Determination of the Elastic Modulus of *Dinizia excelsa* Wood at High Temperatures Using Impulse Excitation Technique (IET)

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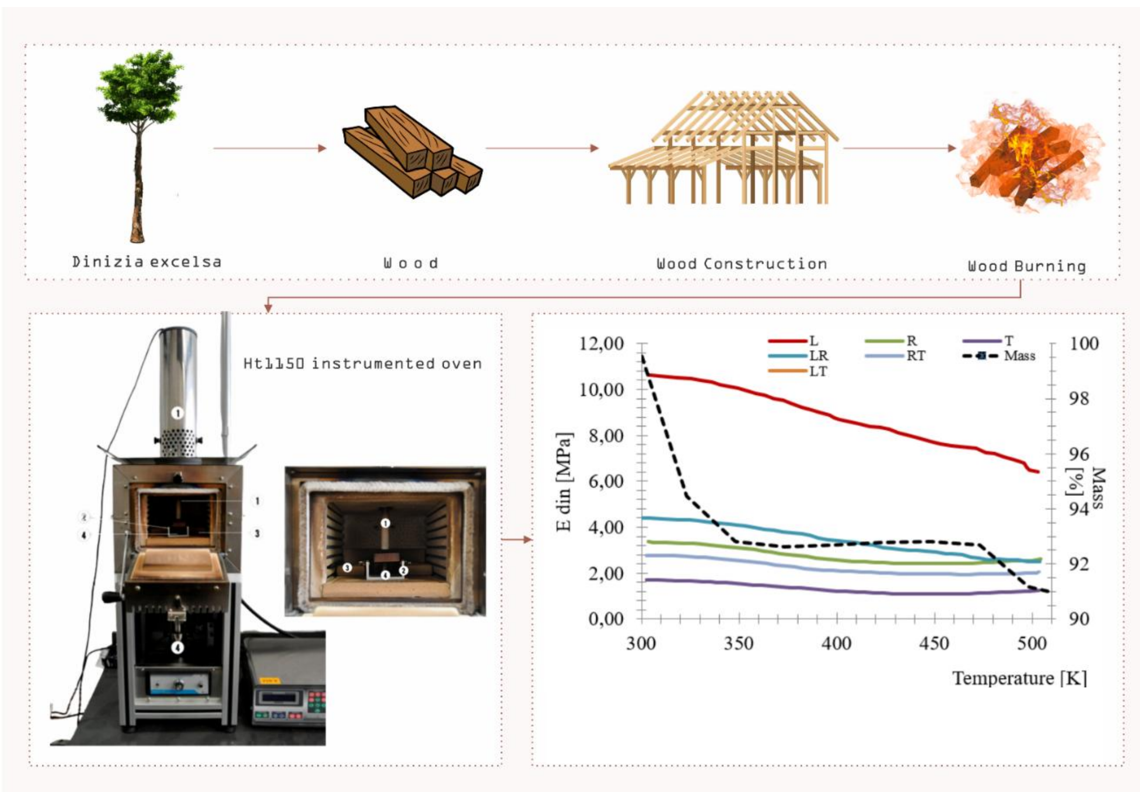
GRAPHICAL ABSTRACT



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Wood, as a renewable and highly abundant material, has been receiving increasing attention for use in high-performance applications, such as a structural element subjected to high temperatures. For its successful implementation in the construction or timber industry sector, it is crucial to understand its behavior during and after exposure to high temperatures. In this study, the red angelim wood, *Dinizia excelsa*, was subjected to high temperatures, up to a temperature of 508 K, using the dynamic excitation wave propagation test. Samples tested in the furnace were dimensioned in six distinct directions: three main ones (radial, tangential, and longitudinal) and three intermediate ones at 45º (longitudinal-radial, longitudinal-tangential, and radial-tangential). The static test used only the main directions of wood orientation. The values of elasticity modulus exhibited a reduction after the heat treatment, resulting in significant decreases of up to 45%. Results demonstrated that the excitation wave propagation method was effective in estimating the elasticity modulus at room temperature up to 508 K. Therefore, this study contributed to the construction of a database that can be expanded by future research focused on Brazilian woods.

