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Effect of acetylation on technological characteristics of *Jacaranda copaia* wood: Part 1 – Physical and mechanical properties

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ABSTRACT: The objective of this work was to evaluate the effect of acetylation on the technological characteristics of *Jacaranda copaia* (Aubl.) D. Don. wood. Physical (apparent and basic densities, water absorption, and thickness swelling) and mechanical (Rockwell hardness) properties were evaluated. Wood acetylation was carried out by immersion of samples in acetic anhydride; five treatments were evaluated: control (no acetylation), acetylation for 2 hours, acetylation for 4 hours, acetylation for 6 hours, and acetylation for 8 hours. All reactions were carried out at a constant temperature of 90 °C. The results show that the 8-hour acetylation treatment presented the highest weight gain (21.62%). This treatment also presented the lowest water absorption (119.55%) and the highest Rockwell hardness (24.78 HRL). In general, the acetylation treatment resulted in interesting improvements in wood's physical and mechanical properties.
Keywords: chemical modification; dimensional stability; hardness.

Efeito da acetilação nas características tecnológicas da madeira de *Jacaranda copaia*: Parte 1 – propriedades físicas e mecânicas

RESUMO: O trabalho teve como objetivo avaliar a eficiência da acetilação para madeira de *Jacaranda copaia* (Aubl.) D. Don. em suas características tecnológicas. Para isso, foram avaliadas propriedades físicas (densidade aparente e básica, absorção em água e inchamento em espessura) e a mecânica (dureza Rockwell). A acetilação da madeira foi realizada mediante imersão de amostras em anidrido acético, sendo avaliados cinco tratamentos: controle (não acetiladas), acetilação por 2 h, acetilação por 4 h, acetilação por 6 h e acetilação por 8 h. Todas as reações realizadas com a temperatura constante de 90 °C. Os resultados mostram que o maior ganho de massa foi apresentado pelo tratamento de acetilação durante 8 horas, como o valor de 21,62%. O tratamento também apresentou o menor valor de absorção de água, sendo 119,55%, e o maior valor para dureza Rockwell, sendo 24,78 HRL. De modo geral, o tratamento de acetilação promoveu melhorias interessantes nas propriedades físicas e mecânicas da madeira.
Palavras-chave: modificação química; estabilidade dimensional; dureza.

1. INTRODUCTION

Wood is a natural material from renewable sources which has a high demand for applications in construction due to its mechanical properties (BECK et al., 2018). According to Digaitis et al. (2021), wood has been considered worldwide as one of the most important materials for sustainable transition.

According to Makela et al. (2021), wood structures consist of cellulose fibrils in a matrix of lignin and hemicellulose, which together result in a high strength-to-weight ratio, but form a material with vulnerability to hygroscopicity and dimensional instability.

Wood performance for some applications is directly related to the presence of water, as it results in negative changes to wood's physical properties (dimensional instability) and directly affects mechanical strength properties and durability of the material (DIGAITIS et al., 2021). Beck et al. (2018) reported that one of the main disadvantages of wood is its high susceptibility to biological degradation, which should be prevented.

The acetylation process has been studied to minimize negative effects that result in the fragilization of wood's physical and mechanical properties. According to Quintana et al. (2018), this process is a chemical change in which acetyl (CH_3CO-) groups react with hydroxyl (OH) groups present in the cellulose surface, thus resulting in a less hydrophilic surface. Wojeicchowski et al. (2018) reported that hydroxyl groups present different reactivity levels and, during the acetylation process, there are reactions of addition and removal of these groups, which are converted into acetyl.

According to Marsich et al. (2018), acetylation treatments improved the wood capacity to support higher loads than non-modified wood, as the effect of acetylation increases mechanical strength and decreases water absorption.

Acetylation also results in wood dimensional stability. This stability is a result of the grouping of acetate linked to hydroxyls of polymers of the cell wall. The cell wall volume is close to the original fresh matter volume; thus, a little swelling occurs when water enters the wood (ROWELL, 2006).

According to Van Der Lugt et al. (2016), chemical treatments with acetylation at as industrial scale have been carried out in some European countries and acetylated wood is marketed mainly for the manufacturing of products for external use, such as doors, windows, coatings, floors, and light structures. Despite the existence of products based on acetylated wood, and the fact that the acetylation process has been used by industries for decades, the distributions of acetyl groups in the wood are still little understood (MAKELA et al., 2021). In Brazil, the wood acetylation process is under research, but many species were still not studied (FIGUEIREDO et al., 2019).

