

THERMAL MODIFICATION OF A FAST-GROWING COMMERCIAL WOOD IN BRAZIL

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ABSTRACT - Heat treatment of *Pinus elliottii* wood is an essential practice for improving its properties and durability. This study aims to evaluate the changes resulting from this process at three temperature ranges, for a period of 2 h. The main objective of this study was to evaluate the thermal modification process of *P. elliottii* wood at different temperatures over a two-hour period in a forced circulation laboratory oven. The study used samples of *P. elliottii* wood, subjected to heat treatments at three temperatures (160°C, 200°C and 240°C) for 2 h. Apparent density, chemical composition, thermal stability, mechanical and colorimetric properties were analysed to assess the effects of the treatments. The heat treatments on *P. elliottii* wood had varied results. The 160°C treatment increased rigidity and resistance, while the 200°C treatment provided greater thermal stability. Both are recommended for different purposes. However, the 240°C treatment damaged the wood's properties. There was a decrease in mass loss with no significant impact on specific mass. The wood also darkened as a result of the heat treatments.

Keywords: *Pinus elliottii*, thermal modification, mechanical properties.

RETIFICAÇÃO TÉRMICA DE UMA MADEIRA COMERCIAL DE RÁPIDO CRESCIMENTO NO BRASIL

RESUMO - O tratamento térmico em madeira de *Pinus elliottii* é uma prática essencial para melhorar suas propriedades e durabilidade. Este estudo busca avaliar as mudanças resultantes desse processo em três faixas de temperatura, por um período de 2 h. Este estudo teve como principal objetivo avaliar o processo de modificação térmica da madeira de *P. elliottii* a diferentes temperaturas, em estufa laboratorial de circulação forçada. O estudo utilizou amostras de madeira de *P. elliottii*, submetidas a tratamentos térmicos em três temperaturas (160°C, 200°C e 240°C) por 2 h. Foram realizadas análises de massa específica, composição química, estabilidade térmica, propriedades mecânicas e colorimétricas para avaliar os efeitos dos tratamentos. Os tratamentos térmicos na madeira de *P. elliottii* tiveram resultados variados. O tratamento a 160°C aumentou rigidez e resistência, enquanto o de 200°C proporcionou maior estabilidade térmica. Ambos são recomendados para diferentes fins. No entanto, o tratamento a 240°C danificou as propriedades da madeira. Houve diminuição na perda de massa sem impacto significativo na massa específica. Também ocorreu escurecimento da madeira devido aos tratamentos térmicos.

Palavras-chaves: *Pinus elliottii*, modificação térmica, propriedades mecânicas.

INTRODUCTION

Wood is an important natural and renewable resource that exhibits rapid growth and versatility in technological properties, especially when derived from planted forests, making it one of the main sources of raw material for various sectors of the timber industry. However, species planted in Brazil generally have low natural resistance (GUMANE et al., 2019). Due to the heterogeneity of wood, it has some technological limitations, such as water absorption and desorption, resulting in high dimensional instability and making it susceptible to biodegradation and deterioration by insects and fungi (CERMÁK et al., 2022).

For several centuries, wood modification has been used as a method to enhance its properties and lifespan. Chemical, thermal, and impregnation processes are the most

used in industries to modify wood, considered innovative methods to improve its physical, mechanical, and aesthetic properties, with the aim of developing products such as sawn timber, fibre panels, and reinforced composite wood (JONES and SANDBERG, 2020).

Among these methods, thermal treatment has stood out as a viable alternative, as it has a lower environmental impact by not requiring the use of harmful chemicals. In Brazil, the use of thermal treatments has gained prominence in the pursuit of sustainability and greater wood durability without harming the environment (GUMANE et al., 2019).

Thermal treatment of Pinus wood has been applied to generate internal chemical reactions, improve hygroscopic properties, and increase dimensional stability, mechanical strength, and durability, as well as reduce the

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occurrence of defects and decrease wood drying time. This treatment also results in changes in the wood's colour, depending on the temperature used (ACOSTA et al., 2021; TALGATTI et al., 2016). According to the European Committee for Standardization (ECS, 2008), cited by Jones and Sandberg (2020), thermal treatment occurs when wood is exposed to a temperature above 160°C with limited oxygen access, ranging between 160°C and 220°C. Temperatures below 140°C result in insignificant modifications, while temperatures above 300°C cause severe degradation of all major wood constituents. In this context, this study aims to evaluate the changes in physical, thermal, mechanical, and colorimetric properties after the heat treatment of *Pinus elliottii* wood at three temperature ranges (160°C, 200°C, 240°C) for a period of 2 h in a laboratory forced-air circulation oven.