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Keywords: Wood; Temperatures; Elasticity modulus; Wave propagation

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INTRODUCTION

The use of wood in construction has a long history; it is a widely used material in various structures due to its availability, strength, beauty, and ease of processing (Vilela and Mascia 2021; Ruthes *et al*. 2022; Eloy *et al*. 2023; Rodrigues *et al*. 2023; Lima *et al*. 2024). In the Brazilian context, where vast expanses of tropical forests sustain a robust forestry industry, wood continues to play an essential role in architecture and civil engineering (Araújo *et al*. 2021; Batista *et al*. 2023). However, the mechanical response of wood under extreme conditions, such as high temperatures, is an aspect that has received relatively limited attention compared to its widespread use (Calonego *et al*. 2020; Bandera *et al*. 2021; Lengowski *et al.* 2021; Oliveira *et al*. 2021).

Determining the modulus of elasticity, one of the fundamental mechanical properties of wood, is crucial for structural design and evaluation. This parameter quantifies the material's ability to withstand loads and deformations under the action of external forces. However, the variation of the modulus of elasticity under different environmental conditions, particularly at high temperatures, represents a critical aspect that directly influences the performance of structures built with wood (Juizo *et al.* 2019; Aydin 2020; Vieira *et al*. 2024). These changes, in turn, have a direct impact on the mechanical characteristics of the material (Barros and Politano 2019; Menezes *et al.* 2019). However, most studies in this area focus on woods from European and North American regions, leaving a notable gap in understanding the behavior of Brazilian tropical wood under high temperatures.

Wave propagation in orthotropic materials subjected to high temperatures is a complex and ever-evolving topic. The reviewed studies emphasize the importance of considering temperature as a determining factor, while recent research indicates promising advancements in the use of non-destructive techniques to assess the behavior of these materials under extreme conditions (Bucur 1984; Reem and Samir 2024).

One aspect to be considered is the significant innovation that the application of the impulse excitation technique (IET) in high-temperature environments represents for the analysis of materials under extreme conditions (El Najjar and Mustapha 2020; Palma and Steiger 2020; Bandera *et al*. 2021). This technique, based on the generation of short-duration energetic pulses, allows for a dynamic assessment of the mechanical properties of materials even under unfavorable circumstances, such as high temperatures. Such an innovative approach plays an essential role in understanding the behavior of materials in situations of intense thermal exposure, contributing to the design and maintenance of structures subject to such challenging conditions.

In recent years, there has been a notable increase in interest in the application of IET, with recent research highlighting its effectiveness in obtaining precise data on the mechanical properties, such as the modulus of elasticity, of materials under these circumstances (Almeida *et al.* 2016; Ferreira *et al.* 2023; Yassine and Mustapha 2023). This technique offers a unique opportunity for engineers and researchers to explore the behavior of structural materials under conditions of extreme thermal exposure, providing valuable insights for the development of safer and more efficient structures.

The goal of this study is to provide an in-depth understanding of how wood reacts when exposed to high-temperature conditions, such as during the industrial drying process aimed at reducing moisture content, or in certain painting procedures where wood is heated to facilitate material drying. This analysis is essential for evaluating the behavior of wood under these circumstances and its implications for the physical and mechanical properties of the material.

Despite notable progress, the application of IET at high temperatures in various materials still represents a relatively new research field, with vast possibilities to be explored. Therefore, this article aims to analyze the behavior of impulse wave propagation in high-density tropical wood under temperature variation of up to 508 K and evaluate its efficiency for estimating the modulus of elasticity.

EXPERIMENTAL

Materials and Methodology

The study was conducted at the Advanced Research Laboratory for Wood and New Materials (CPAM) at the Federal University of Minas Gerais (UFMG). The Brazilian tropical wood species, *Dinizia excelsa*, known as angelim vermelho, was utilized. Seven replicates per orientation direction of the specimens (cps) were employed, oriented in six different directions: the three principal (radial, tangential, and longitudinal) and the three intermediate directions at 45º (longitudinal-radial, longitudinal-tangential, and radial-tangential). Dynamic tests using IET were then conducted. For the static test, samples were sized according to NBR 7190 (2023) and oriented in three principal directions (radial, tangential, and longitudinal) for the compression test.

