

Article

Not peer-reviewed version

Enhanced Properties of *Cryptomeria japonica* from the Azores Through Heat-Treatment

[Bruno Esteves](#)*, [Lina Nunes](#), [Rogério Lopes](#), [Luísa Cruz-Lopes](#)

Posted Date: 20 December 2024

doi: 10.20944/preprints202412.1752.v1

Keywords: Chemical composition; *Cryptomeria japonica*; Dimensional stability; Heat treatment; Mechanical properties; Termite resistance



Preprints.org is a free multidisciplinary platform providing preprint service that is dedicated to making early versions of research outputs permanently available and citable. Preprints posted at Preprints.org appear in Web of Science, Crossref, Google Scholar, Scilit, Europe PMC.

Copyright: This open access article is published under a Creative Commons CC BY 4.0 license, which permit the free download, distribution, and reuse, provided that the author and preprint are cited in any reuse.

Disclaimer/Publisher's Note: The statements, opinions, and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions, or products referred to in the content.

Article

Enhanced Properties of *Cryptomeria japonica* from the Azores Through Heat-Treatment

Bruno Esteves ^{1,2,*}, Lina Nunes ³, Rogério Lopes ^{4,5} and Luísa Cruz-Lopes ^{1,6}

¹ CERNAS (Centre for Natural Resources, Environment and Society)-IPV Research Centre, Polytechnic University of Viseu, Av. Cor. José Maria Vale de Andrade, 3504-510 Viseu, Portugal

² Department of Wood Engineering, Polytechnic University of Viseu, Av. Cor. José Maria Vale de Andrade, 3504-510 Viseu, Portugal

³ Structures Department, LNEC, National Laboratory for Civil Engineering, Av. do Brasil, 101, 1700-066 Lisbon, Portugal

⁴ CISEd – Research Centre in Digital Services, Instituto Politécnico de Viseu, Portugal, Polytechnic University of Viseu, Av. Cor. José Maria Vale de Andrade, 3504-510 Viseu, Portugal

⁵ Department of Mechanical Engineering and Industrial Management, Polytechnic Institute of Viseu, Viseu, Portugal

⁶ Department of Environmental Engineering, Polytechnic University of Viseu, Av. Cor. José Maria Vale de Andrade, 3504-510 Viseu, Portugal

* Correspondence: bruno@estgv.ipv.pt (B.E.)

Abstract: This study evaluates the chemical, physical, mechanical, and biological properties of untreated and heat-treated *Cryptomeria japonica* wood from the Azores, Portugal. Heat treatment was performed at 212°C for 2 hours following the Thermo-D class protocol. Chemical analysis revealed an increase in ethanol extractives and lignin content after heat treatment, attributed to hemicellulose degradation and condensation reactions. Dimensional stability improved significantly, as indicated by reduced swelling coefficients and higher anti-swelling efficiency (ASE), particularly in the tangential direction. Heat-treated wood demonstrated reduced water absorption and increased density, enhancing its suitability for applications requiring dimensional stability. Mechanical tests showed a decrease in bending strength by 19.6% but an increase in the modulus of elasticity (MOE) by 49%, reflecting changes in the wood's structural integrity. Surface analysis revealed significant color changes, with darkening, reddening and yellowing, aligning with trends observed in other heat-treated woods. Biological durability tests indicated that both untreated and treated samples were susceptible to subterranean termite attack, although heat-treated wood exhibited a higher termite mortality rate, suggesting potential long-term advantages. This study highlights the impact of heat treatment on *Cryptomeria japonica* wood, emphasizing its potential for enhanced stability and durability in various applications.

Keywords: chemical composition; *Cryptomeria japonica*; dimensional stability; heat treatment; mechanical properties; termite resistance

1. Introduction

Commonly referred to as Japanese cedar or Sugi, *Cryptomeria japonica*, is a superb evergreen tree deeply rooted in Japan's natural and cultural heritage [1]. Renowned for its symmetrical form and towering height, it is a dominant feature of Japanese landscapes, often reaching heights of 50 meters (164 feet) or more in its native habitat. With a straight, robust trunk and dense foliage composed of spirally arranged, needle-like leaves, *Cryptomeria japonica* creates a lush canopy that provides essential shelter for diverse flora and fauna. This adaptability has made it a staple in various environments, thriving in both humid coastal regions and rugged mountainous terrains [2,3].

Beyond its ecological significance, *Cryptomeria japonica* is also a cultural symbol. In Japan, it is frequently planted in temple gardens, sacred groves, and along shrine pathways, symbolizing

longevity and spiritual importance. The wood, known as Japanese cedar, is highly valued for its durability, fine grain, and natural resistance to decay, making it a preferred material for construction, furniture, and traditional crafts [4].

This species was introduced to the Azores archipelago (Portugal) in the mid-19th century, where it has thrived due to the region's favorable climate and volcanic soil [3]. While São Miguel Island is the primary location for *Cryptomeria japonica* plantations, it has also been cultivated on other islands, including Terceira, Faial, and Pico. In the Azores, *Cryptomeria japonica* has become an essential part of the local forestry industry, contributing to timber production and ecological conservation efforts [5].

The leaf essential oil of *Cryptomeria japonica* has demonstrated antitermitic activity against *Coptotermes formosanus* Shiraki [6]. Terpenes and terpenoids are the major components of this essential oil, which contribute to the wood's natural resistance to termites. Compounds like cubebol, epicubebol, sandaracopimarinol, and ferruginol with antitermitic activities were also found in the heartwood of *Cryptomeria* trees grown in Japan [7]. This intrinsic property makes untreated *Cryptomeria* wood somewhat resistant to biological degradation. Despite its many advantages, the main challenge of *Cryptomeria* wood lies in its high dimensional instability. The wood tends to expand, contract, or warp due to changes in moisture content, limiting its application in demanding environments.

