior characteristics compared to pure phenol-formaldehyde, as well as expanding the research and, in the future, the use of these adhesives in the wood composites industry.

Material and methods

Obtaining and preparing the raw material

To carry out this study, three ocote pine (*Pinus oocarpa* Schiede ex Schltdl.) trees with 28 years of age were used, located in an experimental plantation on the campus of the Federal University of Lavras. Brazil (21°14'43" S, 44°59'59" W) with climate classified according to Köppen, type Cwa. Five disks were removed from each tree at heights corresponding to 0 %, 25 %, 50 %, 75 % and 100 % of the commercial height, for chemical characterization and basic density.

The wood veneers were obtained in a veneer lathe, after heating the logs at a temperature of 70 ºC, for a period of 24 h in water, as recommended by Iwakiri (2011). They were generated with a nominal thickness of 2 mm and guillotined in the dimensions 480 mm x 480 mm (width x length) and later tabiqued in the horizontal plane for natural drying in a covered place until they reached the equilibrium moisture content. Then, the material was dried in an oven with forced air circulation until it reached a final humidity of approximately 8 %.

Subsequently, the veneers were classified into quality classes in accordance with the ABNT NBR 9531 (1986b), where veneers classified as "A" and "B" were used for the cover and the remaining veneers were used for the core of the panels.

Physical and chemical characterization of the lignocellulosic material

The basic density of ocote pine (*Pinus oocarpa* Schiede ex Schltdl.) wood was obtained from the specimens obtained from the opposite wedges, according to the water immersion method, described by the technical standard ABNT NBR 11941 (2003), with five repetitions.

For the analysis of the chemical properties of lignocellulosic material tests were performed in triplicates. Thus, determining the total extractives (TE) ABNT NBR 14853 (2010), the insoluble lignin (IL) ABNT NBR 7989 (2010), ash content (AC) ABNT NBR 13999 (2017) and the holocellulose that was obtained by adding the levels of lignin (IL), extractives (ET) and ash (AC), subtracted from 100, as shown in Equation 1:

$$Holocellulose\ (\%) = 100 - (IL + ET + AC) \qquad (1)$$

Adhesives production

Due to the hydrophobic nature of talc, it was necessary to use a surfactant (Tween 80) to help mix the talc with water and then the solution was added to the phenol-formaldehyde adhesive.

Different concentrations of talc were used in the formulation with the phenol-formaldehyde adhesive, being the treatments 0 %; 0,05 %; 1 %; 1,5 % and 2 % of talc (mass/mass) in relation to the adhesive, with three panels per treatment, totaling 15 panels.

Production and characterization of plywood panels

The plywood panels were made with five sheets arranged crosswise in relation to the direction of the fibers, glued with phenol-formaldehyde commercial (pH 11,8; solid content of 50,37 % and viscosity of 4200 cP), and introduced 10 % solution (water, talc and Tween 80) in its formulation. A grammage of 320 g/m² was used, with a pressing cycle of 10 minutes, the pressing time was 8 minutes with temperature of 160 ºC and pressure of 1 MPa. (Irle *et al*. 2010, Xiong *et al*. 2023, Silva *et al*. 2010, Lisboa *et al*. 2016).

The panel production process is shown in Figure 1, which illustrates the weighing of the amount of adhesive per blade, the spreading of the adhesive and the hydraulic press used in the process, ending with hot pressing.