Thermogravimetry

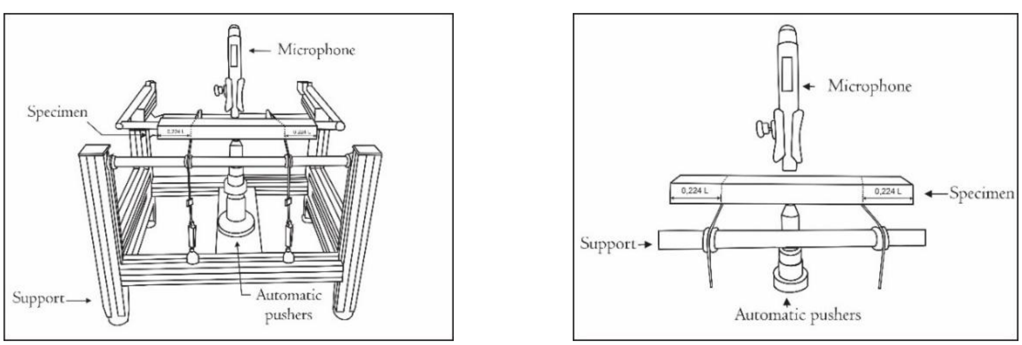
The wood samples were converted into sawdust using a Wiley-type laboratory mill, following the TAPPI 257 om-85 standard (2001). The seven samples of the species were combined into a single sample, resulting in a composite sampling from the seven repetitions. The mixture was then sifted, and the fraction classified between the 200 and 270 mesh sieves was collected.

For thermogravimetric characterization, the DTG-60H apparatus from Shimadzu was used. The analyses were conducted under a nitrogen gas atmosphere at a constant flow rate of 50 mL·min^{-1}, using approximately 2 mg in an open alumina capsule. Thermogravimetric curves were obtained from 323.15 K up to a maximum temperature of 673.15 K, with a heating rate of 283.15 K·min^{-1}. The thermogravimetric curve (TG) assesses mass loss as a function of temperature, and the first derivative thermogravimetric curve (DTG) evaluates the rate of mass loss.

Excitation Wave Propagation (IET)

Samples were subjected to characterization of flexural vibration modes. For this purpose, an adjustable support for bars, a pulser, a directional sensor, and specific software were used. Figure 1 illustrates the test methodology: in Fig. 1(A), the sample positioning for the determination of longitudinal elastic modulus (E) can be observed, and in Fig. 1(B), a zoom of the sample at the moment of the test.

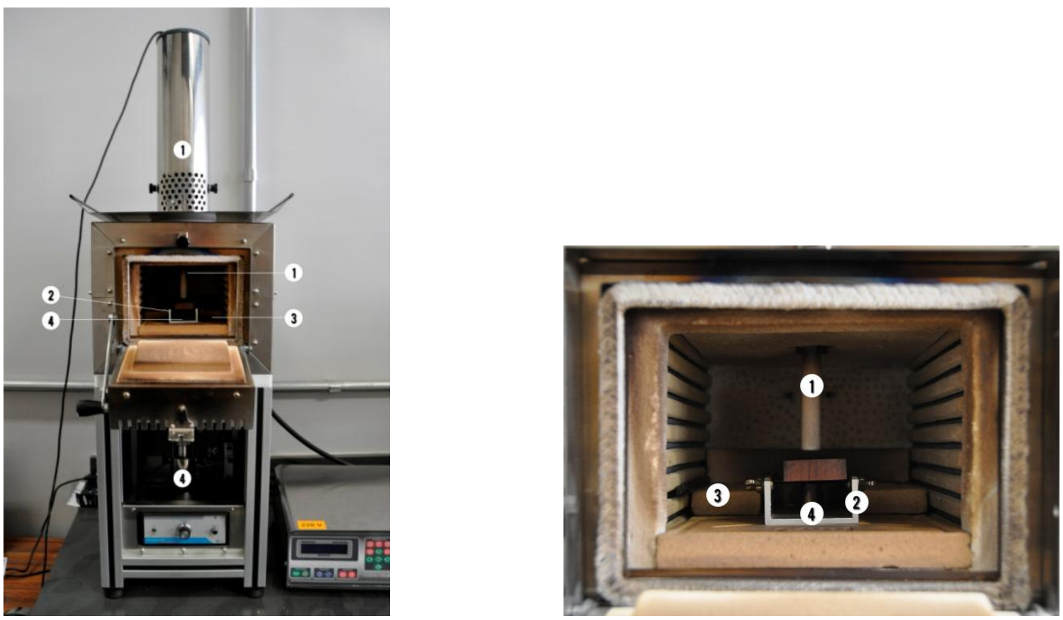
The impulse excitation technique (IET) is based on ASTM E1876 (2021). The Sonelastic testing instrument was employed to determine the elastic moduli from the natural vibration frequencies of the specimen (SP). The frequencies are excited by the striker, and the acoustic response is captured by a sensor. The signal undergoes mathematical treatment (Fourier transform) to obtain the corresponding frequency spectrum. Then, the dynamic elastic moduli are calculated, taking into account the SP's geometry, mass, dimensions, and the frequency obtained by the equipment used, Eq. 1.