Heat treatment is considered to be an environmentally friendly method that improves dimensional stability, durability, and resistance to biological degradation without the use of harmful chemicals [8]. Established processes like Thermowood, Plato Wood, and Bois Perdure have laid the groundwork for new techniques, including vacuum-based methods (e.g., Termovuoto®, Moldrup-SSP®, TanWood®) and superheated steam processes (e.g., ThermoTreat 2.0®, Firmolin®) [9]. During heat treatment, wood is exposed to elevated temperatures (160 °C–260 °C) in an oxygen-poor environment, often with water vapor acting as a shielding gas. This controlled process prevents combustion, reduces oxidation, and facilitates chemical transformations in the wood's structure. Components like hemicellulose, cellulose, and lignin undergo changes that result in reduced moisture absorption, enhanced resistance to decay, and a darker appearance [10–13].

By addressing the dimensional instability of *Cryptomeria japonica*, heat-treatment unlocks its potential for high-demand applications such as outdoor furniture, decking, and structural components. This process aligns with sustainable forestry practices, eliminates the need for chemical additives, and enhances the global appeal of *Cryptomeria* as a versatile and eco-friendly resource.

2. Materials and Methods

Sapwood samples from *Cryptomeria japonica* originated from Azores region (Portugal) were used for the tests. Wood was treated in a Portuguese company known as Palser at 212 °C for 2h in accordance to Thermo D class from Thermowood®.

Chemical Composition

Samples for chemical analysis were prepared by first cutting the sample into pieces of approximately 1 cm³ using a chisel and hammer, rejecting all bark. After natural drying in air and room temperature, the samples were crushed in a Retsch SMI mill. It was then screened in a Retsch 5657 HAAN type 1 vibrating screen for half an hour to obtain the following fractions: >40 mesh (>0.425 mm), 40-60 mesh (0.425-0.250 mm), 60-80 mesh (0.250-0.180 mm) and 80 mesh (<0.180 mm) for 30 minutes.

Extract content was determined by sequential solvent extraction. In order to do this, 10 g (±0.0001 g) of sample (40-60 mesh) was placed in a Soxhlet extractor and extracted with solvents of increasing polarity: dichloromethane (6 h), ethanol (16 h) and hot water (16 h). The extract content (E) was determined in relation to dry mass.

For determination of lignin content by the Klason method, approximately 350 mg per sample was used from extract-free samples as described in the standard TAPPI T222 om-02 [14], which quantifies lignin as solid residue. The method is based on two hydrolysis steps. The first was with

72% sulfuric acid at 30 °C for 1 hour, and the second at 120 °C in an autoclave for 1 hour. After hydrolysis, samples were stored in pre-weighed and filtered with N°4 crucibles, rinse with warm water and dried in an oven at 60 °C overnight and then at 100 °C for 1 hour. Insoluble lignin content (L) was determined in relation to dry mass.

The holocellulose content of extracted wood was determined using the acid chlorite method, which determines holocellulose as an insoluble residue. The method consisted of dissolving 2 g of wood without extractives in a 1 L flask, adding 160 mL of 70 °C distilled water, 20 mL of a solution containing 8.5 g of sodium chlorite in 250 mL of distilled water and a further 20 mL of a solution with 13.5 g of NaOH in 50 mL of distilled water and 37.5 g of acetic acid. A total of 20 mL of each solution was added until the sample turned white, which may take up to 8 h [13]. The sample was then filtered in N°2 crucibles and washed with cold water and 15 mL acetone. The holocellulose (HC) content was also determined in relation to dry wood.

To determine the α -cellulose content, dry holocellulose was hydrolyzed with 17.5% NaOH at 20 °C for 30 minutes, rested outside the bath for 15 minutes and then adding 8.25 mL of water to the sample which was kept in a heat bath at 20 °C for another hour. The sample was then filtered and washed with 25 mL of 8.3% NaOH and distilled water, and finally with 3.75 mL of 10% acetic acid. The sample was dried at 105 °C overnight, and the α -cellulose content was determined in relation to dry mass. The hemicellulose content was determined indirectly by the difference between corrected holocellulose and α -cellulose [15].

Physical and Mechanical Properties

The oven-dry wood density was determined at 100 °C and by weighing a cube sample with 20 mm edges and measuring the wood dimensions in the three directions. An average of 10 repetitions were used.

The dimensional stability of untreated and treated specimens along tangential, radial and axial directions was evaluated over three cycles of wet (100%) and dry (0%) conditions using the swelling coefficient (S) and anti-swelling efficiency (ASE), calculated using Equations (1) and (2), respectively.

Swelling Coefficient (S):

$$S (\%) = \frac{L_{(100\%)} - L_{(0\%)}}{L_{(0\%)}} \times 100 \quad (1)$$

where:

$L_{100\%}$ = dimension of the saturated specimen (mm) after each cycle,

$L_{0\%}$ = dimension of the dried specimen (mm) after each cycle.

$$ASE (\%) = \frac{S_u - S_{ht}}{S_u} \times 100 \quad (2)$$

where:

S_u = swelling coefficient of the untreated specimen,

S_{ht} = swelling coefficient of the heat-treated specimen, measured after each cycle.

Water absorption was evaluated in wooden cubic samples with approximate edge lengths of 20 mm. The assessment involved three consecutive cycles of conditioning at 0% equilibrium moisture content (EMC), achieved by drying the samples in an oven at 100 °C, followed by exposure to 100% EMC through immersion in water at 20 °C.

