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Effect of acetylation on technological characteristics of *Jacaranda copaia* wood: Part 2 – Chemical and colorimetric changes

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ABSTRACT: Acetylation is a chemical change to improve wood properties through a chemical reaction that substitutes hydroxyl with acetyl groups. Thus, the objective of this work was to assess the efficiency of acetylation for the improvement of *Jacaranda copaia* wood properties. Chemical characterization, infrared spectroscopy, and colorimetry tests were carried out. The infrared spectra were qualitatively analyzed for chemical changes caused by acetylation. Changes in wood color parameters (L*, a*, b*, C, and h*) were evaluated. The wood acetylation was carried out through immersion of samples in anhydride acetic; 5 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 under a constant temperature of 90±2 °C. The results showed the occurrence of chemical changes in structural components of the acetylated wood by increases in 1735, 1375, and 1250 cm bands referring to the addition of acetate groups. Regarding colorimetry, darkening of wood was found for all acetylation treatments.
Keywords: Amazon wood; surface properties; color; FTIR.

Efeito da acetilação nas propriedades tecnológicas da madeira de *Jacaranda copaia*: Parte 2 – alterações químicas e colorimétricas

RESUMO: A acetilação é uma modificação química que visa a melhoria das propriedades da madeira a partir de uma reação química que substitui um grupo hidroxila por um grupo acetil. Assim, este trabalho teve como objetivo comprovar a eficiência da acetilação na melhoria das propriedades da madeira de *Jacaranda copaia*. Para isso, foram realizados a caracterização química, testes de espectroscopia na região do infravermelho e testes de colorimetria. Buscou-se por meio dos espectros de infravermelho uma análise qualitativa das modificações químicas proporcionadas pela acetilação. Adicionalmente, foram avaliadas as alterações proporcionadas na coloração da madeira (parâmetros L*, a*, b*, C e h*). A acetilação da madeira foi realizada mediante imersão de amostras em anidrido acético, sendo avaliados 5 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 ± 2°C. Os resultados evidenciaram que ocorreu uma modificação química nos componentes estruturais da madeira acetilada, por meio do aumento nas bandas 1735, 1375 e 1250 cm^{-1} referentes a adição de grupos acetatos. Em relação à colorimetria verificou-se o escurecimento da madeira acetilada em todos os tratamentos.
Palavras-chave: Madeiras da Amazônia; propriedades de superfície; cor; FTIR.

1. INTRODUCTION

According to Cermák et al. (2022), wood is an important natural material extracted from renewable sources which presents many advantages, such as mechanical strength properties and rigidity, considering its weight, is a source of energy, and has esthetical characteristics. However, it also presents characteristics that affect its properties, including a strong tendency for water absorption and desorption, which results in a high dimensional instability and susceptibility to wood-decay fungi (CERMÁK et al., 2022).

According to Jones; Sandberg (2020), chemical, thermal, and impregnation processes carried out for wood changes are novelty methods for enhancing wood's physical, mechanical, and esthetical properties for the development of products from sawn timber, fiberboards, and composite strengthened wood. Another important advantage is the decrease of environmental impacts caused by the discarding of these materials at the end of their useful life, which are lower than those of non-modified wood (JONES; SANDBERG, 2020).

The changing of wood chemical properties through acetylation with anhydride acetic is worldwide known as one of the most promising methods for improving wood properties, considering technical and economic aspects. Despite this method being used by industries in some

countries, in Brazil, it is still experimental and an object of scientific study (GOMES et al., 2006; CASTRO et al., 2013; CASTRO; IWAKIRI, 2014; FIGUEIREDO et al., 2019).

Bi et al (2021) reported that manufactured products based on wood treated with acetylation still require better physical and mechanical properties and lower production costs. Nonetheless, advances in the development of new acetylation techniques for wood modification have been promising, and it is expected that these techniques provide modified materials with better performance, are easy processing methods, and require less raw material (BI et al., 2021).

According to Sandak et al. (2021), the wood chemical change process is a result of the interaction between chemical reagents and wood constituents, which are covalently linked. Chung et al. (2018) reported that the chemical change process by acetylation is known as the result of the replacement of hydroxyl groups present in wood fibers to decrease the hydrophilicity of the material.

According to Rowell (2020), in the acetylation treatment, hydroxyl groups present higher availability in polymers in the wood cell wall. During the acetylation process, a single addition reaction occurs, substituting hydroxyl with acetyl groups without the polymerization process, and the weight gain enabled by the acetyl group can be transformed into blocked hydroxyls (ROWELL, 2020).