(A) (B)

Fig. 1. (A) Sample positioning for the determination of longitudinal elastic modulus (E); (B) zoom of the sample at the moment of the test

The samples had dimensions of 17 x 10 x 150 mm, with 42 SPs, equally distributed in the radial, tangential, and longitudinal directions, as well as in the intermediate directions (RT, LR, and LT). The SPs were marked with lines positioned 0.224 L away from each end (where L represents the length of the SP). These positions correspond to the nodal lines of the SP concerning the fundamental flexural vibration mode for rectangular bar geometries. The test was conducted under ambient conditions and at high temperatures since, along with the Sonelastic equipment, an HT1150 type oven was attached. The oven was specifically designed for calculating the elastic and damping moduli as a function of temperature, allowing continuous use up to 1,423.15 K. The oven features a proportional integral derivative (PID) controller and internal dimensions of 100 x 150 x 200 mm. Figure 2 presents the equipment version, where the dynamic modulus of elasticity response is given as a function of the internal temperature inside the oven.



(A) (B)

Fig. 2. Equipment images. (A) Sonelastic HT1150 instrumented oven. (B) Zoom inside the Sonelastic HT1150 instrumented oven. Where 1 is Acoustic sensor; 2 - Metal profile for support of the test specimen; 3 - Ceramic mold for accommodating the test specimen support; 4 – Pulsator.

The IET also uses ASTM E1876 (2021) as the basis for its determinations. The methodology is similar to tests at room temperature; however, in this case, the value of dynamic modulus of elasticity was calculated up to a temperature of 508 K.

Regarding the determination of apparent density values, the test is not capable of detecting the mass and volume relationship simultaneously during the experiment. Therefore, the determinations occurred at two different times: before the tests began, when the sample had 12% moisture content, and after the completion of the test when a temperature of 508 K was reached. Overall, there was an apparent density loss of about 23%.

Next, the formulas employed in determining the longitudinal modulus of elasticity (E) will be presented, in which the bending vibration frequency (f_f) plays a crucial role in the calculation process, following the guidelines established by ASTM E1876 (2021).

$$R = \left[\frac{1+\left(\frac{b}{t}\right)^2}{4-2{,}521\frac{t}{b}\left(1-\frac{1{,}991}{e^{\pi\frac{b}{t}}+1}\right)}\right]\left[1+\frac{0{,}00851n^2b^2}{L^2}\right]-0{,}060\left(\frac{nb}{L}\right)^{\frac{3}{2}}\left(\frac{b}{t}-1\right)^2$$

$$T1 = 1+6{,}585\,(1+0{,}0752\mu+0{,}8109\mu^2)-\left(\frac{t}{L}\right)^2-0{,}868\left(\frac{t}{L}\right)^4 - \left[\frac{8{,}34\,(1+0{,}2023\mu+2{,}173\mu^2)\left(\frac{t}{L}\right)^4}{1+6{,}338\,(1+0{,}1408\mu+1{,}536\mu^2)\left(\frac{t}{L}\right)^2}\right]$$

$$\mathrm{E} = 0{,}9465\left(\frac{mf_f^2}{w}\right)\left(\frac{l^3}{t^3}\right)T1 \quad (1)$$

where E is the dynamic modulus of elasticity (Pa), m is the mass of the CP (g), b is the width of the CP (mm), L is the length of the CP (mm), t is the thickness of the CP (mm), f_f is the fundamental resonance frequency of the CP in bending (Hz), f_t is the fundamental resonance frequency of the torsion bar (Hz), and $T1$ is the correction factor for the fundamental bending mode for finite thickness.

Compression

To assess the accuracy of the IET, specimens oriented in the three main directions of the wood (radial, tangential, and longitudinal) were subjected to compression testing to determine the static modulus of elasticity. Figure 3 shows the specimen to be tested under compression according to NBR 7190 (2023), using the EMIC universal testing machine, model DL 30,000, with a load capacity of 300 kN, on a cell with a capacity of 100 kN, previously calibrated.