The bending strength and stiffness of wooden specimens were determined by a three-point bending tests with longitudinal, radial and tangential dimensions of 360 mm × 20 mm × 20 mm according to Portuguese standard NP-619 [16] on a universal testing machine (Servosis ME-405/5). Each test was replicated ten times.

For the mechanical tests, the samples were maintained at 20 °C and 65% relative humidity before testing. During the test, the radial surface of the sample faced upward and was placed on two supports spaced 300 mm apart. The carrier was moved until the pulley contacted the sample without applying force. The test was conducted at a constant speed of 3 mm·min⁻¹.

The modulus of elasticity (MOE) was calculated using the formula from EN 310 [17]:

$$MOE (MPa) = \frac{\Delta F \cdot L^3}{4 \cdot \Delta x \cdot b \cdot h^3} \cdot 9.8 \quad (3)$$

where:

$\Delta F/\Delta x$ represents the elastic strain ($\text{kg}\cdot\text{mm}^{-1}$), L is the span length between supports (mm), b is the sample width (mm), and h is the sample height (mm).

The bending strength tests were performed using the same machine and setup as described above. The average test speed was determined as the time required for braking, approximately 2 minutes after the test began. Bending strength was calculated using the formula:

$$\text{Bending strength (MPa)} = \frac{F_f \cdot 3 \cdot L}{2 \cdot b \cdot h^2} \cdot 9.8 \quad (4)$$

where F_f is the maximum load (kg).

For each test, ten replicates were conducted to ensure reliability.

Surface Properties

Color parameters were determined in the tangential section. The 0% color was calibrated with white standards and the 100% color with black standards using a Minolta cm-3630 color spectrophotometer. The CIELAB technique was used to determine the color parameters L^* , a^* , and b^* for untreated and treated wood. Each result is the mean of three replicates for untreated and heat-treated wood.

Termite Durability

Wood samples were subjected to subterranean termite attack under controlled conditions at UPB.LNEC, following the EN 118:2013 standard [18]. Termites were confined within glass tubes containing a substrate to support colony development. The samples were exposed for 8 weeks at a temperature of 24–26 °C and relative humidity above 75%. Daily inspections were conducted to monitor termite activity, such as gallery openings and chimney-like structures, as well as to ensure appropriate substrate moisture and record any mold growth. Termite attack was visually evaluated and classified according to the EN 118:2013 grading system. Grade 0 indicates no attack, while Grade 1 corresponds to superficial erosion. Grade 2 represents a light attack with limited erosion or a single tunnel. Grade 3 signifies a moderate attack, characterized by widespread shallow erosion or an isolated deep tunnel. Grade 4 indicates a severe attack, with deep erosion or extensive cavities.

3. Results and Discussion

The chemical composition of untreated and heat-treated *Cryptomeria japonica* wood is detailed in Table 1 and Figure 1. In untreated *Cryptomeria* wood, extractives are primarily composed of ethanol extractives (1.3%), followed by water extractives (0.99%) and dichloromethane extractives (0.6%). These findings are consistent with previous research by Fonte et al. [19], who reported 1.81% water extractives and 1.23% ethanol-toluene extractives on sapwood of *Cryptomeria* wood sourced from experimental plantings at the Experimental Station of the Federal University of Paraná in Rio Negro, Brazil. Yinodotlgör and Kartal [20] documented 1.36% cyclohexane/ethanol extractives, 0.49% ethanol extractives, and 2.78% water extractives in their analysis but it is well known that the composition and amount of extractives depends on the region.

Table 1. Extractives of untreated and heat-treated *Cryptomeria* wood.

Sample	Extractives (%)											
	Dichloromethane			Ethanol			Water			Total		
Untreated	0.60	±	0.00	1.30	±	0.43	0.99	±	0.11	2.89	±	0.44
Heat-treated	0.55	±	0.04	2.05	±	0.13	0.99	±	0.24	3.59	±	0.27

Lipophilic extractives from cryptomeria wood were reported to be mainly terpenes, such as diterpenes, sesquiterpenes, triterpenes and sesquiterpenes and polyphenols like flavonoids, lignans, norlignans and anthraquinones [21].

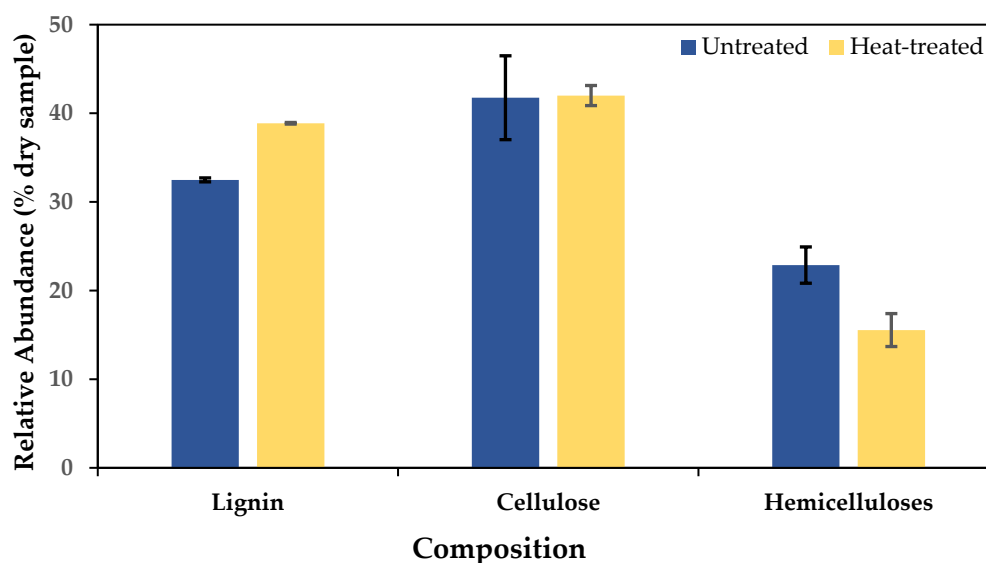


Figure 1. Chemical composition of untreated and heat-treated *Cryptomeria* wood.

