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Articles

Physicochemical properties of *Paulownia tomentosa* Steud. wood

Propriedades físico-químicas da madeira de *Paulownia tomentosa* Steud.

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ABSTRACT

The wood of *Paulownia tomentosa* Steud. has the potential to develop products with high-added value. The wood is lightweight and soft, with excellent machining and finishing properties, and is therefore used to produce composite materials, wood panels, furniture, paper, and musical instruments. Thus, the objective of this study was to evaluate the physical and chemical characteristics of *Paulownia tomentosa* wood in order to recommend its use for the production and generation of bioproducts. Five trees, thirteen years old, were harvested to obtain discs at different heights, which were then processed into wedges and sawdust. The methodology described in standard NBR 11941 was employed to determine the basic density. The total extractive, lignin, and holocellulose levels were determined according to the standards of the Technical Association of the Pulp and Paper Industry. Infrared spectrometry, immediate chemistry analysis (volatile materials, fixed carbon content, and ash content), and thermogravimetric analysis of the wood were also performed. The results obtained for *Paulownia tomentosa* wood were as follows: a basic density of 0.269 g/cm³, 17.3% of total extractives, 17.1% of Klason lignin, an S/G ratio of 1.15, and holocellulose content of 67.7%. The immediate chemical analysis of the wood resulted in 86.5% of volatile matter, 12.9% of fixed carbon, and 0.6% of ash content. The low basic density of *Paulownia tomentosa* wood and the low lignin and fixed carbon content are unfavorable characteristics from an energetic point of view. Conversely, due to the high holocellulose and low lignin content, *Paulownia tomentosa* wood shows promise for producing cellulose-based bioproducts, such as nanocellulose.

Keywords: Kiri; Chemical composition; Basic density; Bioproducts

RESUMO

A madeira de *Paulownia tomentosa* Steud. tem potencial para o desenvolvimento de produtos com alto valor agregado. A madeira é leve e macia, com excelentes propriedades de usinagem e acabamento, e por isso é usada para fabricação de materiais compósitos, chapas de madeira, móveis, papel e instrumentos musicais. Assim, o objetivo deste estudo foi avaliar as características físicas e químicas da madeira de *P. tomentosa*, a fim de recomendar seu uso para a obtenção e geração de bioprodutos. Cinco árvores, com treze anos de idade, foram abatidas para retirada de discos em diferentes alturas, os quais foram processados em cunhas e serragem. Para a determinação da densidade básica, foi empregada a metodologia descrita na norma NBR 11941. Os teores de extrativos totais, ligninas e holocelulose foram determinados segundo as normas da Technical Association of the Pulp and Paper industry – TAPPI. Análises de espectrometria no infravermelho, de química imediata (materiais voláteis, carbono fixo e cinzas) e termogravimétrica da madeira também foram realizadas. Os resultados obtidos para a madeira de *P. tomentosa* foram: densidade básica de 0,269 g/cm³, 17,3% de extrativos totais, 17,1% da lignina Klason, 1,15 de relação S/G e teores de holocelulose de 67,7%. A análise química imediata da madeira resultou em 86,5% de materiais voláteis, 12,9% de carbono fixo e 0,6 % de cinzas. A baixa densidade básica da madeira de *P. tomentosa*, bem como o baixo teor de lignina e de carbono fixo da madeira são características desfavoráveis do ponto de vista energético. Por outro lado, devido ao alto teor de holocelulose e baixo teor de lignina, a madeira de *P. tomentosa* se mostra promissora para a produção de bioprodutos de base celulósica, como a nanocelulose.

Palavras-chave: Kiri; Composição química; Densidade básica; Bioprodutos

1 INTRODUCTION

Wood is a renewable natural resource, primarily composed of cellulose, hemicellulose, and lignin, and it can be categorized into different types based on its technological characteristics. Each wood species has distinct characteristics that vary according to the tree species, site characteristics, and/or production process (Farias; Melo, 2020). Basic density is an important physical property of wood used to assess its quality and determine its suitability as a raw material. It is utilized in various applications such as cellulose pulp production, sawmills, board and joinery manufacturing, and energy generation by burning firewood, charcoal, pellets, or briquettes.

Understanding the chemical and physical properties of wood enables one to adapt any type of processing, make informed decisions regarding its application as a raw material, and calculate transportation and logistics (Carvalho *et al*., 2023). Apart from the physical properties, the quantitative evaluation of the chemical constituents is crucial to determine the suitability of wood as a raw material for various applications. The presence and relative content of each constituent play a significant role in the derived products (Nascimento *et al*., 2021; Li *et al*., 2023).