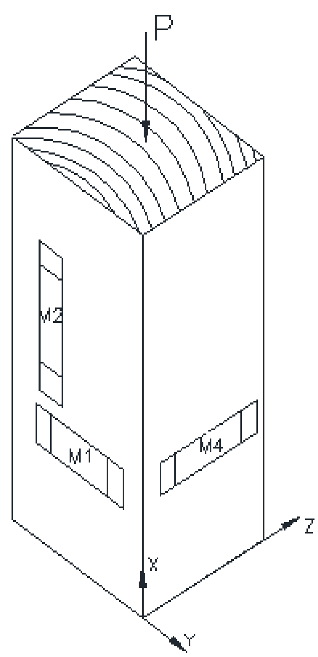


Fig. 3. Specimens tested under compression using the portable meter subjected to load P oriented in the longitudinal direction

Statistical Analysis

The data were subjected to Shapiro-Wilk normality and Bartlett's homoscedasticity tests. MiniTab 16 softwares was used for the aforementioned analyses.

Test Standards

The standards used throughout the research were ASTM E 1876 (2021), NBR 7190 (2023), and TAPPI 257 om-85 (2001).

RESULTS AND DISCUSSION

Figure 4 shows the determined values for the modulus of elasticity in samples of red angelim oriented in the three main directions (tangential, radial, and longitudinal), along with the average values of apparent density at 12% moisture content.

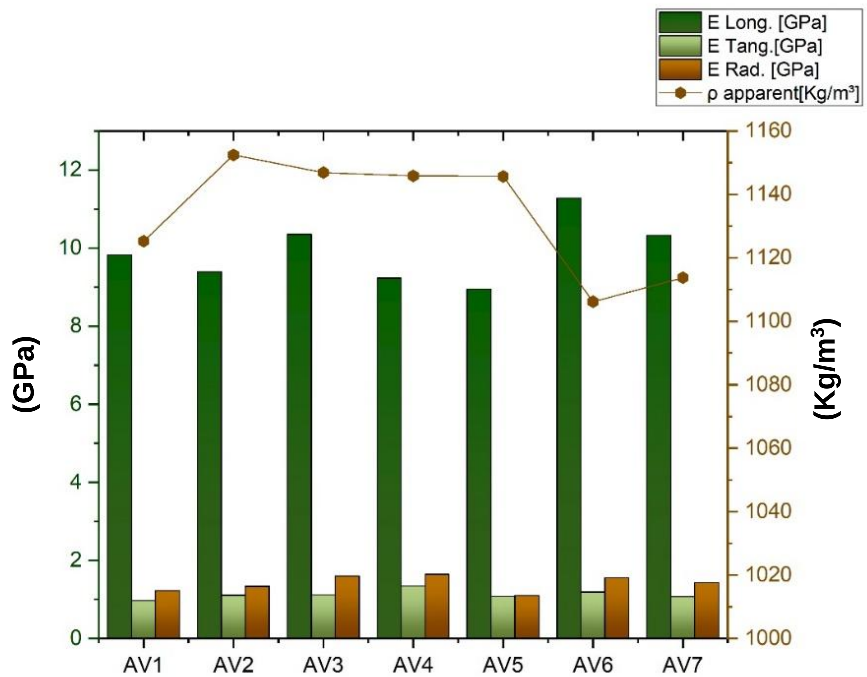


Fig. 4. Determined values of static modulus of elasticity in samples oriented in the three main directions of the wood, along with their respective apparent densities

The highest values of modulus of elasticity were not observed in samples with the highest apparent density. This phenomenon can be justified by variations in microfibril angle (MFA) and grain orientation. Alves (2017) supports this relationship by indicating that *Dinizia excelsa* exhibits high values of MFA (8.36) and grain deviation (3.89) when compared to other studied Brazilian species, contrary to results found by Lai *et al*. (2020). It can be observed that, as expected, the modulus of elasticity values is higher in the longitudinal direction of the fibers, followed by the radial and tangential directions. This is because the wood fibers are more strongly oriented in the longitudinal direction, providing greater stiffness and strength in this direction. The tangential and radial directions have a less organized structure, resulting in lower modulus of elasticity values. The longitudinal modulus of elasticity values ranges from 8.95 to 11.28 GPa, while the radial modulus of elasticity ranges from 1.1 to 1.64 GPa and the tangential modulus of elasticity ranges from 0.97 to 1.35 GPa. The proportions of the modulus EL/ET are: 8.79; EL/ER: 7.00; and ER/ET: 1.26.

The results obtained from the test using IET under high temperatures are shown in Fig. 5.