Heat treatment appears to induce a slight increase in ethanol extractives from 1.3% to 2.0%, a trend observed in other wood species such as heat-treated *Paulownia* [22], *Eucalyptus globulus* [23], and *Pinus pinaster* [24]. This increase in ethanol extractives is likely due to the thermal degradation of hemicelluloses and the subsequent formation of low-molecular-weight compounds, which are more soluble in ethanol. However, this trend is not universal. For instance, in thermally modified tropical hardwoods like *Afromosia* (*Pericopsis elata*) and *Duka* (*Tapirira guianensis*), no significant increase in ethanol extractives was observed. This discrepancy may be attributed to the already higher ethanol extractives content in the untreated wood of these species, which reduces the potential for further increase during heat treatment since several of the original compounds are degraded [25]. The changes in extractive content during heat treatment play a critical role in determining the wood's physical and chemical properties, including its resistance to decay, dimensional stability, and overall durability. Understanding these variations helps optimize heat treatment processes for specific wood species and applications.

A slight reduction in dichloromethane extractives and no change in water extractives were also observed. Nevertheless, it is expected that the composition of heat-treated wood extractives differs from untreated wood.

Cryptomeria japonica has been known to possess some extractives with antithermic properties, comprising a complex mixture of chemical compounds that have been studied as potential natural termiticidal agents [6,7]. Recent investigations have sought to identify and characterize the specific extractives responsible for its termite-repelling and toxic properties, shedding light on their mechanisms of action and potential applications in eco-friendly pest control solutions [26]. Despite these extractives, that depend on the origin of the wood, the species remains consistently susceptible to some level of termite attack. One of the main questions is whether heat treatment degrades termiticidal compounds, negatively affecting the wood's resistance to subterranean termites.

Untreated *cryptomeria* wood has a high amount of lignin (32.5%), while it has around 41.8% Cellulose and 22.9% hemicelluloses. The *cryptomeria* from Brazil mentioned before has a similar amount of lignin, around 35.0%, same as the one reported by Yinodotlgör and Kartal [20] with 35.7%.

With heat-treatment the percentage of lignin increases to around 38.9% likely due to the greater degradation of hemicelluloses, which decrease from 22.9% to 15.5% but also to the condensation

reactions between lignin and degradation compounds from polysaccharides has stated before for Norway spruce, beech wood or maritime pine [27–29]. No significant difference is observed in cellulose percentage which can be due to the equilibrium between some degradation of amorphous regions and the higher degradation of hemicelluloses.

Physical and Mechanical Properties

The main objective of heat-treatment is to improve the dimensional stability of wood. Figure 2 compares dimensional changes (radial, tangential, and axial) over multiple cycles (three cycles) for untreated and heat-treated wood.

In the tangential direction, untreated wood experiences significant swelling, with values like 8.48%, 7.65%, and 7.16% in cycles 1, 2, and 3. This highlights the natural tendency of untreated wood to expand tangentially due to moisture. Heat-treated wood, however, has much lower tangential changes, with values like 5.38%, 5.37%, and 5.42%, indicating reduced swelling and improved stability. No significant differences were observed between cycles for both untreated and heat-treated wood showing that the improvements still remain after three wet and dry cycles.

For the radial direction, untreated wood has also higher swellings with values such as 3.73%, 3.72%, and 3.98% in cycles 1, 2, and 3, indicating instability. In contrast, heat-treated wood has lower values, such as 2.67%, 2.78%, and 3.26%, with smaller swellings, showing greater stability.

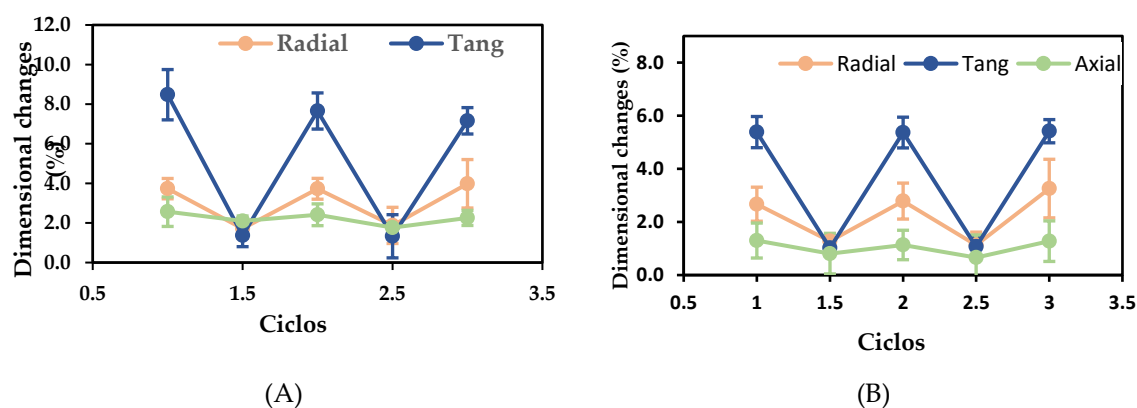


Figure 2. Dimensional stability of untreated (A) and heat-treated (B) cryptomeria wood.