Jacaranda copaia (Aubl.) D. Don is among wood species that are native, but not endemic, to Brazil. The geographical distribution of this species in Brazil includes several states: Acre, Amazonas, Amapá, Pará, Rondônia, and Roraima in the North, Maranhão in the Northeast, and Mato Grosso in the Central-West region (FARIAS-SINGER, 2020).

J. copaia wood presents specific properties, including low density, dimensional instability, little mechanical strength, easy water exchange with the external medium, low resistance to deterioration, and low market cost (ELEOTÉRIO; SILVA, 2014). Thus, this species has been used for purposes that do not require high technological properties, such as the production of boxes and toys and the manufacturing of ferries. In this context, the objective of this work was to evaluate the physical and mechanical properties of *J. copaia* wood after acetylation, and assess whether this process is efficient and results in improvements in the material properties.

2. MATERIAL AND METHODS

2.1. Sample collection and preparation

Jacaranda copaia wood was obtained from timber boards stored by the Laboratory of Wood Technology (LTM) of the Institute of Agricultural Defense of the State of Mato Grosso (INDEA), in Cuiabá, MT, Brazil.

One-hundred samples with nominal dimensions of 2.5 × 2.5 × 1.0 cm (width × length × thickness) were used, all free from pronounced defects such as cracks and knots.

The samples were placed in a forced air circulation oven at a temperature of 60±2 °C until they presented anhydrous weight and volume. The weight was determined using an analytical balance, and the volume was determined using a digital caliper, at the end of the drying. The samples were then divided into five groups (treatments) with 20 samples: four groups with samples to be subjected to acetylation and one control group (non-acetylated samples).

2.2. Acetylation

The acetylation treatment was applied using the adapted methodology of Gomes et al. (2006). The wood was subjected to acetylation treatments four different times (2, 4, 6, and 8 hours) under constant temperature (90±2 °C). The wood samples were immersed in four glass bottles containing 1000 mL of acetic anhydride, and the material was maintained warm in a water bath.

The samples were then withdrawn from the immersion and washed with water for removing most of the reagent and subjected to drying in a forced air circulation oven at 60±2 °C. The samples were then weighed in an analytical balance and measured for width, length, and thickness using a digital caliper, thus obtaining the anhydrous volumes.

The weight gain of the acetylated wood was determined using the ratio between the dry weights before and after the treatments (Equation 1):

$$WG = \left(\frac{Wt - Wn}{Wnt}\right) * 100 \qquad (01)$$

where: WG = weight gain (%); Wt = treated wood dry weight (g); Wnt = non-treated wood dry weight (g).

2.3. Physical properties

The tests of physical properties followed the procedures established by the Brazilian Association of Technical Standards (NBR 7190) (ABNT, 1997). Twelve samples of each treatment were immersed in containers with distilled water at 30±2 °C, and their weights and volumes were determined after the following times: 2, 24, and 720 hours (total saturation).

Water absorption and volumetric swelling were obtained using Equations 2 and 3, respectively:

$$ABS = \left(\frac{Ww - Wd}{Wd}\right) * 100 \qquad (02)$$

$$\mathrm{VS} = \left(\frac{Vw - Vd}{Vd}\right) * 100 \qquad (03)$$

where: ABS = water absorption (%); Ww = wet weight (g); Wd = dry weight (g); VS = volumetric swelling (%); Vw = wet volume (cm³); Vd = dry volume (cm³).

In addition, basic and apparent anhydrous densities were determined, using the weight-to-volume ratio, according to Equations 4 and 5:

$$pb = \left(\frac{Wd}{Vsat}\right) \qquad (04)$$

$$pa = \left(\frac{Wd}{Vd}\right) \qquad (05)$$

where: pb= basic density (g cm^{-3}); Wd = dry weight (g); Vsat = saturated volume after 30 days of immersion in water (cm³); pa = apparent density (g cm^{-3}); Vd = dry volume (cm³).