The genus *Paulownia* consists of nine fast-growing species native to China and Southeast Asia, commonly known as 'kiri.' The *Paulownia tomentosa* Steud. species is used for ornamental purposes, reforestation of degraded areas, and in the lumber industry. The wood is lightweight and soft, with excellent machinability and finishing properties (Jakubowski, 2022). The *Paulownia tomentosa* Steud. species stands out for its high productivity, allowing its timber potential to be exploited in short rotation periods. It has a specific mass of approximately 0.35 g/cm³, is resistant, easy to dry, and has good workability (Esteves *et al*., 2022).

Kiri is considered a key species for the future due to its adaptability to various soil and climate conditions. Its wood has the potential to be used in the manufacturing of boards, composite building materials, furniture, paper production, and musical instruments (Suri *et al*., 2022). This study aimed to evaluate the physical and chemical properties of *Paulownia tomentosa* Steud. wood in order to recommend its use in the production of energy and cellulose-based bioproducts.

2 MATERIALS AND METHODS

2.1 Materials

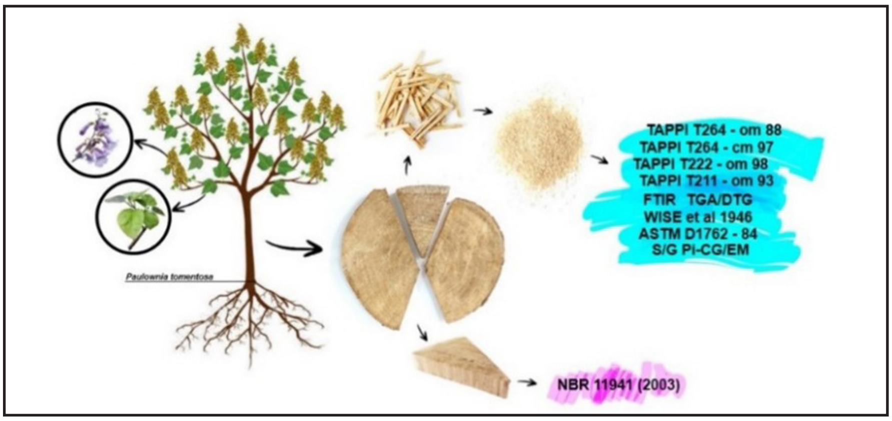
The study was conducted using five *Paulownia tomentosa* trees from a 13-year-old forest stand in Tuparendi, a municipality in the northwestern region of Rio Grande do Sul State (southern Brazil). To sample the trees, a chainsaw was used to remove five 3-cm thick discs from each tree at different heights: 0% or base, 25%, 50%, 75%, and 100% of the tree's commercial height (minimum diameter of 12 cm). This resulted in a total of 25 samples. The discs were then divided into four wedges, with two wedges used for determining basic density and the other two for chemical analysis. The wedges designated for chemical analysis were converted into sticks and then processed into sawdust using a Willey mill. The sawdust was separated using an electromagnetic shaker, specifically the 40/60 mesh fraction. Finally, the material was stored in glass jars to determine its moisture content.

2.2 Methods

The hydrostatic balance method, as outlined in standard NBR 11941, was used to determine the basic density of *Paulownia tomentosa* wood, and TAPPI standards were employed to assess chemical composition. The total extractives content was determined using T264 cm-97, while lignin insoluble in sulfuric acid was measured using T222 om-98. The holocellulose fraction was analyzed using the Wise et al. method (1946). The Syringyl/Guaiacyl (S/G) ratio of the lignin was determined through Py-CG-MS. For this analysis, pyrolysis was conducted in a micro-furnace pyrolyzer (Frontier Laboratories Ltda., Fukushima, Japan) connected to a GC-MS apparatus (Shimadzu, model QP2020), with an Ultra-ALLOY® capillary column (UA5, 30 m x 0.25 mm ID Film thickness 0.25 μm). The sample was analyzed in duplicate, following the method adapted from Silvério *et al*. (2008). Infrared spectrometry was performed on a Shimadzu IR Prestige apparatus, utilizing the direct transmittance method and the KBr pellet technique. The spectra were obtained in the range of 400 to 4500 cm^{-1}, with 45 scans and a resolution of 2 cm^{-1}.