The properties of acetylated wood depend on the acetylation method used and are directly related to other factors, such as the temperature of the treatment, reaction time, presence or absence of catalyzer, sample water content at the treatment time, and adequate distribution of the reagent in regions accessible by water in the cell wall (ZHANG et al., 2015; MANTANIS, 2017).

Jacaranda copaia is among wood species with potential for acetylation treatment; it presents a basic specific weight of 0.31 g cm^{-3}, is considered a light wood of easy workability, and is very used for carpentry purposes, fiberboards, boxes, and laminated wood (IBAMA, 2011; EMBRAPA, 2017). Nonetheless, *J. copaia* wood is dimensionally unstable and susceptible to wood-decay fungi (ELEOTÉRIO; SILVA, 2014). These characteristics hinder the use of this species in the manufacturing of high-added value products. Therefore, it requires the use of techniques that enable better applicability of this wood species to different environments and promote a better quality for the development of products.

The objective of this work was to assess the efficiency of acetylation for the improvement of technological properties of *J. copaia* wood, considering the effect of treatments with different acetylation reaction times on chemical changes of the wood cell wall and on changes in wood color.

2. MATERIAL AND METHODS

2.1. Sample collection and preparation

Jacaranda copaia (Aubl.) D. Don. 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 and twenty 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 24 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.

2.3. Chemical characterization

The wood boards were transformed into chips and, then, into sawdust, using a Wiley mill. The ground material was then subdivided into three granulometric fractions: larger than 40 mesh, between 40 and 60 mesh, and smaller than 60 mesh, as described in the NBR 14660 (ABNT, 2004).

The fraction between 40 and 60 mesh were used for gravimetrical chemical analysis, in duplicate, to determine extractive contents, lignin, and ashes, as described in the NBR 14853 (ABNT, 2010), NBR 7989 (ABNT, 2017), and NBR 13999 (ABNT, 2010). Holocellulose contents was determined by the difference between the total chemical the composition and composition of the non-carbohydrate fraction.

2.4. Fourier Transform Infrared Spectroscopy - FTIR

For specters, infrared analysis, a spectrophotometer FTIR Shimadzu Iraffinity-1 (Model: IRAffinity-1; Cat.No. 206-73500-38; Serial No. A21374902249S1; Brand: Shimadzu Corporation) was used.

To obtain the FTIR spectra of the samples, potassium bromide (KBr) tablets were used. The KBr was previously dried in an oven at a temperature of 110±2 °C for approximately 3 hours. After drying/activation, the KBr was transferred to a desiccator, where it remained until the preparation of the tablets.

The tablets were prepared as follows: control and acetylated samples (12 samples for each treatment) were ground using a Wiley mill equipped with a 60-mesh sieve, and then placed in an oven at 60±2 °C for 24 hours. A subsample of 1.0 mg of each sample was collected per treatment and mixed with 100 mg of dry KBr at the proportion of 1:100 mg. The mixture was placed in an agate mortar and ground until it becomes a thin powder, using an agate pistil.

The mixture was placed in a previously assembled stainless steel mold and pressed with a loading of 8 tons for 5 minutes, using a hydraulic press (Brand: Shimadzu Corporation; Model: SSP-10A; P/N: 200-64175; S/N: 310314902239).

After the sample tablet preparation procedure (Figure 1), the qualitative acquisition of the FTIR spectra was performed. Initially, the background procedure was performed using potassium bromide (KBr) tablet. The spectral acquisition of the samples was performed using the IRSolution software (Version: 1.50). The measurement

parameters: Measurement Mode (% Transmittance); Apodizaiton (Happ_Genzel); Number of Scans (200); Resolution (16); Range (400 to 4000 cm^{-1}); Gain (1).

Figure 1. Hydraulic press, KBr tablet with the sample, and Fourier transformed infrared spectrophotometer (FTIR).
Figura 1. Prensa hidráulica, pastilha de KBr com amostra e espectrofotômetro de infravermelho por transformada de Fourier (FTIR).

2.5. Colorimetry

Colorimetric parameters were obtained using a colorimeter with a resolution of 3 nm of diffuse illumination and a D65 illuminator composed of a xenon lamp, with an observation angle of 10° and an illumination area of 11 mm diameter (Figure 2).

Twelve samples per treatment were measured twice in the transversal section, and the mean values were calculated.