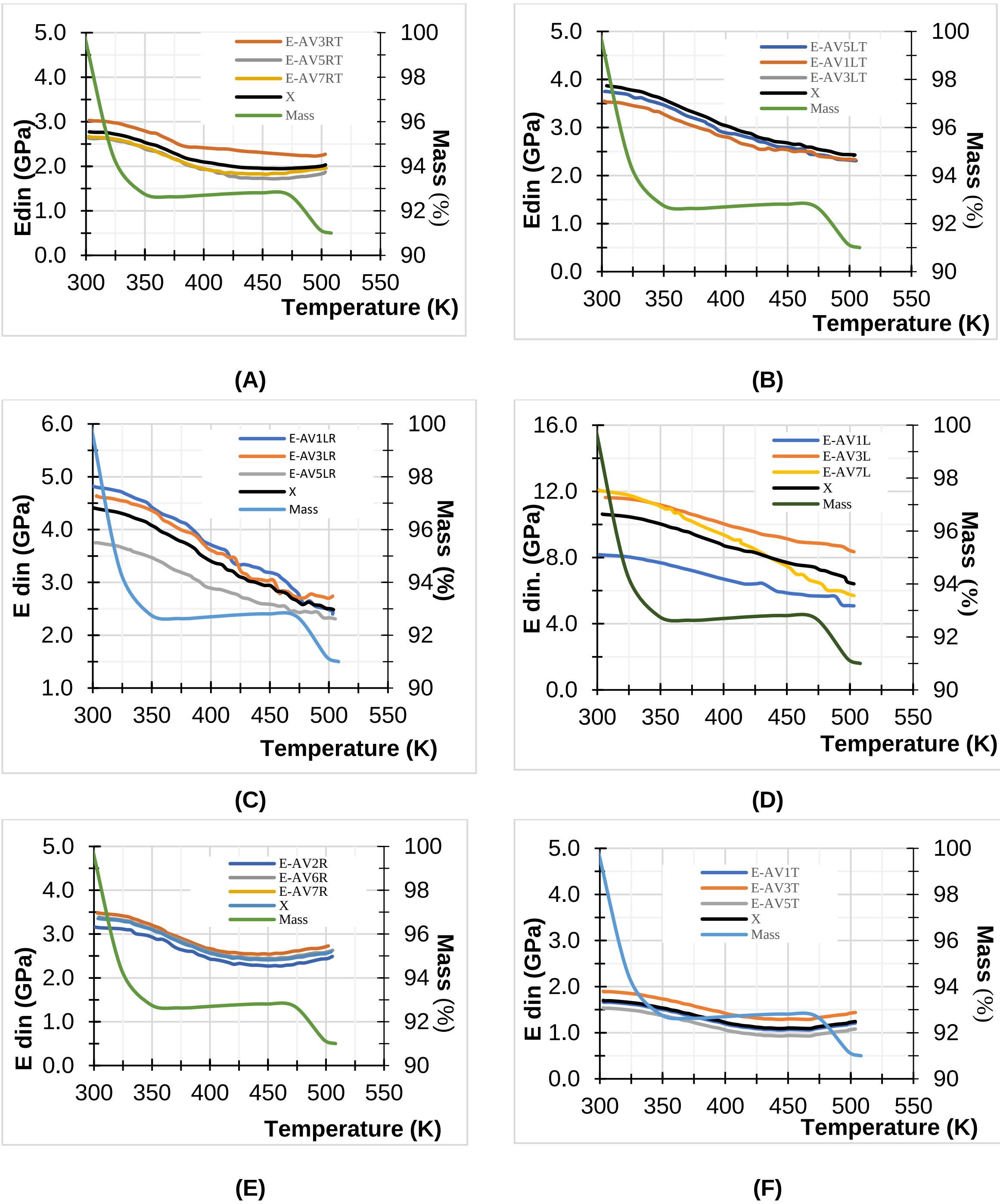


Fig. 5. Variation of longitudinal modulus of elasticity and CP mass with increasing temperature. (A) Radial Tangential Direction; (B) Longitudinal Tangential Direction; (C) Longitudinal Radial Direction; (D) longitudinal to the fibers; (E) Radial Direction; and (F) Tangential Direction.

