*forests*

*Article*

# Enhanced Properties of *Cryptomeria japonica* (Thunb ex L.f.) D.Don from the Azores Through Heat-Treatment †

**Bruno Esteves [1,2,*], Lina Nunes [3,‡], Rogério Lopes [4,5] and Luísa Cruz-Lopes [1,6]**

1 Centre for Natural Resources, Environment and Society (CERNAS)—IPV Research Centre, Polytechnic Institute of Viseu (IPV), Av. Cor. José Maria Vale de Andrade, 3504-510 Viseu, Portugal; lvalente@estgv.ipv.pt
2 Department of Wood Engineering, Polytechnic Institute of Viseu (IPV), 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 Research Centre in Digital Services (CISeD), Polytechnic Institute of Viseu (IPV), Av. Cor. José Maria Vale de Andrade, 3504-510 Viseu, Portugal; rogeriolopes@estgv.ipv.pt
5 Department of Mechanical Engineering and Industrial Management, Polytechnic Institute of Viseu (IPV), Av. Cor. José Maria Vale de Andrade, 3504-510 Viseu, Portugal
6 Department of Environment, Polytechnic Institute of Viseu (IPV), Av. Cor. José Maria Vale de Andrade, 3504-510 Viseu, Portugal
* Correspondence: bruno@estgv.ipv.pt
† This article is a revised and expanded version of a paper titled "Heat Treatment of *Cryptomeria japonica* from Azores", which was presented at the 11th European Conference on Wood Modification (ECWM11), Florence, Italy, 15–16 April 2024.
‡ The author has passed away.

**Abstract:** This study evaluates the chemical, physical, mechanical, and biological properties of untreated and heat-treated *Cryptomeria japonica* (Thunb ex L.f.) D.Don wood from the Azores, Portugal. Heat treatment was performed at 212 °C for 2 h following the Thermo-D class protocol. Chemical analysis revealed an increase in ethanol soluble 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 swelling, 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; color; dimensional stability; mechanical properties; termite resistance; thermal modification



Academic Editor: Ian D. Hartley

Received: 19 December 2024
Revised: 8 January 2025
Accepted: 14 January 2025
Published: 17 January 2025

**Citation:** Esteves, B.; Nunes, L.; Lopes, R.; Cruz-Lopes, L. Enhanced Properties of *Cryptomeria japonica* (Thunb ex L.f.) D.Don from the Azores Through Heat-Treatment. *Forests* **2025**, *16*, 166. https://doi.org/10.3390/f16010166



## 1. Introduction

Commonly referred to as Japanese cedar or Sugi, *Cryptomeria japonica* (Thunb ex L.f.) D.Don, 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 m (164 ft) 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®). During heat treatment, wood is exposed to elevated temperatures (160–260 °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 [9–12].

The main objective of this work is to improve the dimensional stability of *Cryptomeria japonica* without reducing its somewhat resistance to termites. 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* (Thunb ex L.f.) D.Don originating from the Azores region, Portugal, were used for the tests. Four boards of approximately 1000 mm × 300 mm × 20 mm were cut from the same plank. Air dried wood with approximately 12% moisture content was treated in a Portuguese company known as Palser at 212 °C for 2 h in accordance with the Thermo D class from Thermowood®. The process began with a heating phase, where the wood was rapidly heated to 130 °C in a kiln. Steam was introduced to prevent wood from cracking and to create a protective atmosphere, reducing the risk of combustion. Following this, the wood underwent a thermal modification phase. The temperature was raised to 212 °C and kept for 2 h at this temperature. In the final stage, the cooling and conditioning phase, the wood was gradually cooled using water and steam [13].

### *2.1. Chemical Composition*

The samples for chemical analysis were cut into small pieces, air-dried, and crushed in a Retsch SMI mill (Retsch GmbH, Haan, Germany) and afterwards screened in a Retsch AS200 (Retsch GmbH, Haan, Germany) vibrating sieve for 30 min. The extract content was determined by sequential extraction with dichloromethane, ethanol, and hot water using the 40–60 mesh fraction. The lignin content was obtained by the Klason method [14], which consists of sequential hydrolysis twice with sulfuric acid (72% at 30 °C for 1 h and 120 °C in the autoclave for 1 h), with filtration and drying in crucibles No. 4. The insoluble lignin content was quantified in relation to the dry mass.