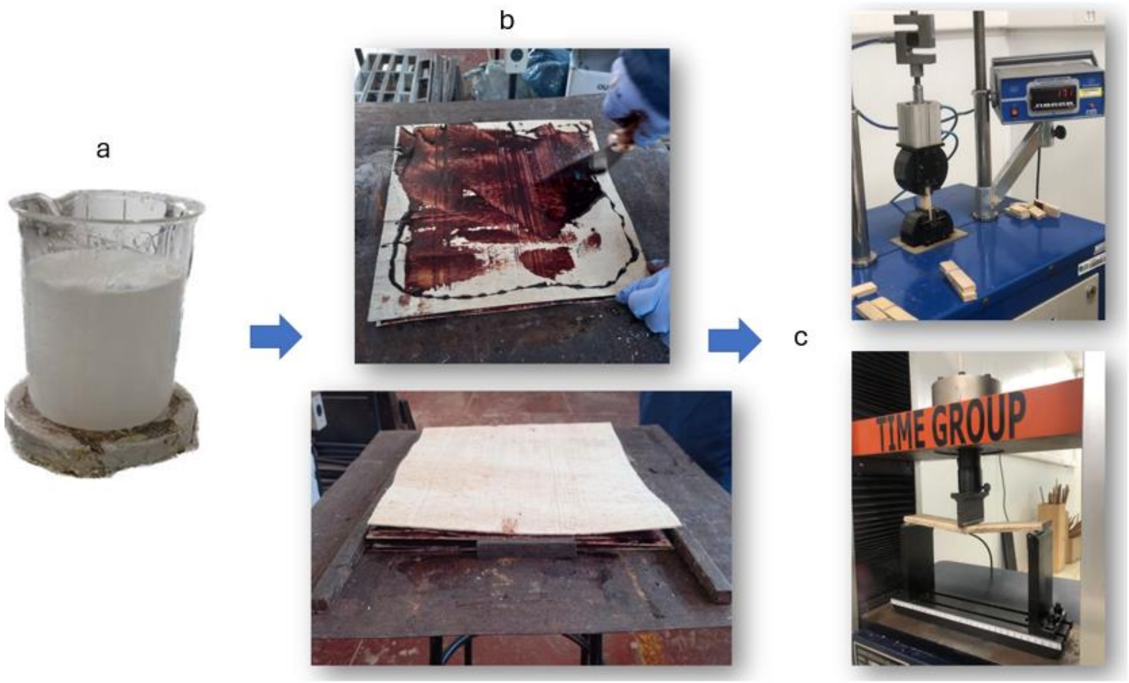

Figure 1: Production of plywood panels. a) Nanotalc mixed with Tween 80 surfactant. b) Plywood panels c) Shear strength test.

Following the recommendations of ABNT NBR 9484 (1986), after hot pressing, the plywood panels were placed in a climate control room where they remained stacked, separated by wooden boards, at a temperature around 20 ºC and relative humidity of 65 %, with the purpose of standardizing the humidity of the panels.

Afterwards, specimens were taken to perform physical and mechanical tests: specific mass according to ABNT NBR 11941 (2003), water absorption according to ABNT NBR 9486 (2011), modulus of rupture (MOR) and modulus of elasticity (MOE) to flexural strength according to ECS EN-310 (1993a) and shear strength at the glue line, in dry, wet and post-boiling conditions, according to ECS EN-314-1 (1993b).

The experimental design used to evaluate the physical-mechanical properties was the entirely randomized design. The results were evaluated by variance analysis and regression analysis, both at 5 % significance level.

Results and discussions

Physical and chemical characterization of the lignocellulosic material

The average values obtained for the basic density and chemical characterization of ocote pine (*Pinus oocarpa* Schiede ex Schltdl.) wood are shown in Table 1.

Table 1: Physical and chemical characterization of ocote pine (*Pinus oocarpa* Schiede ex Schltdl.) wood.

Material	Density (kg/m^3)	Extractives (%)	Lignin (%)	Ashes (%)	Holocellulose (%)
P. oocarpa	510±0,04	6,8±0,12	28,31±0,18	1,20±0,03	63,81 ± 0,61

The value obtained for the density of ocote pine (*Pinus oocarpa* Schiede ex Schltdl.) wood was 510 g/m^3, according to Silveira *et al*. (2013) the wood is classified as low density, since it is below 550 kg/m^3. According to the literature, the value obtained is compatible, Matos *et al.* (2019) studying the wood of ocote pine (*Pinus oocarpa* Schiede ex Schltdl.) obtained an average of 530 kg/m^3 and Pinati *et al*. (2018) obtained an average value of 470 kg/m^3. According to Iwakiri *et al*. (2011) the low density for plywood panels contributes to greater absorption of the adhesive, resulting in the reduction of the glue line thickness and strength of the adhesive bond between the veneers.

The average value obtained in this study for extractives was lower than that reported by Furtini *et al.* (2021) who obtained an average of 7,38 % for ocote pine (*Pinus oocarpa* Schiede ex Schltdl.) wood. According to Iwakiri *et al*. (2020) high levels of total extractives can block the adhesive-wood contact, causing deficiencies in panel bonding. Therefore, the value obtained in this study is acceptable.

The mean value of insoluble lignin obtained in this study, compared to the literature, proved to be higher than that obtained by Furtini *et al.* (2022) who reported an average of 6,18 % and Brito *et al*. (2021) who obtained an average of 6,34 %. The higher value is considered ideal, because the lignin contributes to the adhesion mechanisms and may contribute to the bonding process of the panels.