2.4. Mechanical properties

The mechanical strength was evaluated using the Rockwell hardness test (L scale), according to the adapted methodology of Stangerlin et al. (2013). The tests were carried out in an analog bench hardness tester with a spherical penetrator of 0.25 inches. The load was applied to the transversal section (2.5 × 2.5 cm) in two different phases. In the first, a 98.1 N pre-load was applied; and in the second phase, a 588,4 N load was applied. The results were collected directly from the hardness tester analog gauge. Three measurements of Rockwell hardness were carried out for each sample, totaling 8 samples for each treatment.

2.5. Statistical analysis

The results obtained for weight gain, water absorption, volumetric swelling, and basic and apparent densities were organized in spreadsheets, analyzed, and then applied to a completely randomized design with 12 replications for each one of the five treatments. The results of Rockwell hardness were organized in spreadsheets, analyzed, and then applied to a completely randomized design with 8 replications for each one of the five treatments. The data were subjected to analysis of variance (ANOVA) and the means were compared by the Tukey's test at a 5% significance level.

3. RESULTS

3.1. Weight gain and density

The results obtained for the weight gain tests showed that all treatments presented increases in weight due to the acetylation process. The treatment with acetylation for 8 hours presented the highest weight gain, 21.62%, which did not statistically differ from that of the treatment with acetylation for 6 hours (Figure 1). The treatment with acetylation for 2 hours presented the lowest gain weight, 18.06%, which did not statistically differ from that of the treatment for 4 hours (Figure 1). The results showed that treatments with shorter acetylation times (2 and 4 hours) presented lower weight gains than treatments with longer times (6 and 8 hours). These differences denote that the time for the acetylation directly affects the weight gain percentages.

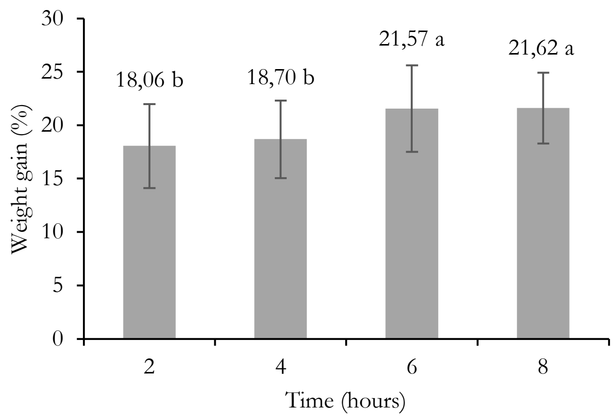

Figure 1. Weight gain percentages of *Jacaranda copaia* wood after different acetylation treatments.
Figura 1. Ganho de massa percentual da madeira de *Jacaranda copaia* após os diferentes tratamentos de acetilação.

The results showed that the treatment with acetylation for 6 hours presented the highest basic density, 0.429 g cm^{-1} (Table 1), which did not statistically differ from that of the treatment for 4 hours. The control treatment presented the lowest basic density, 0.347 g cm^{-1} (Table 1). The analyses of the results showed that the acetylation process resulted in an increase of approximately 23% in basic density when compared to the control treatment. This increase may be due to the weight gain presented by the wood.

The treatment with acetylation for 6 hours presented the highest apparent density, 0.455 g cm^{-3} (Table 1), and the control treatment presented the lowest, 0.406 g cm^{-3} (Table 1). The analyses of the results showed that the acetylation process resulted in an increase of approximately 12% in apparent density when compared to the control treatment. This increase may be due to the weight gain presented by the wood.

Table 1. Mean basic density (ρ_b) and an apparent density (ρ_a) of *Jacaranda copaia* wood subjected to acetylation.
Tabela 1. Valores médios de densidade básica (ρb) e densidade aparente (ρa) das madeiras de *Jacaranda copaia* submetidas à acetilação.

Treatments	ρ_b (g cm^{-3})	ρ_a (g cm^{-3})
Control	0.347 b	0.406 b
2 hours	0.416 ab	0.445 ab
4 hours	0.420 a	0.447 ab
6 hours	0.429 a	0.455 a
8 hours	0.415 ab	0.446 ab

Means followed by the same letters in the columns are not different from each other by the Tukey's test at 5% significance level.