The holocellulose content was determined by the acid chlorite method by dissolving 2 g of wood without extractives in a 1 L flask, adding 160 mL of distilled water at 70 °C, 20 mL of a 34 g/L sodium chlorite solution, 20 mL of a 270 g/L NaOH solution, and 37.5 g of acetic acid. A total of 20 mL of each solution was added until the sample turned white, which may require 8 h [13]. The sample was then filtered in № 2 crucibles and washed with cold water and 15 mL acetone. The holocellulose (HC) content was also determined in relation to dry wood. For the $\alpha$-cellulose content, holocellulose was hydrolyzed with NaOH (17.5% at 20 °C for 30 min), filtered, washed with NaOH (8.3%) and acetic acid (10%) and dried at 105 °C. The hemicellulose content was calculated from the difference between holocellulose and $\alpha$-cellulose [15].

### *2.2. Physical and Mechanical Properties*

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) [16], calculated using Equations (1) and (2), respectively. Each cycle consisted of immersion in water at 20 °C for 48 h and drying in an oven at 100 °C for 24 h. After the soaking step the samples were removed from the bath and the surface was cleaned. All the samples were weighed and measured after each step.

Swelling Coefficient (S):

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

where $L_{100\%}$ equals the dimension of the saturated specimen (mm) after each cycle, and $L_{0\%}$ is the dimension of the dried specimen (mm) after each cycle.

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

where $S_u$ is swelling coefficient of the untreated specimen, and $S_{ht}$ equals the 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 [17] on a universal testing machine Servosis ME-405/5 (Servosis, Madrid; Spain). 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 [18]:

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

where ΔF/Δx represents the elastic strain (kg·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 breaking, approximately 2 min after the test began. Bending strength was calculated using the formula:

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

where $F_f$ is the maximum load (kg).

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

### *2.3. Surface Properties*

Samples were conditioned at 20 °C and 65% relative humidity for 2 weeks prior to color measurement. 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 (Konica Minolta Holdings, Ramsey, NJ, USA). The CIELAB technique was used to determine the color parameters L*, a*, and b* for untreated and treated wood and the resulting total color change (ΔE) was determined by Equation (5). Each result is an average of three replicates for untreated and heat-treated wood.

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \tag{5}$$

### *2.4. Termite Durability*

Wood samples were subjected to subterranean termite attack under controlled conditions at UPB.LNEC, following the EN 118:2013 standard [19]. 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 a 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 soluble extractives (1.3%), followed by water soluble extractives (0.99%) and dichloromethane soluble extractives (0.6%). These findings are consistent with previous research by Fonte et al. [20], who reported 1.81% water soluble extractives and 1.23% ethanol-toluene soluble 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 [21] documented 1.36% cyclohexane/ethanol soluble extractives, 0.49% ethanol soluble extractives, and 2.78% water soluble 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 japonica* (Thunb ex L.f.) D.Don 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 |