MATERIAL AND METHODS

For this study, *Pinus elliottii* wood from a 22-year-old reforestation area located in the municipality of Piratini, Rio Grande do Sul, was used. Twenty-eight test specimens were prepared, each measuring 15 × 15 × 250 mm³ in the radial, tangential, and longitudinal planes, respectively. The test specimens were then randomly selected following the American Society for Testing and Materials standard (ASTM D 5536, 1995), with seven test specimens allocated to the control group and seven for each of the three treatments applied.

The heat treatment was performed under three temperature conditions (160°C, 200°C, and 240°C) for a period of 2h in a laboratory forced-air circulation oven. Afterwards, the wood samples were placed in a climate chamber at a temperature of 20 ± 2°C and relative humidity of 65 ± 3% until they reached the equilibrium moisture content (12%).

The characterisation of the samples included the calculation of apparent density (ρ) and weight percent gain (WPG) using an analytical balance and digital calliper. The chemical groups were evaluated by Fourier-transform infrared spectroscopy, coupled with an attenuated total reflectance device (ATR-FTIR), using Jasco® equipment (model 4100), covering a wavelength range from 2000 cm⁻¹ to 600 cm⁻¹, with a resolution of 4 cm⁻¹, a scanning speed of 2 mm s⁻¹, and a filter of 30,000 Hz.

Thermal stability was assessed by thermogravimetric analysis (TGA) on cubic samples of 10 mm³ using Navas equipment (model TGA-1000). The thermogravimetric curves were obtained through two heating ramps, both with a heating rate of 10°C min⁻¹, under an inert atmosphere and a nitrogen gas flow of 2 L min⁻¹. The first ramp was from 23°C to 105°C, and the second from 105°C to 600°C.

Mechanical properties were evaluated using a universal testing machine (Emic brand, model DL 3000) according to the ASTM D 143 standard. The modulus of elasticity (MOE) and modulus of rupture (MOR) were determined based on the results obtained from static bending tests conducted on samples with dimensions of 25 mm × 25 mm × 100 mm (radial × tangential × longitudinal). For toughness determination, an adaptation of

the ASTM D6110 standard was used, with the aid of a Charpy pendulum with a capacity of 500 kg cm⁻¹ up to 140 mm, subjecting the central region of the test specimen to impact in the tangential direction.

For the colorimetric analysis, four readings were taken on each test specimen, two for each plane (radial and tangential), totalling sixteen readings for each treatment. A Konica Minolta colorimeter, model CR-400, with a D65 illuminant source and a 10° observation angle, was used. The evaluated parameters were lightness (L*), red-green coordinate or red matrix (a*), blue-yellow coordinate or yellow matrix (b*), colour saturation (C*), and hue angle (h*), based on the CIELab 1976 colour system.

Statistical analysis was conducted using simple analysis of variance (ANOVA), and when a statistically significant difference was observed between the values, Fisher's LSD test was applied with a significance level set at 5%. Additionally, Pearson correlation was used to correlate the results obtained when necessary. All statistical procedures were performed using the Jamovi statistical software.

RESULTS AND DISCUSSION

As shown in Figure 1A, which presents the results obtained for mass loss, a distinct behaviour is observed among the treatments. As the temperature increases, there is a significant mass loss due to the decomposition of chemical constituents, primarily hemicelluloses, which are associated with low thermal stability. This reduction is attributed to the constant decrease in the number of hemicelluloses with the increased intensity of the treatment, caused by the degradation of OH groups (HAN et al., 2022). The figure shows a variation in mass loss from 3.7% to 15.4%, results that are similar to those obtained by Gallio et al. (2019) in their research on the thermal treatment of *Pinus elliottii* wood.

According to the treatments used, Figure 1B shows that the apparent specific mass presents an extremely small variation of 12%, which was not statistically significant. This indicates that the temperature increase did not cause a proportional mass decrease. This trend was also observed in the study by Taraborelli et al. (2022), who found no significant difference in specific mass when thermally treating *Pinus elliottii* wood.