The parameters evaluated were: lightness (L*), which varies between 0 and 100, with 0 representing black and 100 representing white, which is also termed the gray axis; chromatic coordinates a* and b*, which represent the positions of color points on the green-red and blue-yellow axes, respectively, with values between 0 and 60, and positive numbers indicating red and yellow, and negative numbers indicating green and blue; color saturation (C), which represents the distance of the lightness axis: the higher the gray axis distance, the more saturated the color; and the hue angle (h*), which represents the dominance of a hue component of a color, according to the CIEL*a*b* system (Commission Internationale of L'éclairage).

Figure 2. Spectrophotocolorimeter used for the wood colorimetric characterization.
Figura 2. Espectrofotocolorímetro utilizado para caracterização colorimétrica da madeira.

Changes in wood color after acetylation were determined by the total color variation, according to the ASTM D 2244 (ASTM, 2021). The values of color variation (ΔE) were used to classify the wood into perception levels, according to Hikita et al. (2001) (Table 1).

Table 1. Classification of the wood total color variation (ΔE).
Tabela 1. Classificação da variação total da cor (ΔE) da madeira.

Color variation (ΔE)	Classification
0.0 – 0.5	Negligible
0.5 – 1.5	Slightly perceptible
1.5 – 3.0	Noticeable
3.0 – 6.0	Apparent
6.0 – 12.0	Very apparent

2.6. Statistical analysis

The results of the colorimetry were organized in spreadsheets, analyzed, and then applied to a completely randomized design with 12 replications and five treatments: control, acetylation for 2 hours (T1), acetylation for 4 hours (T2), acetylation for 6 hours (T3) and acetylation for 8 hours (T4). The results were subjected to analysis of variance (ANOVA) and the means were compared by the Tukey's test at a 5% significance level. The results obtained for the infrared spectra were analyzed qualitatively, considering the chemical attributions.

3. RESULTS

3.1. Chemical characterization

The results obtained for the chemical properties of the *Jacaranda copaia* wood presented high holocellulose contents (67.02%) and low ash and extractives concentrations. Lignin contents presented a mean percentage (29.18%) above that found for dicotyledon wood species (Table 2).

Table 2. Mean results of chemical analysis of *Jacaranda copaia* wood.
Tabela 2. Resultados médios da análise química da madeira de *Jacaranda copaia*.

Analysis	Concentration (%)
Ash	0.45
Extractives	3.35
Lignin (Klason)	29.18
Holocellulose	67.02

3.2. Infrared spectra (FTIR)

The analysis of the bands enabled to development of a theoretical referential indicating the connection attributed to each vibration observed in the treated wood samples (Table 3). The results of the infrared spectroscopy were visually analyzed, evaluating the relative intensity between bands characteristic of cellulose, hemicellulose, and lignin. The analysis was carried out by the comparison between the control and acetylated wood spectra (Figure 3).

The increase in vibration of the bands 1735, 1375, and 1250 cm^{-1} became more intense as the reaction time was increased. The band decreased when the reaction time decreased, as observed for the band 3400 cm^{-1} (Figure 3). These results indicated that a longer reaction time results in more replacement of OH groups, thus resulting in higher weight gains for the material.

3.3. Colorimetric parameters

The mean values of colorimetric parameters of the samples with and without treatment with acetylation are shown in Table 4. The analyses showed that the acetylation resulted in a statistically significant decrease in the lightness parameter (L*) for treatment T2, which presented a value of 60.40. However, this treatment T2 presented the highest values of a* and b* parameters (6.11 and 21.67), which were statistically equal to those presented by treatment T1. Treatment T4 presented the highest value of color saturation

(C), 23.41, which was statistically equal to that of the other acetylated treatments. T4 presented the highest value of hue angle (h*), 75.65; however, it was statistically equal to that of T3 (Table 4). The variations found affected the color of acetylated *J. copaia* wood, which presented significant differences when compared to the control treatment.

The lightness variation (ΔL*) was negative for all treatments; however, a more expressive darkening was found for the treatment with acetylation for 4 hours (T2): -3.510. The total color variation (ΔE) was also higher for T2: 6.227 (Table 5). These variations provide a general view of the performance of acetylation regarding the colorimetry of *J. copaia* wood. Considering the classification proposed in Table 1 and the analysis of the values presented in Table 5, the total color variation was considered apparent in treatments T1, T3, and T4, and very apparent in treatment T2.

Table 3. Attribution of infrared transmittance bands for treated wood.
Tabela 3. Atribuição de bandas de transmitância de infravermelho para madeiras tratadas.