Regarding the ash, Santos *et al*. (2022) have found a content of 1,20 % close to the present study. However, Andrade *et al*. (2019) obtained a lower content of 0,20 %. High concentrations of this component can block reactive sites for adhesion, affecting the bonding quality and mechanical performance of the panel (Soares *et al*. 2017).

For holocellulose, Mendes *et al*. (2014) indicates that *Pinus* species present a content between 50 % to 85 %, thus the value obtained in this study corresponds to the stipulated range. According to Soares *et al.* (2017) high contents of this component can block reactive adhesion sites, affecting the bonding quality and the mechanical performance of the panel.

Physical characterization of the plywood panels

The average apparent density results of the plywood panels glued with pure phenol-formaldehyde adhesives and with the addition of nanotalc are illustrated in the figure 2. The apparent density of the panels showed no difference between the treatments studied, with average values of apparent density between 0,56 g/cm³ and 0,64 g/cm³. According to ABNT NBR 9531 (1986b) panels of 12 mm and five pine veneers, for commercialization produced in Brazil, requires that the average values are between 0,47 g/cm³ and 0,64 g/cm³, therefore all treatments had compatible results.

Pinati *et al*. (2018) working with plywood panels of *Acrocarpus, Fraxinifolius*, ocote pine (*Pinus oocarpa* Schiede ex Schltdl.) achieved values close to those obtained in this work, presenting apparent density between 0,53 g/cm³ and 0,57 g/cm³. In the study of Machado *et al*. (2018), where the authors produced plywood for outdoor use with *Pinus*, *Paricá* and *Embaúba* woods, varying the adhesives, presented values close to the study of 0,40 g/cm³ to 0,57 g/cm³ for the panels with phenol-formaldehyde adhesive and 0,38 g/cm³ to 0,53 g/cm³ for the panels with tannin-formaldehyde adhesive.

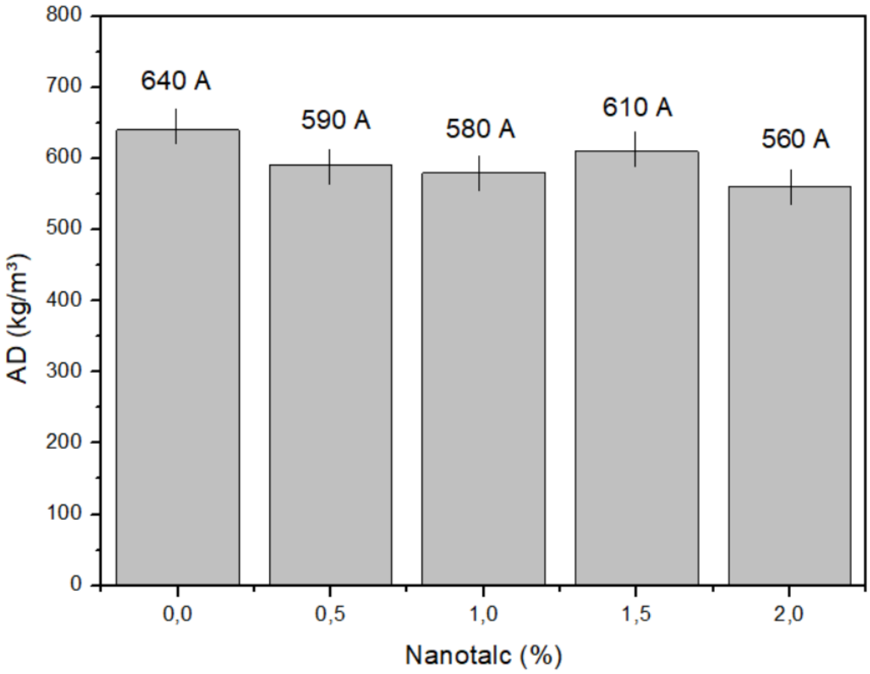

Figure 2: Average values of apparent density (AD) for the plywood panels glued with pure phenol-formaldehyde adhesives and modified with talc nanostructures. Averages followed by letters that do not differ ($p \leq 0,05$) by the Scott-Knott test.

In Candan and Akbulut (2015) work, the average density values found for plywood panels with nanoSiO_2, nanoAl_2O_3, nanoZnO nanoparticles, ranged between 0,55 g/cm³ and 0,64 g/cm³, being values close to the one found. It is worth noting, that the authors also produced plywood panels using nanoengineering, as in the present study to obtain several desired improvements in the properties of the panels.