As can be seen, there was a marked mass loss in the final treatment at 240°C, probably due to the gradual and increasing mass loss as the temperature of the treatment's increases (PENG et al., 2022). This loss can be explained by the fact that hemicelluloses are more thermally unstable, decomposing first due to their low molecular weight and branched structure compared to cellulose and lignin (BARLOVIĆ et al., 2022; WANG et al., 2022).

Cellulose exhibited greater thermal stability as the temperature increased, which explains the gradual decrease in mass due to the treatments. After thermal treatment, the amount of cellulose, hemicellulose, and extractives decreased in different proportions (AKYUREK et al., 2021). When the treatments are compared to the control group, a gradual and continuous mass loss is observed, with approximate decreases of 3.73%, 7.43%, and 15.43%,

corroborating the findings of Taraborelli et al. (2022), who also confirmed mass loss at high temperatures.

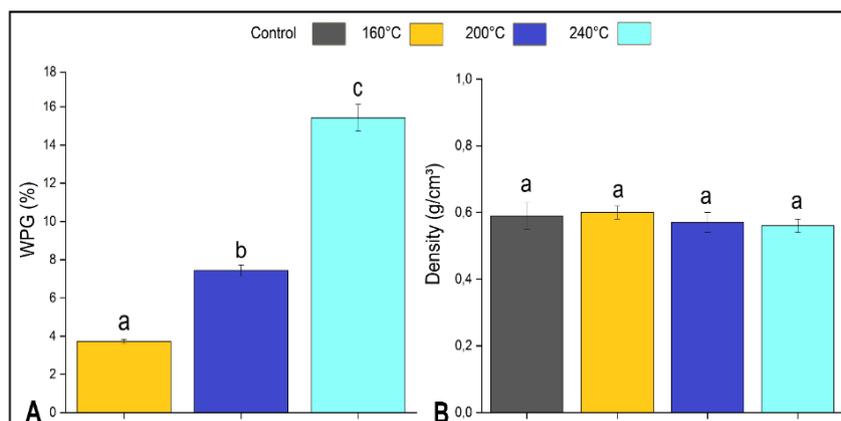


FIGURE 1 - Comparison of mass loss according to the treatments used (A). Apparent density at 12% of the treated wood in relation to the control (B).

Figure 2 shows the infrared spectra for the studied woods. Compared to untreated wood, treated samples showed a reduction in the prominent peaks at 1030, 1250, 1510, and 1650 cm^{-1} corresponding to lignin units, syringyl lignin, cellulose, hemicelluloses, and hydroxyl groups, respectively (BÁDER et al., 2020). The reduction in bands at 890 and 1030 cm^{-1} is likely due to the degradation of the

major wood components, related to the strong absorption of -C-O- present in polysaccharides, with 890 cm^{-1} (C-H deformation of glucose in cellulose) and 1030 cm^{-1} (C-O stretching of primary alcohols in cellulose and hemicelluloses), with the control peak being more prominent in the wood and characteristic of any species (TIMAR et al., 2016).

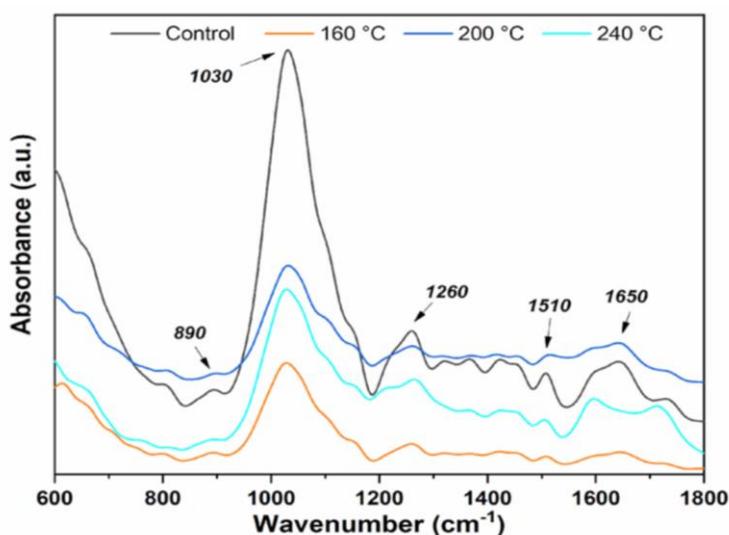


FIGURE 2 - Infrared spectra for the untreated and treated pine woods.