Paulownia tomentosa wood underwent immediate chemical analysis following ASTM D1762-84 standards, which included quantifying volatile material, ash content, and fixed carbon content. Thermogravimetric analysis (TGA) was carried out using NETZSCH TG 209F1 equipment, with the samples heated to a temperature of 800 ºC at 10 ºC/min under a nitrogen atmosphere (10 mL/min). The analysis produced thermograms, representing the loss of mass (%) starting from room temperature. The first derivative was then calculated by derivative thermogravimetry (DTG) to identify the points where mass loss peaks occurred. Figure 1 illustrates the sample preparation and the physicochemical analyses performed on *Paulownia tomentosa* wood.

Figure 1 – Preparation of *Paulownia tomentosa* wood and the physicochemical analyses carried out



Source: Authors (2023)

Descriptive statistics were employed to evaluate the data generated by the wood analysis, providing the standard deviation and general average of the data.

3 RESULTS AND DISCUSSIONS

3.1 Basic density

In this study, the wood of *Paulownia tomentosa* exhibited a low density, with an average value of 0.269 ± 0.10 g/cm³, which is consistent with the values reported in the literature for species of the same genus, ranging from 0.240 to 0.350 g/cm³ (Akyildiz; Kol, 2010; Kaymakci *et al*., 2011). Woods with densities below 0.550 g/cm³ are classified as low density, while those with values ranging from 0.550 to 0.720 g/cm³ are considered medium density, and those above 0.730 g/cm³ are classified as high density (Silveira *et al*., 2013).

Lower-density woods have lower mechanical resistance compared to high-density woods. Based on the classification of the basic density of *Paulownia tomentosa* wood, its usage should be limited to products that do not require significant mechanical effort. It could be utilized for boards and battens, temporary constructions, joinery, light packaging, and panel core (Chen *et al*., 2020).

For cellulose pulp production, wood density is a crucial parameter that must be analyzed because increasing the basic density of the wood allows for a greater quantity of wood in the digester, leading to higher production (Pereira *et al*., 2000). Nevertheless, higher wood density makes it more challenging to individualize the fibers, necessitating a higher alkaline load and longer time/temperature for delignification, which results in reduced yield. Therefore, due to its low density and rapid growth, *Paulownia tomentosa* wood could be considered for pulp extraction.

In paper production, cellulose pulps obtained from less dense woods (e.g., *Paulownia tomentosa*) have larger fiber and lumen diameters, lower fines content, thinner fiber walls, and lower coarseness values (Mokfienski *et al*., 2008). In fact, fibers with thinner walls and larger lumen diameters have a higher potential for collapse, are easier to refine, provide greater contact area between fibers during paper sheet formation, and consequently exhibit higher tensile strength (Mokfienski *et al*., 2008). Mechanical strength and opacity are the primary quality attributes of writing and printing papers.

3.2 Chemical characterization

Lignocellulosic biomass consists of cellulose, hemicelluloses, lignin, and extractives, which vary in quantity and chemical composition. Understanding the chemical makeup of wood is important for identifying potential future uses (Hsing *et al*., 2016). Table 1 presents the average values for the chemical composition of various hardwoods and compares these values with the findings for *Paulownia tomentosa* wood.

Cellulose and hemicellulose are the main components of the carbohydrate fraction in wood, collectively known as holocellulose. As shown in Table 1, *Paulownia tomentosa* wood exhibited the highest holocellulose content (81.9%) and the lowest lignin content (17.1%) compared to values reported in the literature for wood of the same genus (Kalaycioglu *et al*., 2005; Ashori; Nourbakhsh, 2009). Therefore, it can be considered a high-quality raw material for producing cellulose-based products such as nanocellulose and cellulose pulp for paper. Holocellulose is desirable in the pulp and paper industry as it determines the characteristics of the pulp, such as strength and yield (Oliveira *et al*., 1982). Additionally, the compounds in wood contribute to producing a significant amount of acetic acid during thermal decomposition.