3.2. Water absorption and thickness swelling

The results of the water absorption tests showed that the acetylation process resulted in decreases in wood hydrophilicity in all acetylated treatments (Table 2). No significant statistical differences were found between treatments with 2 and 24 hours of absorption. However, after reaching stability after 720 hours, the acetylation treatment for 8 hours presented better performance, with water absorption of 119.55%; and the control treatment presented the worse performance, 220.17%.

The thickness swelling caused by the treatments showed that the acetylation contributed positively to preventing wood swelling, after water absorption. Treatments with acetylation presented the lowest thickness swelling for all immersion times and presented no statistically significant differences from each other. Nonetheless, the treatment with acetylation for 8 hours presented the lowest swelling values at the stability time (720 hours): 7.13%. The control treatment presented the highest swelling values at the stability time (720 hours): 20.50% (Table 3). The analysis of the results showed that the decrease in hydrophilicity caused by the acetylation treatments also improved the wood's dimensional stability.

Table 2. Mean water absorption of samples of *Jacaranda copaia* wood subjected to acetylation.
Tabela 2. Valores médios de absorção de água das amostras de madeira de *Jacaranda copaia* submetidas à acetilação.

Treatments	Immersion time		
	2 hours (%)	24 hours (%)	720 hours (%)
Control	98.25 a	138.80 a	220.17 a
2 hours	61.56 b	88.87 b	159.04 b
4 hours	59.63 b	83.39 b	158.50 b
6 hours	53.22 b	75.90 b	149.34 b
8 hours	61.10 b	76.04 b	119.55 c

Means followed by the same letters in the columns are not different from each other by the Tukey's test at 5% significance level.

Table 3. Mean volumetrically swelling of *Jacaranda copaia* wood subjected to acetylation.
Tabela 3. Valores médios de inchamento volumétrico das madeiras de *Jacaranda copaia* submetidas à acetilação.

Treatments	Immersion time		
	2 hours (%)	24 hours (%)	720 hours (%)
Control	12.02 a	16.74 a	20.50 a
2 hours	4.42 b	6.80 b	8.26 b
4 hours	4.11 b	6.70 b	8.82 b
6 hours	3.43 b	6.08 b	7.34 b
8 hours	3.79 b	5.84 b	7.13 b

Means followed by the same letters in the columns are not different from each other by the Tukey's test at a 5% significance level.

3.3. Hardness Rockwell

The results of the Rockwell hardness test showed that the acetylation process increased the hardness of all treated samples in all evaluation times, and presented no statistically significant differences from each other. Despite the non-significant differences, the treatment with acetylation for 8 hours presented the highest hardness (24.78 HRL) and the control treatment presented the lowest hardness (5.05 HRL) (Table 4). Considering these results, hardness increases as the time of the acetylation process is increased.

Table 4. Mean Rockwell hardness of samples of *Jacaranda copaia* wood subjected to acetylation.
Tabela 4. Valores médios de dureza Rockwell L das amostras da madeira de *Jacaranda copaia* submetidas à acetilação.

Treatments	Rockwell hardness (HRL)
Control	5.05 b
2 hours	21.65 a
4 hours	22.61 a
6 hours	23.42 a
8 hours	24.78 a

Means followed by the same letters in the columns are not different from each other by the Tukey's test at 5% significance level.

4. DISCUSSION

4.1. Physical properties

The weight gain of wood samples of *Jacaranda copaia* (Aubl.) D. Don. after the acetylation treatment is probably due to the reaction of hemicellulose to acetylation. Brum et al. (2012) reported that one or more hydroxyl groups of the wood chain are exchanged by acetyl groups during the material acetylation process, thus, an increase in the material weight is expected. According to Elrhayam and Elharfi (2019), the chemical composition of hydroxyl groups present in the wood is strongly dependent on electronic chemical parameters, which makes it very important to the material weight gain.

According to Brum et al. (2012), during the material acetylation process, the higher the weight gain, the higher the acetylation rate. Hunt et al. (2018) evaluated acetylation for *Pinus taeda* and reported that the weight gain occurs because the atomic weight of the hydroxyl group (17u) is lower than that of the acetate group (59u), which substitutes it during the chemical changes.

Ajdinaj et al. (2013) reported that acetyl molecules easily penetrate low-density wood, making it possible to block a greater number of free hydroxyl groups. Thus, the low density of *J. copaia* wood may explain the expressive weight gain after acetylation.