There is an evident inverse relationship between the modulus of elasticity and temperature, meaning that as the temperature increases, the values of dynamic modulus of elasticity decrease, with no increase compared to room temperature. This contradicts the findings of authors such as Bandera *et al*. (2021), who reported slight increases in modulus of elasticity at high temperatures, and Ferreira *et al*. (2019), who found no influence of high temperatures on modulus of elasticity values determined from static bending. For example, Aydin (2020) observed slight increases for the *Quercus petraea* Liebl species at moderate temperatures (up to 423 K), but in subsequent treatments, all measured properties decreased significantly, including modulus of elasticity.

The main changes in wood subjected to high temperatures occur in the degradation of hemicellulose by deacetylation, depolymerization, and dehydration, as well as in structural changes and rearrangement of lignin (Dalla Costa *et al*. 2020). These chemical changes, primarily the degradation of hemicellulose, are considered responsible for the decrease in mechanical properties, as evidenced by reduced resistance to force application (Wentzel *et al*. 2019; Oliveira *et al*. 2022).

The decreases in modulus of elasticity values occurred differently in relation to the orientation directions, with a milder decrease in the radial direction (22.85%), followed by the longitudinal (23.24%), radial-tangential (28.63%), tangential (33.86%), longitudinal-radial (40%), and longitudinal-tangential (45.62%) directions, as shown in Fig. 6. Corroborating this result, Oliveira *et al*. (2022) found reductions of up to 57% in the modulus of elasticity determined from axial compression in samples subjected to temperatures ranging from 428 to 458 K. In contrast, Danihelová *et al*. (2022) studied *Picea abies* (L.) Karst. and *Acer pseudoplatanus* L. woods with thermal processes using the ThermoWood technique and treatments at 408, 433, and 458 K, reporting an increase in specific modulus of elasticity in all treatments, with the highest increases (6.8%) for spruce wood treated at 433 K and 7.8% for maple wood treated at temperatures of 433 and 458 K.

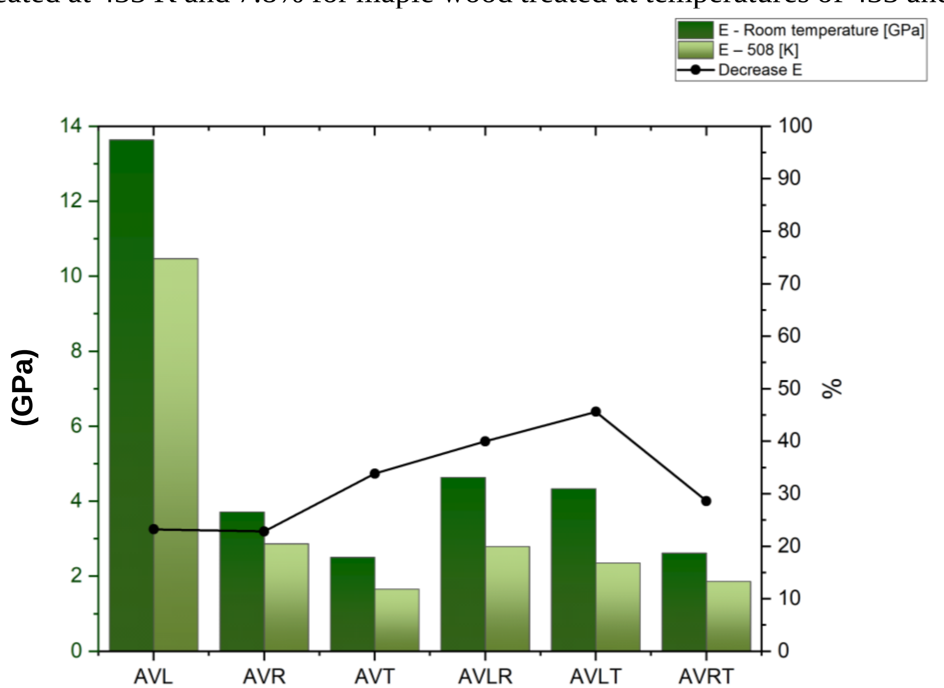


Fig. 6. Relationship of dynamic modulus of elasticity at room temperature and high temperature for the six main orientation directions

All samples in the study confirmed the trend of decreasing elastic modulus with increasing temperature, regardless of the orientation direction. This reduction can be justified by thermal expansion, which reduces the value of d*F*/d*A* (differential force over area, defining stress) and increases the separation distance between atoms, thereby reducing the binding energy between them and the effort needed to produce deformation, as well as the transformation of wood anatomical elements. Irvine (1984) mentions that the softening of constituents is accompanied by significant changes in the material's mechanical properties, with elastic modulus being the main one. Meanwhile, Hill *et al.* (2021) explain that there is a reduction in natural resonance frequency with temperature, which may be associated with the reduction in wood density as well as modifications associated with some chemical constituents of wood cell wall structure, such as degradation of hemicelluloses, free hydroxyl groups (-OH) in the amorphous region of cellulose, extractives, and lignin cross-linking.