Table 1 – Mean chemical composition of *Paulownia tomentosa* wood and other hardwood species

Species	Holocellulose*	Lignin Klason*	Total extractives*	Ash content*
Paulownia tomentosa [a]	81.9	17.1	17.3	0.6
Paulownia tomentosa [b]	78.8	22.1	-	-
Paulownia fortunei [c]	81.2	24.6	-	-
Toona ciliata [d]	70.3	18.6	10.0	1.0
Eucalyptus spp. [e]	71.4	24.3	2.5	-
Eucalyptus saligna [f]	69.9	26.0	4.5	0.5
Ilex paraguariensis [g]	66.6	16.2	18.5	3.6
Ochroma pyramidalis [h]	67.9	26.5	5.6	1.1

Source: Authors (2023)

In where: * values are given in percentage; [a] this study; [b] Kalaycioglu; Deniz; Hiziroglu (2005); [c] Ashori and Nourbakhsh (2009); [d] Bufalino; Protásio; Couto; Nassur; Sá; Trugilho; Mendes (2012); [e] Andrade; Minhoni; Sansígolo; Zied (2010); [f] Trugilho; Lima; Mendes (1996); [g] Rosa (2020); [h] Caldeira (2017).

An increase in lignin content leads to a decrease in holocellulose content, resulting in wood with lower basic density. This finding supports the low density of *Paulownia tomentosa* wood obtained in this study (0.269 ± 0.10 g/cm³) (Vale *et al*., 2010).

In addition to quantifying lignin content, it is also important to study the chemical structures and functional groups that comprise it, as they directly influence the reactivity and yield of wood transformation processes for various applications (Longue et al., 2013).

The S/G ratio obtained in this study was 1.15 ± 0.25, lower than those reported in studies with eucalyptus wood, which range from 2 to 4.3 (Gomes; Vieira, 2023; Santos *et al*., 2016). A lower S/G ratio can positively impact energy yield due to the presence of aromatic C5 positions in the guaiacyl group, which enables the formation of condensed and thermally stable chemical structures during lignin biosynthesis (Santos, 2016).

Therefore, higher lignin content and a lower S/G ratio in wood carbonization processes result in higher charcoal yield due to the greater resistance to thermal degradation from more condensed chemical structures (Santos, 2016). This finding highlights the potential of *Paulownia tomentosa* wood for energy applications.

From the perspective of cellulose-based product manufacturing, the delignification process and accessibility depend on the reactivity of lignin, which is influenced by the S/G ratio. Woods with a higher S/G ratio are easier to process, affecting the fiber yield in pulp mills (Gomes; Vieira, 2023). Despite its lower S/G ratio (1.15), *Paulownia tomentosa* wood could still serve as a fiber source for pulp industries due to its lower lignin content (17.1%) compared to eucalyptus wood (as per Table 1), which is commonly used in Brazil for cellulose pulp production.

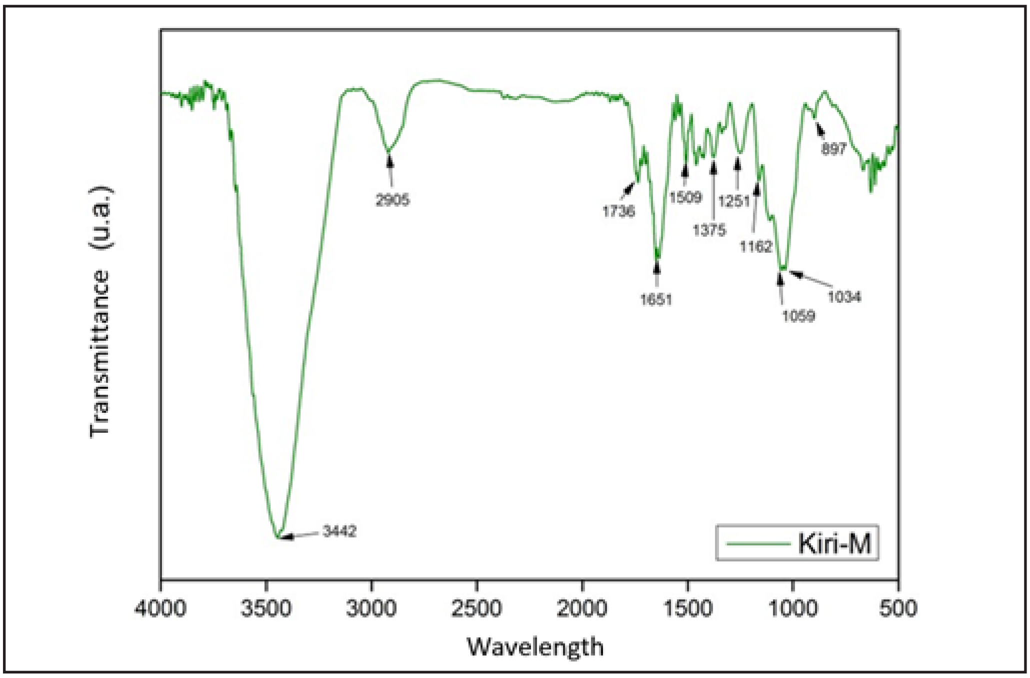
Extractives in wood play a crucial role in its use and impact its physical properties. Colored and volatile constituents add aesthetic value, while certain phenolic compounds provide resistance against fungal and insect attacks, increasing wood durability (Wastowski, 2018). *Paulownia tomentosa* wood exhibited a higher extractive content (17.3%) than eucalyptus wood (see Table 1), which could potentially affect the pulping yield of *Paulownia tomentosa* wood.