In the axial direction, untreated wood shows moderate instability, with values fluctuating between 2.57% and 1.76%. Heat-treated wood, on the other hand, has lower axial changes, such as 1.30%, 1.28%, and 0.66%, showing some improvement in stability. Overall, untreated wood exhibits larger dimensional changes, especially in the tangential direction, where it is most affected by moisture. The lower values and reduced fluctuations confirm that heat treatment reduces moisture absorption and minimizes swelling and shrinking, particularly in the tangential direction.

In order to best evaluate the improvements in dimensional stability, the ASE was determined to show how well the treatment reduces dimensional changes (radial, tangential, and axial) over different cycles, Figure 3.

In the radial direction, ASE starts at 28.53% in the first cycle and slightly decreases to 25.23% in the second cycle. By the third cycle, it drops further to 18.15%, indicating a gradual decline in the treatment's effectiveness in reducing radial swelling over time. Similarly, for the tangential direction, ASE begins at 36.51% in the first cycle, decreases to 29.90% in the second cycle, and drops further to 24.34% in the third cycle. In the axial direction, ASE ranged from 49.42% to 43.37% but it has to be taken into account that in this direction the swelling is minimal. Even though there is a reduction on ASE along the wet and dry cycles heat-treated wood still performs significantly better than untreated wood. Yang et al [30] determined the ASE for heat-treated *Cryptomeria* wood at temperatures ranging from 170 to 210 °C but without using a shielding gas. ASE ranged from 20% to 54%. The higher ASE observed for 210 °C during 4h (54%) is obtained with 18% mass loss which is much higher

than the commonly accepted 3% for heat-treatments due to the higher degradation of wood. Heat treatment of Rubberwood and Silver Oak was studied by Srinivas and Pandey[31] that reported a volumetric ASE of 20% and 12% for wood treated at 210 °C for 4h. To compare with the results presented here the volumetric ASE was determined for the first cycle to be 34%, which is significantly better than the reported before.

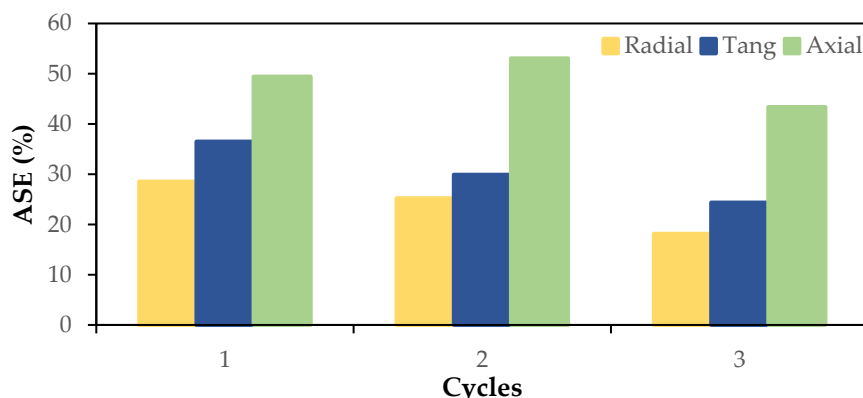


Figure 3. Anti swelling efficiency of untreated and heat-treated *Cryptomeria* wood.

The water absorption data for untreated and heat-treated *Cryptomeria japonica* wood across three cycles is presented on Table 2. Results show that there is a significant difference in the behavior of untreated and heat-treated wood in response to moisture exposure. These results highlight the impact of heat treatment on the wood's ability to absorb water, an important factor in its dimensional stability and durability.

In the first cycle, untreated wood exhibited an average water absorption of 160%. This significant variability indicates that untreated wood is highly susceptible to water uptake, likely due to its porous structure and hydrophilic nature. In contrast, heat-treated wood absorbed only 130%. This reduction in water absorption can be attributed to the heat treatment process, which typically causes the wood's cell wall structure to undergo chemical and physical changes. In the second cycle, untreated wood showed a similar water absorption of 160%. Heat-treated wood's absorption decreased further to 113%, highlighting the lasting benefits of heat treatment in water absorption. In the last cycle heat-treated wood absorbed 125% maintaining its water absorption in the same levels.

Table 2. Water absorption (%) of untreated and heat-treated *Cryptomeria* wood.

Cycle	Treatment	Average	Std
1st cycle	Untreated	160.409	42.202
	Heat-treated	130.439	0.0768
2nd cycle	Untreated	160.685	31.031
	Heat-treated	113.143	22.605
3rd cycle	Untreated	157.167	20.274
	Heat-treated	125.148	17.336

The results from the three cycles demonstrate the significant impact of heat treatment on the water absorption behavior of *Cryptomeria japonica* wood. These findings align with previous research, which has demonstrated that heat treatment reduces the water absorption of wood. Results presented before for fast growing hardwoods showed that heat treatment decreased water absorption in jabor, sengon, and mangium wood samples. The control samples of jabor, sengon, and mangium had

average water absorption rates of 43%, 40%, and 15%, respectively. Following heat treatment, water absorption decreased by 34% in jabon, 30% in sengon, and 49% in mangium [32]. The same happened for radiata pine, a known softwood [33]. Nevertheless, results presented before show that heat treatment not always decreases water absorption. For example heat treatment affected differently the water absorption of Scots pine and Norway spruce [34]. Pine sapwood absorbed water more quickly than heartwood, and heat treatment increased water absorption in pine sapwood. However, heat treatment reduced water absorption in pine heartwood and spruce, with the reduction being proportional to the treatment temperature.