The average values of Moisture and Water Absorption in 24 hours of the plywood panels are represented in the following Figure 3a and Figure 3b, respectively.

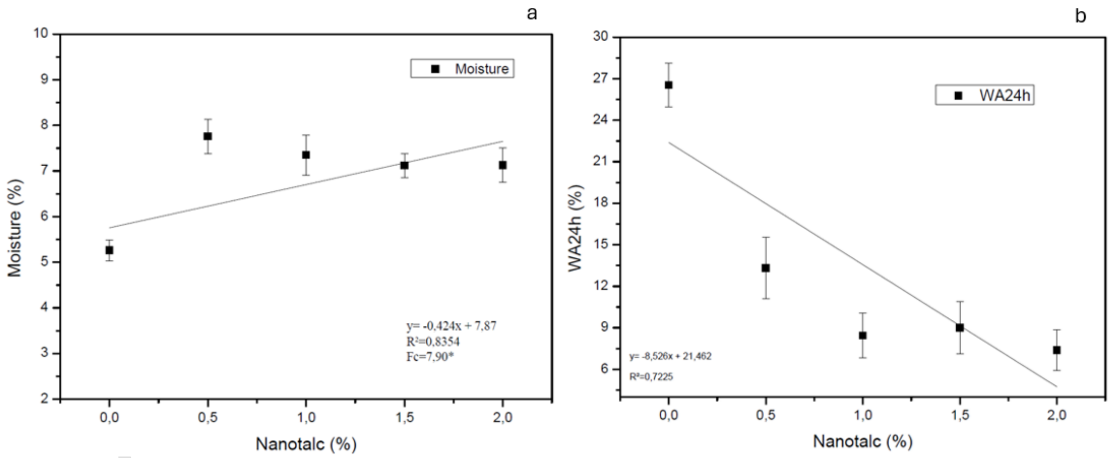

Figure 3: (a) Average values of Moisture and (b) Water absorption in 24 hours of plywood panels glued with pure phenol-formaldehyde and with different percentages of nanotalc.

By performing water absorption tests, it was observed that for each 1 % of nanotalc inserted there was a decrease of 8,52 %. As shown in Figure 3b, plywood panels produced only with phenol-formaldehyde-based adhesive showed higher water absorption rates, while those produced with adhesives containing 0,5 % to 2 % of nanotalc were those that showed the lowest averages, that is, as the insertion of nanotalc begins, the water absorption in the panels decreases, which can be explained by the natural hydrophobicity of talc.

Souza *et al*. (2020) observed absorption rates of 58,4 % for 24 h water immersion of *Pinus sp*. plywood panels with phenol-formaldehyde adhesive, results higher than those found in this study. For Reis *et al*. (2019) water absorption was 65,7 % for plywood panels produced with veneers exclusively with *Pinussp*. and 50,5 % for those with indian cedar (*Cedrus deodara* (Roxb.) G. Don), with all treatments using the phenol-formaldehyde adhesive. Therefore, the panels with nanotalc showed a large variation with this study, having water absorption rates of less than 20 %.

The average values of moisture, presents a significant and positive linear relationship. For each 1 % of nanotalc inserted there was an increase of 0,42 % for moisture. The produced panels presented moisture in accordance with the technical parameters of ABNT NBR 9531 (1986b) which suggest values below 11 %.

Iwakiri *et al*. (2020) obtained the moisture content of the panels ranging from 11,51 % with loblolly pine (*Pinus taeda* L.) presenting higher moisture values. Mendonza *et al*. (2017) have found 8,69 % for panels produced with veneers of the wood of Amescla (*Trattinnickia burserifolia*), being closer with those of the study. Mendonza *et al.* (2017) who worked with panels produced with three loblolly pine (*Pinus taeda* L.) veneers and phenol formaldehyde adhesive found a higher moisture content of 11,31 %.

Mechanical characterization of plywood panels

The mean values obtained for the shear strength of the plywood panels are presented in Figure 4. According to the Scott-knott test there was no statistical difference between the treatments.