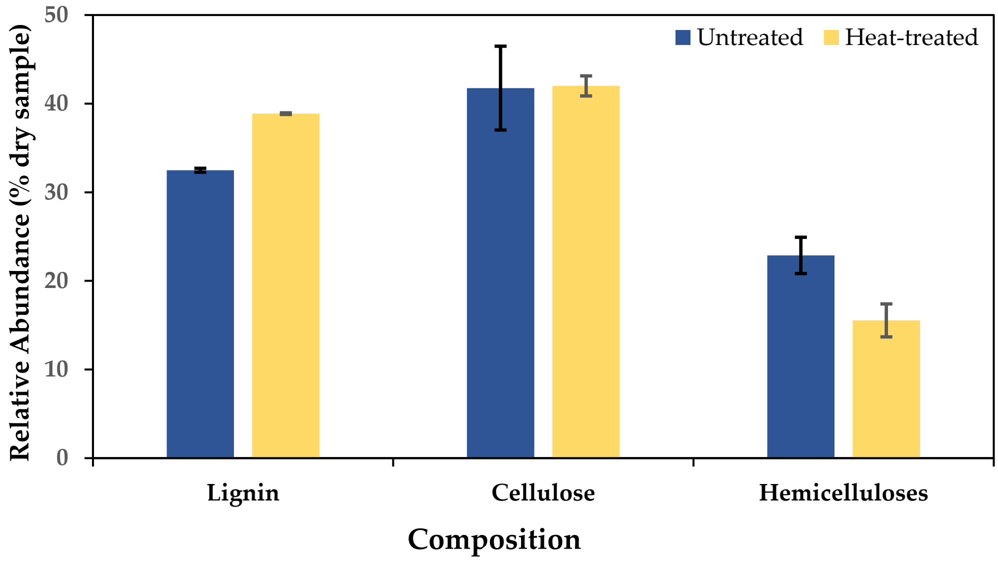


**Figure 1.** Chemical composition of untreated and heat-treated *Cryptomeria japonica* (Thunb ex L.f.) D.Don.

Lipophilic extractives from *Cryptomeria* wood were reported to be mainly terpenes, such as diterpenes, sesquiterpenes, triterpenes, and sesquarterpenes and polyphenols like flavonoids, lignans, norlignans, and anthraquinones [22].

Heat treatment appears to induce a slight increase in ethanol soluble extractives from 1.3% to 2.0%, a trend observed in other wood species such as heat-treated Paulownia [23]. This increase in ethanol soluble 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. During the thermal modification of wood, volatile compounds like fats, waxes, and furfural are released, while new extractive substances, including polysaccharide degradation products and phenolic compounds, are formed [24]

However, this trend is not universal. For instance, in thermally modified tropical hardwoods like Afrormosia (*Pericopsis elata*) and Duka (*Tapirira guianensis*), no significant increase in ethanol soluble extractives was observed. This discrepancy may be attributed to the already higher ethanol soluble 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 to optimize heat-treatment processes for specific wood species and applications.

A slight reduction in dichloromethane soluble extractives and no change in water soluble extractives were also observed. The reduction in dichloromethane soluble extractives is possibly due to the degradation or volatilization of terpenes while in the case of water extractives some were degraded but new ones were formed.

*Cryptomeria japonica* has been known to pocess 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 [21] with 35.7%.

With heat-treatment the percentage of lignin increases relatively to around 38.9% likely due to the greater degradation of hemicelluloses, which decrease from 22.9% to 15.5%, but also due to the condensation reactions between lignin and degradation compounds from polysaccharides, as stated before for Norway spruce, beech wood, or maritime pine [27,28]. No significant differences were observed in cellulose percentage, although there was a very small increase from 41.7% to 42.0%. Results presented before suggest an apparent increase in cellulose content [29].