Wood density can vary significantly depending on the geographic location, climate, and species. Trees grown in regions with favorable conditions, such as consistent rainfall and moderate temperatures, often exhibit higher wood density due to slower, more uniform growth. In contrast, wood from harsher environments, such as arid or cold regions, may have lower density as a result of faster or irregular growth patterns. This variation in density directly influences the mechanical properties of wood, such as strength, stiffness, and durability. Higher-density wood typically possesses greater mechanical strength and resistance to stress, making it more suitable for structural applications, while lower-density wood may be lighter but less robust, limiting its use in load-bearing scenarios. Untreated cryptomeria wood used in the tests presented a oven-dry density around 0.27 g/cm³ which is slightly smaller than the 0.33 g/cm³ presented by logs obtained from Ibaraki Prefecture in Japan [35]. Density increased for heat treated wood from 0.27 g/cm³ to 0.33g/cm³ and, even though it was a small increase it might have protected wood from a higher decrease on mechanical properties.

Mechanical strength is known to decrease due to heat-treatment, specially bending strength [36,37]. The MOE is often observed to increase at the beginning of treatment, followed by a decrease in more severe treatments. Table 3 shows the mechanical properties of untreated and treated wood, where the bending strength dropped from 52.6 MPa to 42.3 MPa, representing a 19.6% reduction compared to untreated wood, while the MOE increased from 7268 MPa to 10836 MPa. MOE is known to increase and then decrease for higher intensity treatments and tests made with cryptomeria wood treated at 210 °C for 2h without shielding gas lead to a 26% decrease on MOE and a 42% decrease on bending strength [30]. The greater degradation of wood's mechanical properties observed by these authors could hinder the use of treated wood in various applications.

Table 3. Mechanical properties of untreated and heat-treated Cryptomeria wood.

Sample	MOE (MPa)		Bending strength (MPa)	
	Average	Std. Dev.	Average	Std. Dev.
Untreated	7268	1194	52.6	2.4
Heat-treated	10836	771	42.5	6.0

The main changes observed on the surface of heat-treated wood were the color that became darker as can be seen by the L* decrease from 67.5 to 46.6 in heat-treated wood. There has been a significant decrease in a* and b* corresponding to the reddening and yellowing of the samples, Table 4. The changes in color due to heat-treatment have been reported to be dependent on the initial wood color. For instance, a* increased for black locust, linden and willow, but decreased for wild pear and alder while a decrease in b* followed by an increase was reported for black locust and linden, while for wild pear and alder a decrease with the intensity of the treatment was observed and in the case of willow, there was an initial increase in b* followed by a decrease [38]. ΔL was 20.9 similar to the reported before for wood treated at 210°C and 2h but without shielding gas [30].

Table 4. Color parameters of untreated and heat-treated *Cryptomeria* wood.

L*		a*		b*	
Average	Std. Dev.	Average	Std. Dev.	Average	Std. Dev.
67.5	1.2	13.6	0.5	24.5	0.4
46.6	0.8	1.4	0.3	4.2	0.2

Termite Durability

Cryptomeria is known to possess some durability against subterranean termites, especially heartwood. The resistance to termites was assessed using EN117 [39] to determine whether heat treatment enhances or reduces termite resistance. Both untreated and heat-treated samples exhibited high susceptibility to subterranean termites (Table 5). This indicates that while heat treatment may remove some antitermic extractives from the wood, it does not significantly reduce the wood's durability against termites. In the long run heat treated wood might even perform better than untreated wood due to the higher mortality rate observed.

Table 5. Untreated and treated wood resistance to termite.

Material	Survival rate [%]	Moisture content [%]	Mass loss [%]	Attack level
Untreated	40.60	56.89	8.82	3.8
Heat-treated	31.30	64.42	10.48	4.0

4. Conclusions

The study of *Cryptomeria japonica* sapwood from the Azores region (Portugal) demonstrated the effects of heat treatment at 212 °C for 2 hours, following the Thermo D class from Thermowood®, on the wood's chemical composition, physical properties, and termite resistance. The heat treatment resulted in changes to the chemical composition, including a slight increase in ethanol extractives and a reduction in hemicellulose content, while lignin content increased due to the degradation of hemicelluloses and condensation reactions.

The dimensional stability of heat-treated wood showed significant improvements, with reduced swelling and water absorption, particularly in the tangential direction, as indicated by the Anti-Swelling Efficiency (ASE) values.

Mechanical properties were impacted by the heat treatment, with a decrease in bending strength (19.6% reduction) but an increase in the modulus of elasticity (MOE). These results align with previous studies on heat-treated wood, where mechanical properties are generally compromised due to the degradation of cellulose and hemicellulose. The surface color of the wood darkened which is a typical outcome of heat treatment.

In terms of termite resistance, both untreated and heat-treated *Cryptomeria japonica* exhibited high susceptibility to subterranean termites, indicating that the heat treatment did not enhance but also did not reduce significantly the wood's natural resistance. However, it is suggested that the heat treatment might still provide some long-term benefits, possibly due to the higher mortality rate observed in termite tests.

Overall, heat treatment improves dimensional stability and reduces water absorption of *Cryptomeria japonica*, making it a more durable material for various applications.

Author Contributions: Conceptualization, B.E.; methodology, B.E. and L.C.-L.; formal analysis, B.E., L.C.-L. and L.N.; investigation, B.E., R.L. and L.C.-L.; writing—original draft preparation, B.E.; writing—review and editing, B.E., L.C.-L. and R.L.; project administration, B.E.; funding acquisition, B.E., L.C.-L. and R.L. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by National Funds through the FCT—Foundation for Science and Technology (Proj. UIDB/00681/2020 (CERNAS) DOI: <https://doi.org/10.54499/UIDB/00681/2020>, accessed on 30 November 2024 and Ref. UIDB/05583/2020. Furthermore, we would like to thank the Research Centre in Digital Services (CISeD)), and the Polytechnic University of Viseu.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are available on request from the corresponding author.