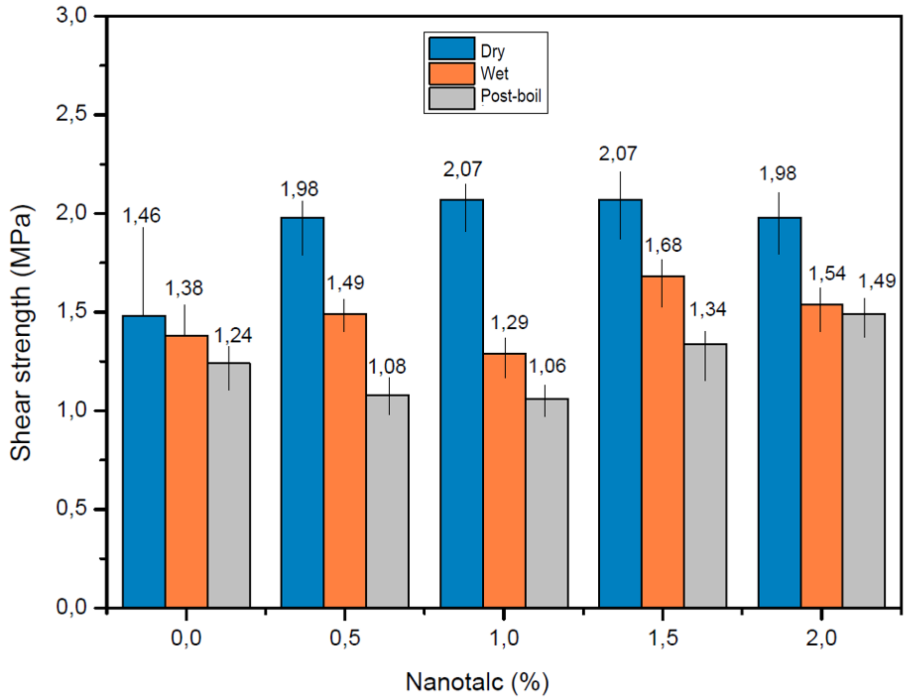

Figure 4: Mean values of shear strength in dry, wet, and post-boil conditions of plywood panels glued with pure phenol-formaldehyde and with different percentages of nanotalc.

According to the ECS-310 (1993) for plywood panels to be considered for outdoor use, they must meet the minimum value of 1 MPa. It is noted that in all conditions submitted (dry, wet, and post-boiling) the panels meet the standard. Yang *et al*. (2019) by adding 5 % nanolignin to the phenol-formaldehyde adhesive obtained an increase in shear strength from 8,7 MPa to 10,9 MPa. The small amount of nanolignin improved the copolymerization reaction between nano and phenol-formaldehyde inducing an increase in crosslinking density and better structural alignment of lignin and phenol. However, when adding 10 % of nanolignin there was no statistical difference, which should occur due to the large aggregates of lignin particles.

The treatments containing small amounts of nanotalc had higher shear bond strength, especially when evaluated in dry conditions, than the treatment that did not incorporate the nanotalc in the adhesive. This fact shows the great potential of inserting small amounts of nanotalc, to improve its resistance. Magalhaes *et al*. (2019) found lower shear strength values, ranging from 1,30 MPa to 1,70 MPa for plywood that was produced with phenol formaldehyde with partial substitution of phenolated Kraft lignin, heat treated and treated with potassium dichromate.

The following Figure 5 presents the average values for the Modulus of Rupture (MOR) and Modulus of Elasticity (MOE) for the produced plywood panels.