Almeida, Brito and Perré (2010) conducted a study to evaluate the impact of wood extractives on pulping. They recommended considering the extractive content as a criterion for selecting wood for kraft pulp production. While most extractives are removed during the pulping process, they consume reagents and negatively affect the quality of the final product, leading to a reduction in pulp yield.

According to the resistance to thermal degradation of wood extractives, high levels of this component enhance energy production by increasing the calorific value (Brand, 2015). Therefore, the *Paulownia tomentosa* wood studied in this research may be suitable for energy production. It is important to note that extractives are constituents that occupy lumens and vacuoles, which are spaces outside the cell walls. Consequently, they contribute to the dry weight of the biomass, resulting in energy gains.

The mean ash content of *Paulownia tomentosa* wood in this study was 0.78% (Table 1). Low ash content, such as that observed in this wood, is favorable for energy use as firewood and charcoal production. According to Andrade (2010), the accumulation of minerals in wood interferes with its combustion and other combustible properties when used as an energy source. The mineral compounds in the wood can also transfer to the charcoal, significantly reducing its quality in some cases. As the ash content increases, the chemical properties of charcoal are negatively affected, leading to a decrease in carbon content.

Figure 2 – Kiri-M - Fourier transform infrared spectroscopy (FTIR) of *P. tomentosa* wood



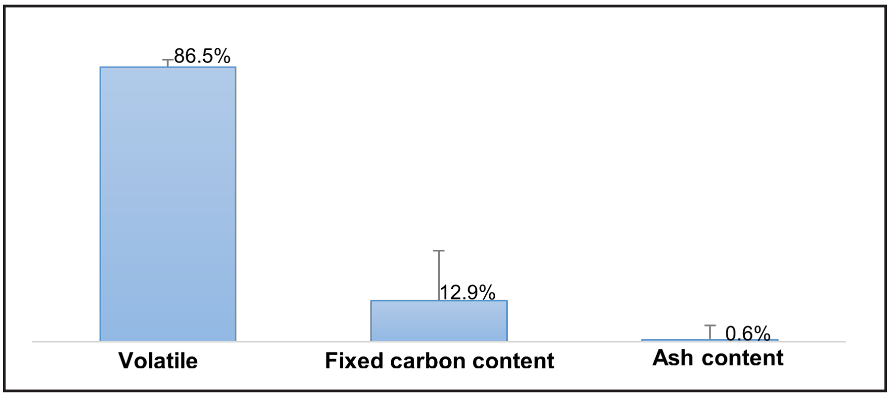
Source: Authors (2023)

The presence of low-intensity spectral bands at 1251 cm^{-1} is characteristic of the C-O ester group attributed to lignin (Xia *et al*., 2016). The band at 1736 cm^{-1} is attributed to the C=0 stretching vibration of the carbonyl and acetyl groups of hemicelluloses (Oun; Rhim, 2016). Additionally, the bands at 1509 and 1251 cm^{-1}, which are vibrations of the aromatic ring, are related to the presence of lignin and the oxygen stretching vibration mode associated with hemicelluloses, respectively (Mokfienski *et al*., 2015).

The band between 3500 and 3200 cm^{-1} refers to the 0-H stretch, which is characteristic of cellulose (Mandal; Chakrabarty, 2011). The bands at 897 and 1059 cm^{-1} also correspond to the structure of cellulose (Alemdar; Sain, 2008), and the band centered at 1162 cm^{-1} is associated with the asymmetric C-O-C stretch of cellulose (Chen *et al*., 2016).

The following peaks were recorded: 1375 cm^{-1} (C-H bending), 1338 cm^{-1} (O-H bond in-plane bending), 1109 cm^{-1} (C-O-C of glycosidic ether bond), 1059 cm^{-1} (C-O-C stretching vibration of pyranose ring), and 1034 cm^{-1} (C-O-C of hemicellulose or lignin ether bond) (Johar *et al*., 2012). The results of the immediate chemical analysis of *Paulownia tomentosa* wood are shown in Figure 3.