For the present study, the changes in apparent density values were approximately equivalent in the three principal directions of the wood, resulting in a reduction of about 23% when subjected to a temperature of 508 K. This confirms that the loss of strength and elasticity is related to a set of factors, including the acid hydrolysis of hemicelluloses, which facilitates the breaking of microfibril bonds, and the softening of lignin, which occurs at temperatures between 413 and 433 K. In this range, lignin begins to disorganize and becomes more pliable, facilitating manipulation and processing. The softening of cellulose generally starts at around 473 to 493 K, at which point the structure of cellulose may destabilize, affecting its physical and mechanical properties. Therefore, the loss of elasticity can be explained by a combination of factors. Another important analysis is that even though the decrease occurred in samples oriented in the six wood directions, it occurred differently, showing that the anatomical arrangement alters the natural frequency response, and consequently the dynamic elastic modulus (Edin). The elastic modulus can be influenced by anatomical characteristics and not just material density (Korkmaz and Büyüksarı 2019; Ferreira *et al.* 2023; Viala *et al.* 2024). The decrease in Edin varied from 23 to 46% for longitudinal and longitudinal-tangential directions, while the mass loss determined from the thermogravimetric test was about 8%, as shown in Fig. 7.

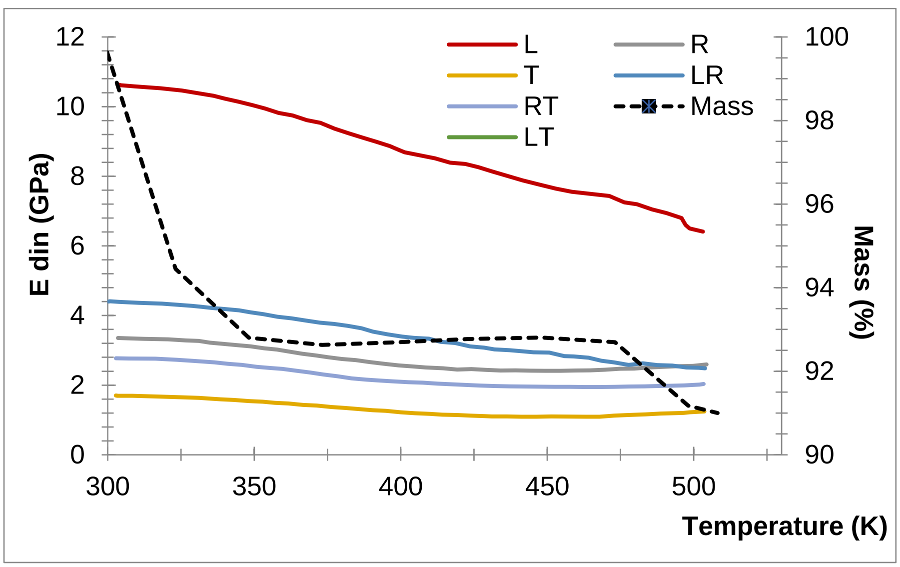


Fig. 7. Variation of longitudinal elastic modulus and sample mass with increasing temperature, in the six sample orientation directions

CONCLUSIONS

1. Reduction in Elastic Moduli: This pioneering study revealed a clear trend of reduction in the elastic moduli of the tropical wood *Dinizia excelsa* with increasing temperature, resulting in significant decreases of up to 45%.
2. Degradation Behavior: Although the degradation behavior of the wood appeared similar in the six directions analyzed under high temperatures, the loss of elasticity did not follow a uniform proportion, highlighting the influence of the wood's anatomical arrangement.
3. Significant Reductions in Secondary Directions: The most significant reductions were observed in the secondary directions, namely longitudinal tangential and longitudinal radial, indicating differentiated sensitivity in these orientations.
4. Importance of Findings: These innovative discoveries underscore the crucial importance of exploring Brazilian tropical woods and understanding the impact of temperature on the elastic properties of these materials.
5. Contribution to Knowledge and Practices: This advancement contributes not only to the existing knowledge but also to the development of more efficient practices in the use of IET under challenging thermo-mechanical conditions.

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