Reductions in the bands at 1260 cm^{-1} are partially related to acetyl and carboxyl vibrations in xylans, C-O stretching in lignin and xylan, and syringyl ring, while the band at 1510 cm^{-1} corresponds to the aromatic ring vibrations of the phenylpropane skeleton (aromatic skeletal vibration of lignin) and at 1650 cm^{-1} to conjugated carbonyl groups, possibly from ortho-quinone structures resulting from lignin modification (C=O and C=C conjugated, aromatic ketones, for example, in the side chain position of lignin units) (POZO et al., 2016).

The thermogravimetric curves shown in Figure 3 represent treated and untreated pine wood. The thermal

behaviours of the studied samples are characterised by three regions: (I) loss of intra- and intercellular moisture (40-150°C), followed by (II) thermal degradation of hemicelluloses (150-380°C), and then region (III) of cellulose (above 380°C) (ACOSTA et al., 2020a).

The wood exhibited greater degradation in region III, likely due to holocellulose (hemicellulose + cellulose), which is the least thermally stable main component (BARBOSA et al., 2022). Wood treated at 200°C and 240°C showed greater thermal stability compared to the 160°C treatment and the control (Figure 3A). At 400°C, the 200°C and 240°C treatments exhibited 57% and 54% mass loss,

respectively, which is 10% less loss than the other treatments.

Regarding the first derivative of thermogravimetry (DTG), a degradation curve for the treatments was observed around 378°C (Figure 3B), representing an 8% increase compared to the curve for other fast-growing species, such

as *Pinus elliottii*, which has a peak degradation temperature at 350°C (ACOSTA et al., 2020b). This trend likely occurred due to increased thermal stability caused by the greater degradation of hemicelluloses after thermal treatment (MISSIO et al., 2015).

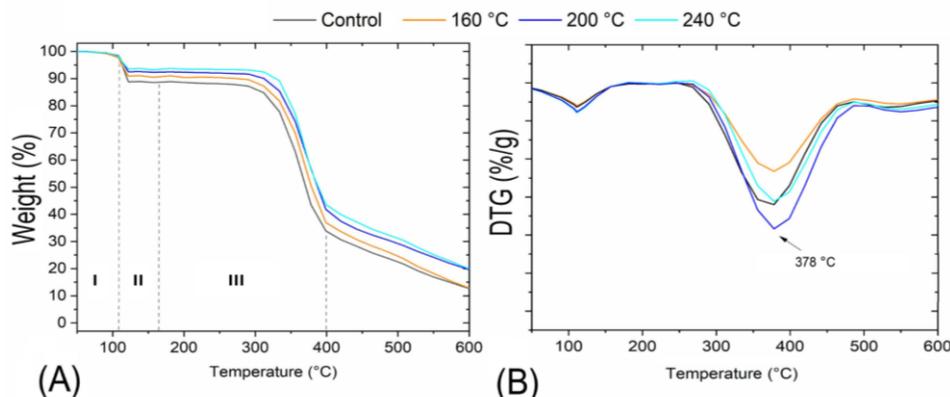


FIGURE 3 - Thermogravimetric curves (TG) and the derivative of the thermogravimetric curves (DTG) of untreated and treated pine wood.

According to the results presented in Table 1 for mechanical properties, there is a noticeable influence of increased temperature on the decrease in values for mechanical tests, particularly in the modulus of rupture, modulus of elasticity, and toughness, as the treatment temperature rises.

From the static bending test, values for the modulus of rupture (MOR) and modulus of elasticity (MOE) were obtained. It was found that the MOR and MOE values were higher with the 160°C treatment compared to

the control group (untreated), showing an increase of 7.4% and 2.6%, respectively. This increase in mechanical properties was also observed by Sharma et al. (2022), who explained this increase. However, treatments at higher temperatures resulted in a decrease in mechanical property values. For the 200°C treatment, there was a reduction of 24.7% in MOR and 2.8% in MOE. At 240°C, the reduction was 48.9% in MOR and 20.3% in MOE. This loss of MOR and MOE properties was also evidenced in the studies by Esteves et al. (2021) and Gao et al. (2022).

TABLE 1 - Descriptive statistics in relation to the modulus of rupture (MOR), modulus of elasticity (MOE) and for the tenacity of the wood studied, in different treatments.