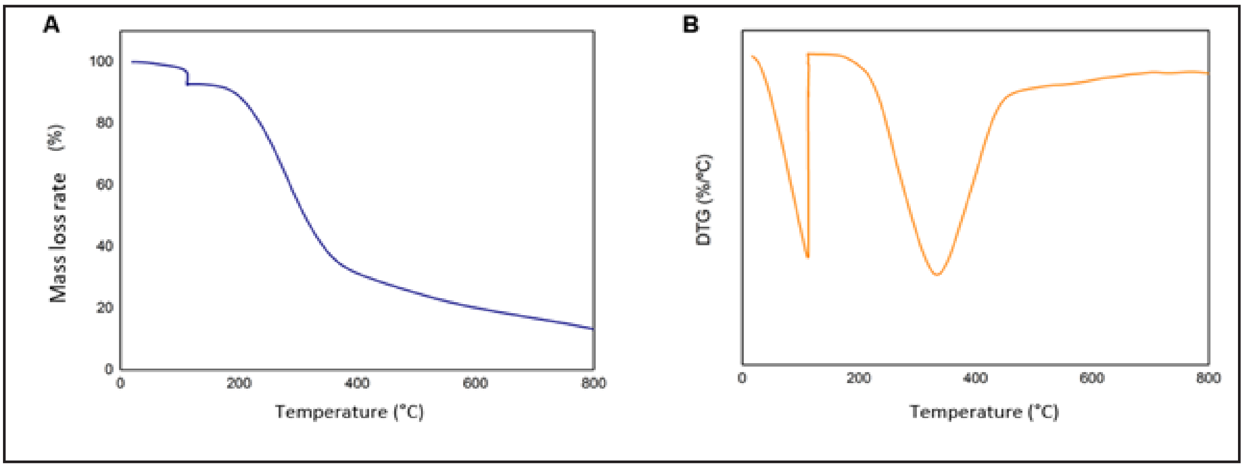
Figure 3 – Volatile material, fixed carbon content, and ash content of *Paulownia tomentosa* wood



Source: Authors (2023)

According to Figure 3, the values for volatile materials, fixed carbon content, and ash content were obtained as 86.5 ± 2.0%, 12.9 ± 2.0%, and 0.6 ± 0.03%, respectively. Gouvêa *et al*. (2017) stated that there is an inverse relationship between lignin content in wood and the production of volatile materials. This can be attributed to the chemical structure of lignin, which contains an aromatic ring and a higher amount of fixed carbon. The findings of this study are consistent with this statement, as *Paulownia tomentosa* wood had a high content of volatile materials (Figure 3) due to its low lignin content (as per Table 1). The thermal degradation behavior of *Paulownia tomentosa* wood was analyzed using TGA and DTG. The results are presented in Figure 4.

Figure 4 – Thermogravimetric and derivative thermogravimetric analyses of *Paulownia tomentosa* wood



Source: Authors (2023)

In where: A: mass loss rate (%) as a function of temperature, determined by TGA; B: mass loss rate, determined by calculating the first derivative (%/ºC)

According to Figure 4, the mass loss of *Paulownia tomentosa* wood can be observed through three distinct events. Firstly, there is the evaporation of water, which occurs up to a temperature of 100 °C. Secondly, there is the thermal degradation of cellulose, which exhibits its highest rate between temperatures of 300–325 °C. Lastly, carbonaceous residues are degraded at temperatures above 400 °C (Ouajai; Shanks, 2005). The thermal degradation of wood initiates around 226 °C, and its degradation rate reaches its peak at 334 °C. This peak is attributed to the degradation of hemicelluloses and lignin.

4 CONCLUSIONS

The basic density of *Paulownia tomentosa* wood was 0.269 g/cm^3, and the wood's chemical composition consisted of 17.3% total extractives, 17.1% Klason lignin (with an S/G ratio of 1.15), and 67.7% holocellulose. The immediate chemical analysis indicated 86.5% volatile materials, 12.9% fixed carbon content, and 0.6% ash content. Based on these results, it is possible to identify potential uses for this wood to increase its value and appreciation as a species. The low basic density, lignin content, and fixed carbon content of *Paulownia tomentosa* wood are not advantageous from an energy standpoint. However, due to its high holocellulose and low lignin content, *Paulownia tomentosa* wood shows promise for producing cellulose-based bioproducts, such as nanocellulose.

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