### *3.1. 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.

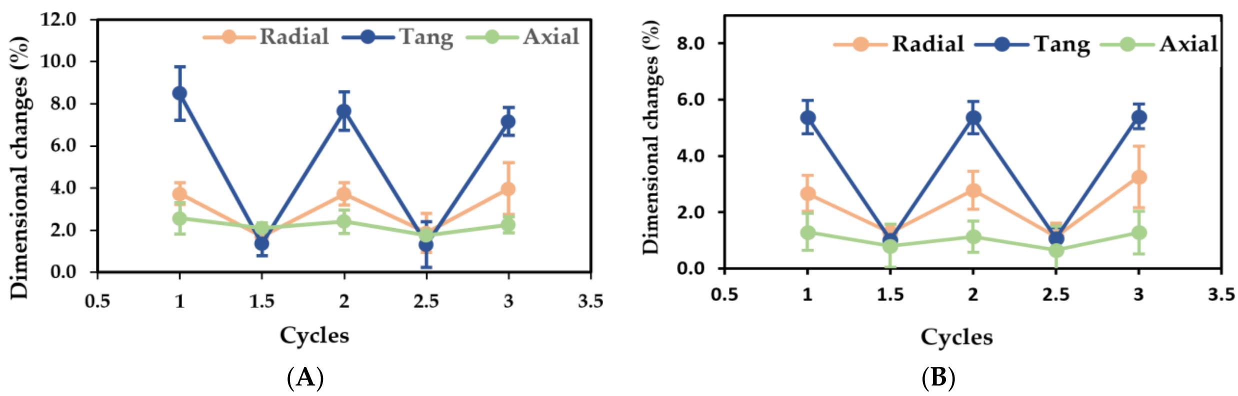


**Figure 2.** Dimensional stability of untreated (**A**) and heat-treated *Cryptomeria japonica* (Thunb ex L.f.) D.Don (**B**).

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. However, heat-treated wood 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.

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, as Figure 3 shows.

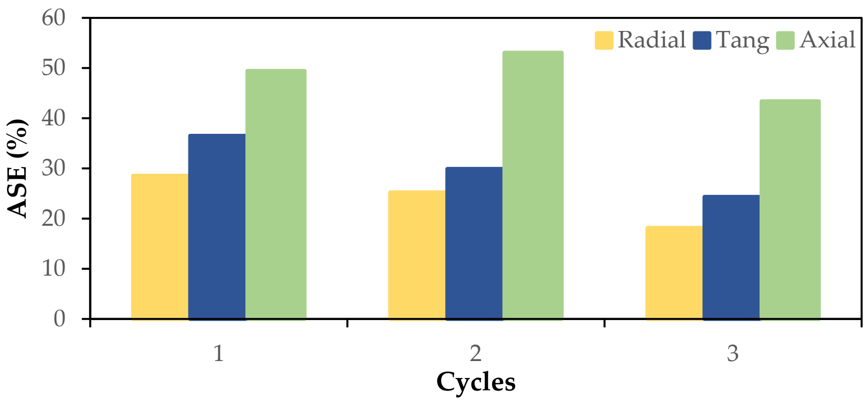


**Figure 3.** Anti-swelling efficiency of untreated and heat-treated *Cryptomeria japonica* (Thunb ex L.f.) D.Don.

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. The values are smaller than those presented before by Herrera-Builes et al. [30] for treated *Pinus oocarpa* at 190 °C which obtained a radial ASE value of 48.34%, and a tangential ASE of 54.12%. 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 in ASE along the wet and dry cycles, heat-treated wood still performs significantly better than untreated wood. Yang et al. [31] determined the ASE for heat-treated *Cryptomeria* wood at temperatures ranging from 170 to 210 °C but without using a shielding gas. The ASE ranged from 20% to 54%. The higher ASE observed for 210 °C during 4 h (54%) is obtained with an 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 [32], who reported a volumetric ASE of 20% and 12% for wood treated at 210 °C for 4 h. To compare with the results presented here, the volumetric ASE was determined for the first cycle to be 34%, which is significantly better than reported before.

The reduction of hemicelluloses, the most hydrophilic component of wood allied with the condensation reactions in lignin, are known to be the responsible for the improvements in dimensional stability.

The water absorption data for untreated and heat-treated *Cryptomeria japonica* wood across three cycles are presented in 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.

**Table 2.** Water absorption (%) of untreated and heat-treated *Cryptomeria japonica* (Thunb ex L.f.) D.Don.

| Cycle | Treatment | Average | Std. Dev. |
|---|---|---|---|
| 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 |

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 at the same levels.

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 jabon, sengon, and mangium wood samples. The control samples of jabon, 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 [33]. The same happened for radiata pine, a known softwood [34]. Nevertheless, results presented before show that heat treatment does not always decreases water absorption. For example, heat-treatment affected the water absorption of Scots pine and Norway spruce differently [35]. 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 an oven-dry density of around 0.27 g/cm$^3$ which is slightly lower than the 0.33 g/cm$^3$ presented by logs obtained from the Ibaraki Prefecture, Japan [36].