Treatment (°C)	MOR (MPa)	MOE (MPa)	Toughness (Kgf cm ⁻²)
Control (no temperature)	84.50 ^(5.3) b*	11265.5 ^(1396.4) a	234.5 ^(36.2) d
160	90.81 ^(10.2) c	11569.1 ^(1114.2) b	163.2 ^(22.6) c
200	63.58 ^(4.3) a	10944.5 ^(762.3) b	110.1 ^(28.5) b
240	43.12 ^(9.9) a	8973.35 ^(751.6) b	46.5 ^(13.17) a

*Values in parentheses and superscripts represent the standard deviation and the means, in the columns, followed by the same letter, do not differ significantly from each other at 5% of probability of error.

In the dynamic bending test, a significant difference was observed between the treatments used, where all treatments showed a decrease in mechanical resistance compared to the control group. The 160°C treatment exhibited a 30.4% decrease, the 200°C treatment showed a 53% reduction, and the 220°C treatment had an 80.2% reduction, consistent with the findings of Boonstra et al. (2007) in their study on the thermal modification of pine species.

The high difference in toughness between the treated samples and the control group can be attributed to changes in how the chemical constituents are bonded, such as the breaking of covalent bonds between hemicellulose and lignin or microfibrils and cellulose fibres, caused by the increase in the amount of crystalline cellulose due to the

degradation and/or crystallisation of amorphous cellulose with rising temperature. This explains the increase in crystalline cellulose, which consequently increases the rigidity of cellulose fibrils, contributing to brittleness upon direct impact on the wood (SIKORA et al., 2022; PIERNIK et al., 2022). This difference in toughness between the treated samples and the control group aligns with what was observed by Davis and Thompson (1964), where the degradation of hemicelluloses is directly linked to reduced toughness.

Heat treatment also affected the wood colours, as demonstrated in Table 2 with decreases in L*, b*, C, and H° (Figure 4). A progressive increase in colour variation is observed with rising temperature, regardless of the anatomical plane observed. The reduction in brightness

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occurs due to the various chemical changes caused by the heat treatment process and the degradation of some chemical constituents of the wood (BONFATTI JÚNIOR and LENGOWSKI, 2018).

For the green-red axis (a*), the treatments employed resulted in an increase in values, both for the

tangential plane with 17.8% (160°C), 88% (200°C), and 10.1% (220°C), and for the radial plane with 6% (160°C), 98.6% (200°C), and 29.3% (220°C), all relative to the control group. This increase indicates changes in the chemical components of the wood, giving it a more reddish hue, as reported by Chen et al. (2012).

TABLE 2 - Descriptive statistics for colorimetric parameters in the tangential and radial plane of *Pinus elliottii* wood at different temperature ranges.

Tangential Plane					
Treatment	L*	a*	b*	c*	H (°)
Control	74,03 ^(3,2) d	6,01 ^(1,1) a	26,23 ^(1,7) bc	26,93 ^(1,8) b	77,03 ^(1,7) cd
Temperature (°C)					
160	68,43 ^(6,3) c	7,08 ^(1,8) b	27,39 ^(3,6) c	28,30 ^(3,9) bc	75,94 ^(1,7) c
200	45,13 ^(3,5) b	11,30 ^(0,1) c	25,51 ^(1,3) b	27,59 ^(0,7) bc	66,77 ^(1,6) b
240	27,24 ^(1,4) a	6,62 ^(0,8) ab	11,76 ^(1,7) a	13,46 ^(1,9) a	61,27 ^(0,8) a
Radial Plane					
Treatment	L*	a*	b*	c*	H (°)
Control	80,08 ^(2,1) d	5,01 ^(0,7) a	23,08 ^(1,1) b	23,66 ^(1,2) b	77,50 ^(1,7) c
Temperature (°C)					
160	75,86 ^(1,5) c	5,31 ^(0,4) a	24,01 ^(1,3) b	24,57 ^(1,3) b	77,57 ^(0,8) c
200	53,94 ^(2,1) b	9,95 ^(0,1) c	26,77 ^(0,7) bc	26,77 ^(0,7) bc	68,28 ^(1,6) b
240	34,05 ^(0,7) a	6,48 ^(0,5) b	12,63 ^(1,1) a	12,63 ^(1,1) a	59,08 ^(0,8) a

Where: values in parentheses and superscript represent the standard deviation and the means, in the columns, followed by the same letter do not differ significantly from each other at 1% of error probability by Tukey test; Where L* - clarity; a* - red-green coordinate or red matrix; b* - blue-yellow coordinate or yellow matrix; c* - colour saturation; h* - tint angle.