Conflicts of Interest: The authors declare no conflicts of interest. This article is a revised and expanded version of a paper entitled Heat treatment of *Cryptomeria japonica* from Azores, which was presented at [ECWM11, Firenze, 2024].

References

1. YASUE, M.; OGIYAMA, K.; SUTO, S.; TSUKAHARA, H.; MIYAHARA, F.; OHBA, K. Geographical Differentiation of Natural *Cryptomeria* Stands Analyzed by Diterpene Hydrocarbon Constituents of Individual Trees. *Journal of the Japanese Forestry Society* **1987**, *69*, 152–156.
2. *Cryptomeria Japonica* (Japanese Cedar, Japanese *Cryptomeria*) | North Carolina Extension Gardener Plant Toolbox Available online: <https://plants.ces.ncsu.edu/plants/cryptomeria-japonica/> (accessed on 13 March 2024).
3. Pavão, D.C.; Brunner, D.; Resendes, R.; Jevšenak, J.; Borges Silva, L.; Silva, L. Climatic Drivers and Tree Growth in a Key Production Species: The Case of *Cryptomeria Japonica* (Thunb. Ex L.f.) D.Don in the Azores Archipelago. *Dendrochronologia* **2024**, *85*, 126204, doi:10.1016/j.dendro.2024.126204.
4. Tsukada, M. *Cryptomeria Japonica*: Glacial Refugia and Late-Glacial and Postglacial Migration. *Ecology* **1982**, *63*, 1091–1105, doi:10.2307/1937247.
5. Almeida, M.H.; Faria, C.; Belerique, J.; Nobrega, C.; Penacho, L.; Rocheta, M. Resultados Preliminares Dos Testes Genéticos Com *Cryptomeria Japonica* Na Região Autónoma Dos Açores. **2005**.
6. Cheng, S.-S.; Lin, C.-Y.; Chung, M.-J.; Chang, S.-T. Chemical Composition and Antitermitic Activity against *Coptotermes formosanus* Shiraki of *Cryptomeria Japonica* Leaf Essential Oil. *Chemistry & Biodiversity* **2012**, *9*, 352–358, doi:10.1002/cbdv.201100243.
7. Shibutani, S.; Takata, K.; Doi, S. Quantitative Comparisons of Antitermite Extractives in Heartwood from the Same Clones of *Cryptomeria Japonica* Planted at Two Different Sites. *J Wood Sci* **2007**, *53*, 285–290, doi:10.1007/s10086-006-0866-4.
8. Esteves, B.; Pereira, H. Wood Modification by Heat Treatment: A Review. *BioResources* **2009**, *4*, 370–404.
9. Esteves, B.; Şahin, S.; Ayata, U.; Domingos, I.; Ferreira, J.; Gürleyen, L. Effect of Heat Treatment on Shore-D Hardness of Some Wood Species. *BioResources* **2021**, *16*, 1482, doi:10.15376/biores.16.1.1482-1495.
10. Nuopponen, M.; Vuorinen, T.; Jämsä, S.; Viitaniemi, P. Thermal Modifications in Softwood Studied by FT-IR and UV Resonance Raman Spectroscopies. *Journal of Wood Chemistry and Technology* **2005**, *24*, 13–26, doi:10.1081/WCT-120035941.
11. Tjeerdsma, B.F.; Boonstra, M.; Pizzi, A.; Tekely, P.; Militz, H. Characterisation of Thermally Modified Wood: Molecular Reasons for Wood Performance Improvement. *Holz als Roh-und Werkstoff* **1998**, *56*, 149–153, doi:10.1007/s001070050287.
12. Boonstra, M.J.; Tjeerdsma, B. Chemical Analysis of Heat Treated Softwoods. *Holz Roh Werkst* **2006**, *64*, 204–211, doi:10.1007/s00107-005-0078-4.
13. Tjeerdsma, B.F.; Stevens, M.; Militz, H.; Van Acker, J. Effect of Process Conditions on Moisture Content and Decay Resistance of Hydro-Thermally Treated Wood. *Holzforschung und Holzverwertung* **2002**, *54*, 94–99.
14. TAPPI T 222 Om-02. Acid-Insoluble Lignin in Wood and Pulp. *TAPPI: Atlanta, GA, USA* **2002**.
15. Mercado, Y.; Nunes, L.; Cruz Lopes, L.P.; Esteves, B. Heat Treatment of *Cryptomeria Japonica* from Azores.; n.a.: Firenze, Italy, 2024; Vol. n.a., p. n.a.
16. NP 619 Static bending test (in Portuguese). Inspeção Geral dos Produtos Agrícolas e Industriais (IGPAI). Lisbon, Portugal 1973;