Mechanical strength is known to decrease due to heat-treatment, especially bending strength [37,38]. 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 10,836 MPa. MOE is known to increase and then decrease for higher intensity treatments and tests made with *Cryptomeria* wood treated at 210 °C for 2 h without a shielding gas led to a 26% decrease on MOE and a 42% decrease on bending strength [31]. 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 japonica* (Thunb ex L.f.) D.Don wood.

| Sample | MOE (MPa) | | Bending Strength (MPa) | |
|---|---|---|---|---|
| | Average | Std. Dev. | Average | Std. Dev. |
| Untreated | 7268 | 1194 | 52.6 | 2.4 |
| Heat-treated | 10,836 | 771 | 42.5 | 6.0 |

Thermal treatment significantly affects wood's mechanical properties by degrading cellulose, hemicelluloses, and lignin. During thermal treatment, cellulose's amorphous regions degrade, reducing polymerization and tensile strength [39,40]. Hemicelluloses decompose at lower temperatures, causing microfibril clustering and reducing wood's plasticity and shear strength [40]. At temperatures higher than 200 °C, which was the case here, hemicelluloses are extensively destroyed, leading to the decomposition of cellulose and lignin, which drastically diminishes mechanical properties such as bending strength.

The higher brittleness of treated wood can be seen in Figure 4, where some examples of the damage caused by bending strength are presented.

The main changes observed on the surface of heat-treated wood were the color, which became darker as can be seen in Figure 4 and 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, as can be seen in 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. In the case of willow, there was an initial increase in b* followed by a decrease [41]. ΔL was 20.9, similar to the reported before for wood treated at 210 °C and 2 h but without a shielding gas [31]. The total color change (ΔE) was 31.6 which is similar to the values presented before for Norway spruce, 31.6, and Scots pine, 33.23, treated by the ThermoWood method at 212 °C for 3 h [42].

**Figure 4.** Bending strength tests of untreated (**bottom**) and heat-treated (**top**) *Cryptomeria japonica* (Thunb ex L.f.) D.Don wood.

**Table 4.** Color parameters of untreated and heat-treated *Cryptomeria japonica* (Thunb ex L.f.) D.Don wood.

| L* | | a* | | b* | | |
|---|---|---|---|---|---|---|
| Average | Std. Dev. | Average | Std. Dev. | Average | Std. Dev. | ΔE |
| 67.5 | 1.2 | 13.6 | 0.5 | 24.5 | 0.4 | 31.6 |
| 46.6 | 0.8 | 1.4 | 0.3 | 4.2 | 0.2 | |

*3.2. Termite Durability*

*Cryptomeria* is known to possess some durability against subterranean termites, especially its heartwood. The resistance to termites was assessed using EN117 [43] to determine whether heat treatment enhances or reduces termite resistance. Both untreated and heat-treated samples exhibited a high susceptibility to subterranean termites (Table 5). This indicates that while heat treatment may remove some antitermic extractives from the wood, it does not substantially 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. Thermal modification is know to enhanced the biological durability against termites but only at temperatures exceeding 215 °C and with a mass loss over 20% [44], indicating the extent of structural changes necessary to improve resistance to biological agents. This threshold highlights the critical balance between effective modification and the potential for material compromise due to degradation. In some cases, even at high temperatures, no improvement is observed. For instance, Scots pine treated at temperatures from 140 to 260 °C showed no improvements in resistance against termites [45].

**Table 5.** Untreated and heat-treated wood resistance to termite.

| Material | Survival Rate [%] | Mass Loss [%] | Attack Level |
|---|---|---|---|
| Untreated | 40.60 | 8.82 | 3.8 |
| Heat-treated | 31.30 | 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 h, 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 soluble 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 amorphous fractions 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 substantially 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 and within the scope of the project Ref. UIDB/05583/2020 (Research Centre in Digital Services, CISeD)), and the Polytechnic Institute of Viseu (IPV).