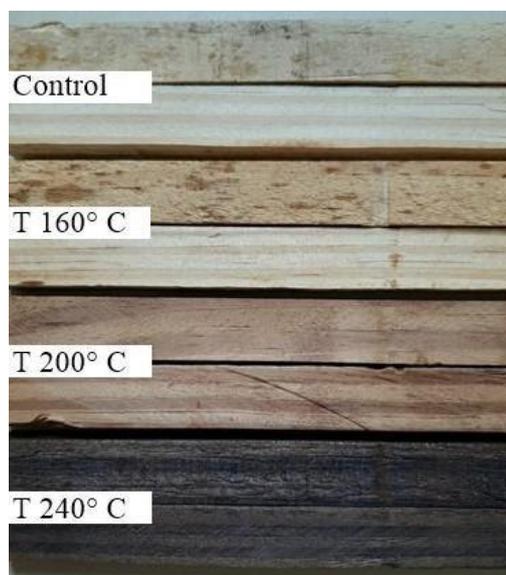


FIGURE 4 - Photographs of heat-treated and untreated *Pinus elliotti* samples.

For the chromatic coordinate (b*), the temperature of 160°C showed a tendency for an increase both for the tangential (4.4%) and radial (4%) planes. At 200°C, there was a reduction for the tangential (2.7%) and an increase for the radial (16%), while at 240°C, both the tangential and radial planes experienced reductions of 55.1% and 45%, respectively, compared to the control. According to Mesquita et al. (2020), higher values of the b parameter indicate a greater presence of yellow colour. The reductions in values are related to the degradation of lignin and extractives, components that give the wood a lighter tone (SCHULZ et al., 2020).

The chromatic saturation (C*) variables reflect the purity of the colour relative to white. Analysis of this variable shows that 240°C stands out with a more significant difference compared to the others, with a reduction of 50% for the tangential and 46.6% for the radial. The saturation values show a reduction in dark grey tones characteristic of heat exposure. These values are similar to the behaviour of the b* matrix, as they are higher than the a* matrix, in line with findings by Zanuncio et al. (2014) and Cademartori et al. (2013).

The hue angle (H°) results from the red (a*) and yellow (b*) matrices. The values for this variable show a

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more significant reduction at 240°C, with 20.4% for the tangential plane and 23.8% for the radial. It is observed that due to the treatment applied, the red hue (related to the a* matrix) increases while the yellow hue (related to the b* matrix) decreases, which is characteristic of the control group. This helps to explain the darkening of the wood, as the hue angle h° approaches the green-red coordinate (b*), according to Schulz et al. (2020).

According to Acosta et al. (2022), darkening due to heat treatment is advantageous because it makes *Pinus* wood (originally pale in colour) resemble more valuable native Brazilian woods, such as jacaranda (*Jacaranda mimosifolia*), jatoba (*Hymenaea courbaril*), mahogany (*Swietenia macrophylla*), and cumaru (*Dipteryx odorata*).

In light of this, the importance of studies on thermal treatments to improve the technological properties of wood is evident, allowing for diversification of use and expansion of commercial potential, solely through heat exposure without chemical additives, aiming to explore the internal reactivity of the wood through softening and redistribution of lignin components, loss of acidic groups, and the cross-linking and repolymerisation that occur to varying degrees without the need for added reagents for this interaction.

CONCLUSIONS

The treatments studied showed distinct results regarding the stiffness and strength of the wood. The treatment at 160°C resulted in an increase in stiffness and strength compared to the modifications at 200°C and 240°C. On the other hand, the 200°C treatment provided greater thermal stability, approximately 10%. Therefore, both the 160°C and 200°C treatments are recommended, as they offer increased mechanical stiffness and thermal stability, respectively. However, the 240°C treatment was deemed unsuitable due to the significant damage it caused to the wood properties. These results indicate that the choice of the appropriate thermal treatment for *Pinus elliottii* wood may depend on the specific goals concerning mechanical properties and thermal stability.

After analysing the thermal modification study on *Pinus elliottii* wood, it was observed that there was no significant reduction in apparent density. However, in the mass loss analysis, a decrease was noted as the treatment temperature increased, which is related to the degradation of hemicelluloses, celluloses, and lignin as temperature rises. Regarding the colourimetric analysis, the use of thermal treatments resulted in the darkening of the wood.

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