17. NP EN 310:2002-Placas de Derivados de Madeira – Determinação Do Módulo de Elasticidade Em Flexão e Da Resistência à Flexão. *Instituto Português da Qualidade* 2002.
18. EN 118:2013 - Wood and Wood-Based Products - Determination of the Resistance to Subterranean Termites. EN 118:2013, 2013. *European Committee for Standardization (CEN), Brussels, Belgium.*;
19. Fonte, A.P.N.; Trianoski, R.; Iwakiri, S.; dos Anjos, R.A.M. Physical and Chemical Properties of Heartwood and Sapwood of *Cryptomeria Japonica*. *Revista de Ciências Agroveterinárias* **2017**, *16*, 277–285, doi:10.5965/223811711632017277.
20. Yinodotlgör, N.; Kartal, S.N. Heat Modification of Wood: Chemical Properties and Resistance to Mold and Decay Fungi. *Forest Products Journal* **2010**, *60*, 357–361, doi:10.13073/0015-7473-60.4.357.
21. Lima, A.; Arruda, F.; Janeiro, A.; Medeiros, J.; Baptista, J.; Madruga, J.; Lima, E. Biological Activities of Organic Extracts and Specialized Metabolites from Different Parts of *Cryptomeria Japonica* (Cupressaceae) – A Critical Review. *Phytochemistry* **2023**, *206*, 113520, doi:10.1016/j.phytochem.2022.113520.
22. Esteves, B.; Ferreira, H.; Viana, H.; Ferreira, J.; Domingos, I.; Cruz-Lopes, L.; Jones, D.; Nunes, L. Termite Resistance, Chemical and Mechanical Characterization of Paulownia Tomentosa Wood before and after Heat Treatment. *Forests* **2021**, *12*, 1114, doi:10.3390/f12081114.
23. Esteves, B.; Graça, J.; Pereira, H. Extractive Composition and Summative Chemical Analysis of Thermally Treated Eucalypt Wood. *Holzforschung* **2008**, *62*, 344–351, doi:10.1515/HF.2008.057.
24. Esteves, B.; Videira, R.; Pereira, H. Chemistry and Ecotoxicity of Heat-Treated Pine Wood Extractives. *Wood Sci Technol* **2010**, doi:10.1007/s00226-010-0356-0.
25. Esteves, B.; Ayata, U.; Cruz-Lopes, L.; Brás, I.; Ferreira, J.; Domingos, I. Changes in the Content and Composition of the Extractives in Thermally Modified Tropical Hardwoods. *Maderas-Cienc Tecnol* **2022**, *24*, 1–14, doi:http://dx.doi.org/10.4067/s0718-221x2022000100422.
26. Arihara, S.; Umeyama, A.; Bando, S.; Kobuke, S.; Imoto, S.; Ono, M.; Yoshikawa, K.; Amita, K.; Hashimoto, S. Termiticidal Constituents of the Black-Heart of *Cryptomeria Japonica*. *Journal of the Japan Wood Research Society (Japan)* **2004**, *50*.
27. Alén, R.; Kotilainen, R.; Zaman, A. Thermochemical Behavior of Norway Spruce (*Picea Abies*) at 180–225 C. *Wood Science and Technology* **2002**, *36*, 163–171, doi:10.1007/s00226-001-0133-1.
28. Windeisen, E.; Strobel, C.; Wegener, G. Chemical Changes during the Production of Thermo-Treated Beech Wood. *Wood Sci Technol* **2007**, *41*, 523–536, doi:10.1007/s00226-007-0146-5.
29. Esteves, B.M.; Domingos, I.J.; Pereira, H. Pine Wood Modification by Heat Treatment in Air. *BioResources* **2008**, *3*, 142–154, doi:10.15376/biores.3.1.142-154.
30. Yang, T.-H.; Chang, F.-R.; Lin, C.-J.; Chang, F.-C. Effects of Temperature and Duration of Heat Treatment on the Physical, Surface, and Mechanical Properties of Japanese Cedar Wood. *BioResources* **2016**, *11*, 3947–3963, doi:10.15376/biores.11.2.3947-3963.
31. Srinivas, K.; Pandey, K.K. Effect of Heat Treatment on Color Changes, Dimensional Stability, and Mechanical Properties of Wood. *Journal of Wood Chemistry and Technology* **2012**, *32*, 304–316, doi:10.1080/02773813.2012.674170.
32. Priadi, T.; Sholihah, M.; Karlinasari, L. Water Absorption and Dimensional Stability of Heat-Treated Fast-Growing Hardwoods. *Journal of the Korean Wood Science and Technology* **2019**, *47*, 567–578, doi:10.5658/WOOD.2019.47.5.567.
33. Fu, Z.; Zhou, Y.; Gao, X.; Liu, H.; Zhou, F. Changes of Water Related Properties in Radiata Pine Wood Due to Heat Treatment. *Construction and Building Materials* **2019**, *227*, 116692, doi:10.1016/j.conbuildmat.2019.116692.
34. Metsä-Kortelainen, S.; Antikainen, T.; Viitaniemi, P. The Water Absorption of Sapwood and Heartwood of Scots Pine and Norway Spruce Heat-Treated at 170 C, 190 C, 210 C and 230 C. *European Journal of Wood and Wood Products* **2006**, *64*, 192–197, doi:10.1007/s00107-005-0063-y.
35. Watanabe, K.; Kobayashi, I.; Kuroda, N.; Harada, M.; Noshiro, S. Predicting Oven-Dry Density of Sugi (*Cryptomeria Japonica*) Using near Infrared (NIR) Spectroscopy and Its Effect on Performance of Wood Moisture Meter. *J Wood Sci* **2012**, *58*, 383–390, doi:10.1007/s10086-012-1268-4.
36. Kubojima, Y.; Okano, T.; Ohta, M. Bending Strength and Toughness of Heat-Treated Wood. *Journal of Wood Science* **2000**, *46*, 8–15, doi:10.1007/BF00779547.

37. Kim, G.-H.; Yun, K.-E.; Kim, J.-J. Effect of Heat Treatment on the Decay Resistance and the Bending Properties of Radiata Pine Sapwood. *Material und Organismen* **1998**, *32*, 101–108.
38. Esteves, B.; Ayata, U.; Gurleyen, L. Effect of Heat Treatment on the Colour and Glossiness of Black Locust, Wild Pear, Linden, Alder and Willow Wood. *Drewno* **2019**, *62*.
39. EN 117 (2012) Wood Preservatives - Determination of Toxic Values against Reticulitermes Species (European Termites) (Laboratory Method). CEN, Brussels.

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.