Bands (cm^{-1})	Functional group / Vibration type / Indication	Reference
3300 – 3500	-OH / Stretching / Cellulose, hemicellulose, and lignin	Sinha and Rout (2008)
2900	C-H link / Stretching in methyl and methylene	Sinha and Rout (2008)
1736	Carbonylic link C=O/ Stretching / Hemicellulose	Takagi et al. (2015)
1645	Carbonylic link C=O/ Stretching / Lignin	Pires et al. (2012)
1510	C=C link / Stretching of aromatic ring / Lignin	Colom et al. (2003)
1460 – 1420	Methyl group / Asymmetrical deformation / Cellulose	Pires et al. (2012)
1375	C-H link / Angular deformation / Cellulose and Hemicellulose	Colom et al. (2003)
1250	Acetyl group / Stretching / Hemicellulose	Pires et al. (2012)
1160 – 1110	C-O-C link / Hemicellulose	Pires et al. (2012)
1058	C-O-C link / Stretching / Cellulose	Colom et al. (2003)

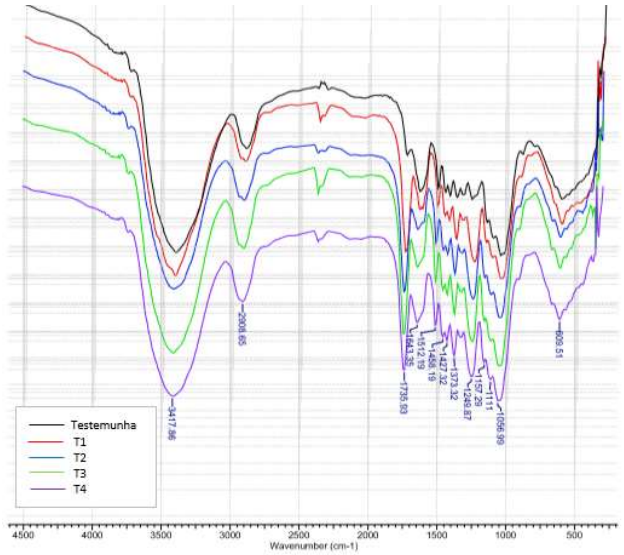

Figure 3. Infrared spectra (FTIR) of control and acetylated samples of *Jacaranda copaia* wood.
Figura 3. Espectros de infravermelho (FTIR) das amostras controle e acetiladas de *Jacaranda copaia*.

Table 4. Colorimetric parameters of samples of *Jacaranda copaia* wood before and after acetylation treatment.
Tabela 4. Parâmetros colorimétricos das amostras de *Jacaranda copaia* antes e após o tratamento de acetilação.

Treatments	Colorimetric parameters				
	L*	a*	b*	C	h*
Control	65.30 a (3.82)	4.90 b (9.14)	16.61 b (4.18)	17.32 b (4.58)	73.53 b (1.24)
T1 (2 h)	62.71 ab (3.80)	6.01 a (9.18)	21.28 a (3.39)	22.18 a (3.91)	74.30 ab (1.30)
T2 (4 h)	60.40 b (4.43)	6.11 a (9.77)	21.67 a (2.98)	22.46 a (3.41)	74.32 ab (1.41)
T3 (6 h)	63.52 ab (4.55)	5.61 ab (15.35)	21.46 a (4.25)	22.22 a (4.81)	75.38 a (2.23)
T4 (8 h)	62.42 ab (4.88)	5.51 ab (18.00)	21.44 a (3.69)	23.41 a (21.16)	75.65 a (2.59)

Means followed by the same letter in the columns are not statistically different from each other by Tukey's test ($p<0.05$). Values within parentheses are the coefficients of variation (%).

Table 5. Variations in the colorimetric parameters of *Jacaranda copaia* wood samples after acetylation treatments for different times.
Tabela 5. Variações dos parâmetros colorimétricos das amostras de *Jacaranda copaia* após diferentes períodos de acetilação.

Treatments	ΔL*	Δa*	Δb*	ΔE
T1 (2 h)	-1.740	1.025	4.653	5.082
T2 (4 h)	-3.510	1.295	4.978	6.227
T3 (6 h)	-1.492	0.603	4.665	4.935
T4 (8 h)	-1.790	0.403	4.970	5.298

4. DISCUSSION

4.1. Chemical properties

The results of the chemical characterization of the *Jacaranda copaia* wood showed high holocellulose content, low ash and extractives concentrations and a lignin content above the mean found for dicotyledon wood species. High holocellulose and lignin contents in non-treated wood can affect the material's mechanical properties. Costa et al. (2017) reported that the mechanical strength of wood to some types of loads is not associated only with thickness, but also with the amounts of their chemical components, such as cellulose, hemicellulose, lignin, and the percentages of extractives in the lumen.