The increases in basic and apparent densities in the treated *J. copaia* wood samples, compared to the control treatment, may be due to the weight gain caused by the acetylation process. According to Olaniran et al. (2019), weight change is a factor that directly reflects changes in the density of wood samples.

The results of the basic density of *J. copaia* wood with no acetylation treatment was very close to 0.340 g cm^{-3}, which was obtained by Eleotério and Silva (2014) for the same species with no treatment. The analysis and comparison between the results showed that the acetylation treatment indeed resulted in increases in basic density for the evaluated wood.

The decreases in physical properties (water absorption and thickness swelling) found for the *J. copaia* wood samples after the acetylation treatments are due to the replacement of hydroxyl (OH) groups by acetyl groups, which results in a lower sensitivity of wood to water absorption and, thus, tends to improve the mechanical properties, durability, dimensional stability, and water absorption stability (NÉMETH et al., 2020).

Castro; Iwakiri (2014) reported that hydrophile groups present in the wood are blocked during the acetylation process, resulting in a lower affinity between the water and the acetylated wood. It generates a decrease in the water absorption capacity of the material and, consequently, an increase in the wood's dimensional stability (CASTRO; IWAKIRI, 2014).

Cermák et al. (2022) evaluated acetylation treatments in *Fagus sylvatica* L. wood and found that the acetylated wood presented an improvement of 50% in characteristics related to the wood-water interaction. They also reported that the acetylation contributed to decreases in the hysteresis effect between adsorption and desorption curves and in the wood anisotropy.

The efficiency of acetylation treatments in decreasing water absorption was also found by Lopes et al. (2011), who reported decreases between 54% and 72% in water absorption rate for fibers of acetylated *Neoglasiovia variegata* wood. Regarding the thickness swelling, Ajdinaj et al. (2013) found that the acetylation treatment resulted in a 61% decrease in swelling for the wood species *Populus alba* L., and 53% for the species *Fagus sylvatica* L.

According to Esteves et al. (2011), decreases in volumetric swelling improve the wood dimensional stability and, thus, reduce the formation of cracks and result in a material with better performance. It also increases the durability of the modified wood (ESTEVES et al., 2011).

Teaca and Tanasa (2020) reported that the wood chemically modified by acetylation tends to present a lower affinity to water when compared to non-modified wood. Bi et al. (2021) reported that costs related to wood acetylation treatments are decreasing and the material performance is improving, which has made wood processing more industrialized.

4.2. Mechanical properties

According to Scharf et al. (2022), hardness is a measure of the resistance of a material to penetration by another material of higher hardness and is very used for evaluating the performance of wood products, mainly for analyzing the efficiency of the densification process.

The results of the Rockwell hardness (L scale) showed increases for all treatments with acetylation. According to Figueiredo et al. (2019), increases in hardness after acetylation treatments can be explained by the increase in weight during the process.

Bongers; Beckers (2003) evaluated acetylation treatments without catalyzer in *Pinus sylvestris* L. end *Populus* sp. wood and found that the acetylated wood exhibited improvements in elasticity and rupture modules, resistance to shear, parallel compression to the grain, and Janka hardness. The application of reactive or non-reactive chemical products to wood cell walls/lumen may result in increases in hardness and in compression force properties due to increases in wood density (XIE et al., 2013).

Sydor et al. (2022) evaluated the wood species *Alnus glutinosa* (L.) Gaertn, *Tilia europaea* L, *Betula alba* L, *Fraxinus excelsior* L, *Milicia excelsa* (Welw.) CC Berg, and *Fagus sylvatica*

L. and found that the higher the wood density, the higher the Brindel hardness. They also found that the harder the wood, the lower the total penetration depth during the tests. These factors confirmed that density is essential for predicting and evaluating wood strength properties.

5. CONCLUSIONS

The physical properties of acetylated wood improved with the acetylation process, with an increase in densities and reductions in water absorption and volumetric swelling during immersion for 2h, 24h, and until full saturation in water. Regarding Rockwell hardness, acetylation provided an increase in surface penetration resistance of acetylated wood.

The 6 and 8h treatments showed the best results for *Jacaranda copaia* wood since chemical modification occurred in its structures, 21% mass gain, and improvement of physical and mechanical properties.

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