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

**Conflicts of Interest:** The authors declare no conflicts of interest.

## References

1. Yasue, M.; Ogiyama, K.; Suto, S.; Tsukahara, H.; Miyahara, F.; Ohba, K. Geogaphical Differentiation of Natural Cryptomeria Stands Analyzed by Diterpene Hydrocarbon Constituents of Individual Trees. *J. Jpn. For. Soc.* **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. [CrossRef]
4. Tsukada, M. *Cryptomeria japonica*: Glacial Refugia and Late-Glacial and Postglacial Migration. *Ecology* **1982**, *63*, 1091–1105. [CrossRef]
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*; SPCF: Angeles, Philippines, 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. *Chem. Biodivers.* **2012**, *9*, 352–358. [CrossRef]
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. [CrossRef]
8. Hill, C.A. *Wood Modification: Chemical, Thermal and Other Processes*; John Wiley & Sons: Hoboken, NJ, USA, 2006; Volume 5, ISBN 0-470-02173-X.

9. Nuopponen, M.; Vuorinen, T.; Jämsä, S.; Viitaniemi, P. Thermal Modifications in Softwood Studied by FT-IR and UV Resonance Raman Spectroscopies. *J. Wood Chem. Technol.* **2005**, *24*, 13–26. [CrossRef]
10. 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 Werkst.* **1998**, *56*, 149–153. [CrossRef]
11. Boonstra, M.J.; Tjeerdsma, B. Chemical Analysis of Heat Treated Softwoods. *Holz Als Roh Werkst.* **2006**, *64*, 204–211. [CrossRef]
12. 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. *Holzforsch. Holzverwert.* **2002**, *54*, 94–99.
13. Mayes, D.; Oksanen, O. *Thermowood Handbook*; Finnish Thermowood Association: Helsinki, Finland, 2002; pp. 5–15.
14. TAPPI. *222 Om-02: Acid-Insoluble Lignin in Wood and Pulp*; TAPPI: Atlanta, GA, USA, 2002.
15. Mercado, Y.; Nunes, L.; Cruz-Lopes, L.; Esteves, B. Heat Treatment of *Cryptomeria japonica* from Azores. In Proceedings of the 11th European Conference on Wood Modification (ECWM11), Florence, Italy, 15–16 April 2024.
16. Skaar, C. *Wood-Water Relations*; Springer Science & Business Media: Berlin/Heidelberg, Germany, 2012.
17. *NP 619*; Static Bending Test (in Portuguese). Inspecção Geral dos Produtos Agrícolas e Industriais (IGPAI): Lisbon, Portugal, 1973.
18. *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: Caparica, Portugal, 2002.
19. *EN 118:2013*; Wood and Wood-Based Products—Determination of the Resistance to Subterranean Termites. European Committee for Standardization (CEN): Brussels, Belgium, 2013.
20. Fonte, A.P.N.; Trianoski, R.; Iwakiri, S.; dos Anjos, R.A.M. Physical and Chemical Properties of Heartwood and Sapwood of *Cryptomeria japonica*. *Rev. Ciênc. Agrovet.* **2017**, *16*, 277–285. [CrossRef]
21. Yinodotlgör, N.; Kartal, S.N. Heat Modification of Wood: Chemical Properties and Resistance to Mold and Decay Fungi. *For. Prod. J.* **2010**, *60*, 357–361. [CrossRef]
22. 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. [CrossRef] [PubMed]
23. 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. [CrossRef]
24. Jurczyková, T.; Šárovec, O.; Kačík, F.; Hájková, K.; Jurczyk, T.; Hrčka, R. Chromophores' Contribution to Color Changes of Thermally Modified Tropical Wood Species. *Polymers* **2023**, *15*, 4000. [CrossRef] [PubMed]
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. [CrossRef]
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*. *J. Jpn. Wood Res. Soc.* **2004**, *50*, 413–421.
27. Alén, R.; Kotilainen, R.; Zaman, A. Thermochemical Behavior of Norway Spruce (Picea Abies) at 180–225 C. *Wood Sci. Technol.* **2002**, *36*, 163–171. [CrossRef]
28. Windeisen, E.; Strobel, C.; Wegener, G. Chemical Changes during the Production of Thermo-Treated Beech Wood. *Wood Sci. Technol.* **2007**, *41*, 523–536. [CrossRef]
29. Kačík, F.; Kubovskỳ, I.; Bouček, J.; Hrčka, R.; Gaff, M.; Kačíková, D. Colour and Chemical Changes of Black Locust Wood During Heat Treatment. *Forests* **2022**, *14*, 73. [CrossRef]
30. Herrera-Builes, J.F.; Sepúlveda-Villarroel, V.; Osorio, J.A.; Salvo-Sepulveda, L.; Ananias, R.A. Effect of Thermal Modification Treatment on Some Physical and Mechanical Properties of *Pinus oocarpa* Wood. *Forests* **2021**, *12*, 249. [CrossRef]
31. 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. [CrossRef]
32. Srinivas, K.; Pandey, K.K. Effect of Heat Treatment on Color Changes, Dimensional Stability, and Mechanical Properties of Wood. *J. Wood Chem. Technol.* **2012**, *32*, 304–316. [CrossRef]
33. Priadi, T.; Sholihah, M.; Karlinasari, L. Water Absorption and Dimensional Stability of Heat-Treated Fast-Growing Hardwoods. *J. Korean Wood Sci. Technol.* **2019**, *47*, 567–578. [CrossRef]
34. Fu, Z.; Zhou, Y.; Gao, X.; Liu, H.; Zhou, F. Changes of Water Related Properties in Radiata Pine Wood Due to Heat Treatment. *Constr. Build. Mater.* **2019**, *227*, 116692. [CrossRef]
35. 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. *Eur. J. Wood Wood Prod.* **2006**, *64*, 192–197. [CrossRef]
36. 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. [CrossRef]
37. Kubojima, Y.; Okano, T.; Ohta, M. Bending Strength and Toughness of Heat-Treated Wood. *J. Wood Sci.* **2000**, *46*, 8–15. [CrossRef]