Gomes et al. (2021) reported that renewable lignocellulosic materials tend to present different cellulose, hemicellulose, and lignin compositions and that these chemical properties are factors that can limit the material applicability. Braz et al. (2014) reported that cellulose and lignin are chemical constituents that affect the fragility of the wood and its byproducts, as they are directly connected to mechanical properties.

Considering the percentages of chemical constituents of the *J. copaia* wood, the acetylation treatment may improve the mechanical properties.

4.2. Changes in infrared spectra (FTIR)

The analyses of the infrared spectra (FTIR) (Figure 3) showed that control samples presented an absorption band in the region of 3400 cm^{-1}, characterizing a stretching of the hydroxyl group present in the cellulose, hemicellulose, and lignin. The greater changes in vibrations occurred in bands

related to hemicellulose (1735 cm^{-1}; C=O), stretching of hemicellulose (1375 cm^{-1}; C-H), and angular deformations of cellulose and hemicellulose (1250 cm^{-1}; acetyl group; stretching of hemicellulose). These results show a higher efficiency of acetylation in this chemical constituent. Rowell (2006) explains that the reaction of hydroxyl groups on wood cell wall constituents affects mainly hemicelluloses and lignin since they are more reactive sites in the wood.

Variations in spectra intensities may represent changes in amounts of wood chemical compounds, which are connected to the formation and changes of chemical compounds and changes in the energy of the connection between atoms of these components (YILGOR et al., 2013; ZHANG et al., 2015).

These were similar results to those obtained by Chauhan et al. (2001), who evaluated acetylated *Hevea brasiliensis* wood through infrared spectroscopy (FTIR) and found increases in absorption for wavelengths of 1740 and 1220 cm^{-1} and decreases in the band between 3000 and 3600 cm^{-1}. They evidenced the chemical reaction between anhydride acetic and hydroxyl groups of wood cell wall polymers and attributed these changes to the replacement of hydroxyl groups with acetate groups.

4.3. Colorimetric changes

The results found for colorimetric parameters (L*, a*, b*, C and h*) and color variations (ΔL*, Δa*, Δb* and ΔE) showed that the acetylation process carried out for 4 hours promoted greater changes in the color of *J. copaia* wood.

The red pigmentation (coordinate a*) affected the wood color composition. Chemically, extractives are chromophore compounds responsible for some properties, such as color and smell. According to Lima et al. (2013), low values of the green-red axis (a*) may indicate a low percentage of extractives in the wood.

The coordinate b*, which evaluates the yellow matrix of the wood, is the main responsible for the formation of the color of *J. copaia* wood. All treatments with acetylation were significantly different from the control treatment. Acetylated *J. copaia* wood became more yellowish as the coordinate b* increased. According to Mesquita et al. (2020), the higher the value of the parameter b*, the higher the participation of the yellow color.

The chromaticity (C) showed increases in values responsible for the wood's total color saturation. This variable represents the deviation from the point corresponding to the lightness axis; the farther the axis, the more saturated the color. According to Grey (2006), highly saturated color is purer and more vibrant, whereas a less saturated color is weaker and less pure.

After the acetylation process, the *J. copaia* wood samples presented significant darkening and color variations that resulted in changes in their classification, from apparent to very apparent.

Castro (2013) evaluated acetylation treatments for wood particles of *Pinus taeda* L. and found wood darkening after the process. Dong et al. (2016) found similar results when evaluating acetylated *Populus tomentosa* Carr. and *Pinus massoniana* Lamb. wood.

In addition, Fodor et al. (2017) evaluated acetylation treatments for *Carpinus betulus* L. wood under industrial conditions, by the Accoya method, and found similar results to those found in the present study, such as wood darkening and increases in red pigmentation on the surface of the treated wood.

5. CONCLUSIONS

The chemical characterization of *Jacaranda copaia* wood (control) showed a higher percentage of holocellulose and lignin in its composition.

The infrared spectroscopy showed the efficiency of the acetylation treatment. Considering the spectra (FTIR) observed, there were changes in the vibrations of bands, denoting the presence of acetate groups formed in the acetylation process.

Regarding the colorimetry, the acetylated wood presented darkening in all treatments. The colorimetric parameters showed the occurrence of changes in the acetylated treatments when compared to the control treatment.

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