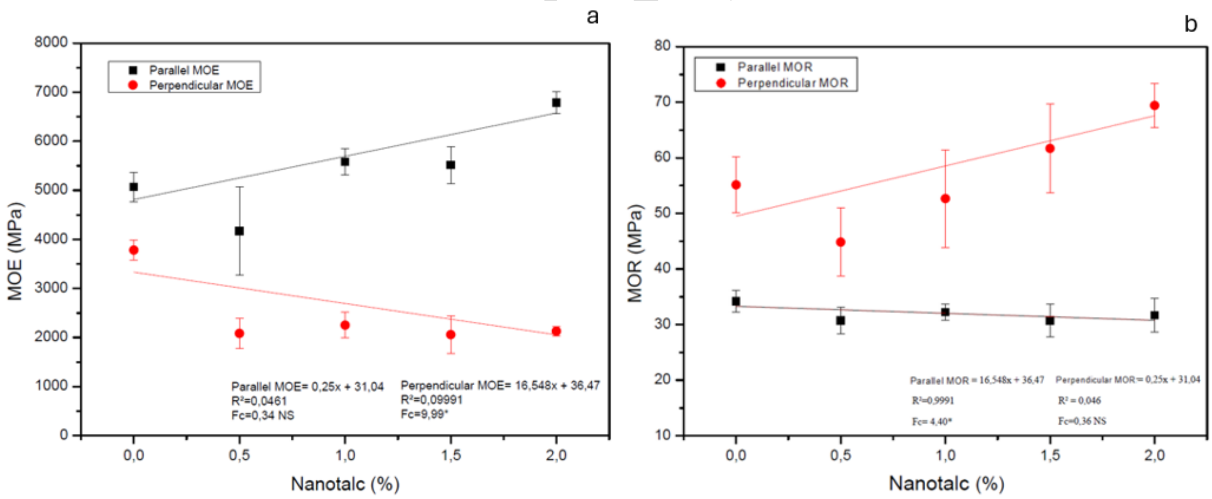

Figure 5: (a) Modulus of elasticity and (b) Rupture of plywood panels bonded with pure phenol-formaldehyde and different concentrations of nanotalc.

The values observed for the plywood panels bonded with phenol-formaldehyde adhesive nanomodified with different percentages of talc met the standards for elastic flexion ABNT NBR 9531 (1986b) with minimum required values of 1485 MPa and 5223 MPa for MOE for perpendicular and parallel 30,9 and 14,0 MPa for MOR parallel and perpendicular, respectively. Also, it is observed that for parallel MOR the results were all above 40 MPa and following this trend as the percentage of nanotalc increased, with the highest results for the concentration of 2 %.

The fiber orientation and the distribution of load on it can result in significant differences in the results of flexural strength tests. Typically, it is expected that the values of tests in the parallel direction are higher than those in the perpendicular directions due to the strength of the fibers in that orientation (Auriga *et al*. 2020). However, the results presented in Figure 5 do not follow this expected trend, which can be explained by the anisotropy of the wood material. Variations in density, presence of defects, and non-uniformity of moisture along the plywood can account for the lower values of flexural strength in the parallel direction compared to the perpendicular direction (Lengowski *et al*. 2021).

Reis *et al*. (2019) produced plywood panels with 28-year-old ocote pine (*Pinus oocarpa* Schiede ex Schltdl.) veneers, glued with phenol-formaldehyde adhesive where they showed values for parallel and perpendicular MOR of 56,8 MPa and 31,5 MPa, respectively. Similar results were found in this study for perpendicular MOR values, with an average of 30 MPa, even with the addition of nanotalc to the adhesive. As for the parallel MOR values, plywood bonded with higher percentages of nanotalc showed values up to 70 MPa, where the incorporation of nanotalc may have increased the strength property in the modulus of rupture.

Comparing with some natural adhesives that received nanostructures in their synthesis, Carvalho *et al*. (2016) compared phenol and urea-formaldehyde adhesives with tannin-based adhesives from barbatimão (*Stryphnodendron adstringens* (Mart.) Coville) and of black wattle (*Acacia mangium* Willd.), obtaining the higher MOR and MOE values for the plywood bonded with phenol-formaldehyde: 56,70 and 5350 MPa for perpendicular

MOR and MOE and 38,54 and 2660 MPa for parallel MOR and MOE, similar to the present study.

Authors such as Magalhaes *et al*. (2019) showed that their MOE and MOR values were not affected by the addition of lignin in the phenol-formaldehyde adhesive formulation and Souza *et al*. (2020) who produced plywood with tannin-based adhesives from black wattle (*Acacia mangium* Willd.) and phenol-formaldehyde, where MOE and MOR there was no statistical difference between the tannin-based adhesives from black wattle (*Acacia mangium* Willd.) and the phenol-formaldehyde formulation. Results that diverge from the study, where the addition of talc nanostructures in the phenol-formaldehyde adhesive formulation influenced its mechanical properties.

Conclusions

The current study presents the talc in nanoscale as an alternative in the optimization of plywood panels produced with the conventional synthetic adhesive phenol-formaldehyde. The quality of the adhesive produced was demonstrated by its resistance to water absorption with the insertion of up to 2 % talc to the adhesive, reducing it considerably. Moreover, from 1 % talc inserted, the plywood panels had their mechanical characteristics superior to those glued with pure phenol-formaldehyde adhesive.

However, as the study is innovative in nature, further research should focus on the application and quality evaluation of other talc nanomodified adhesives on different types of wood panels.

Authorship contributions

Y. B. C. L.: Conceptualization, methodology, data curation, writing - original draft. C. A. S.: Conceptualization, writing - proofreading and editing. A. C. C. F.: Conceptualization, writing - proofreading and editing. J. B. G. J.: Orientation, methodology, writing - review and editing. L. M. M.: Supervision, writing - proofreading and editing.

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