38. 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. *Mater. Und Org.* **1998**, *32*, 101–108.
39. Phuong, L.X.; Shida, S.; Saito, Y. Effects of Heat Treatment on Brittleness of Styrax Tonkinensis Wood. *J. Wood Sci.* **2007**, *53*, 181–186. [CrossRef]
40. Yue, K.; Qian, J.; Wu, P.; Jiao, X.; Lu, D.; Song, X. Experimental Analysis of Thermally-Treated Chinese Poplar Wood with Focus on Structural Application. *Ind. Crops Prod.* **2023**, *197*, 116612. [CrossRef]
41. 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*, 39–52. [CrossRef]
42. Torniainen, P.; Popescu, C.-M.; Jones, D.; Scharf, A.; Sandberg, D. Correlation of Studies between Colour, Structure and Mechanical Properties of Commercially Produced ThermoWood® Treated Norway Spruce and Scots Pine. *Forests* **2021**, *12*, 1165. [CrossRef]
43. *EN 117 (2012)*; Wood Preservatives—Determination of Toxic Values Against Reticulitermes Species (European Termites) (Laboratory Method). CEN: Brussels, Belgium, 2012.
44. Candelier, K.; Hannouz, S.; Thévenon, M.-F.; Guibal, D.; Gérardin, P.; Pétrissans, M.; Collet, R. Resistance of Thermally Modified Ash (*Fraxinus Excelsior* L.) Wood under Steam Pressure against Rot Fungi, Soil-Inhabiting Micro-Organisms and Termites. *Eur. J. Wood Prod.* **2017**, *75*, 249–262. [CrossRef]
45. Surini, T.; Charrier, F.; Malvestio, J.; Charrier, B.; Moubarik, A.; Castéra, P.; Grelier, S. Physical Properties and Termite Durability of Maritime Pine Pinus Pinaster Ait., Heat-Treated under Vacuum Pressure. *Wood Sci. Technol.* **2012**, *46*, 487–501. [CrossRef]