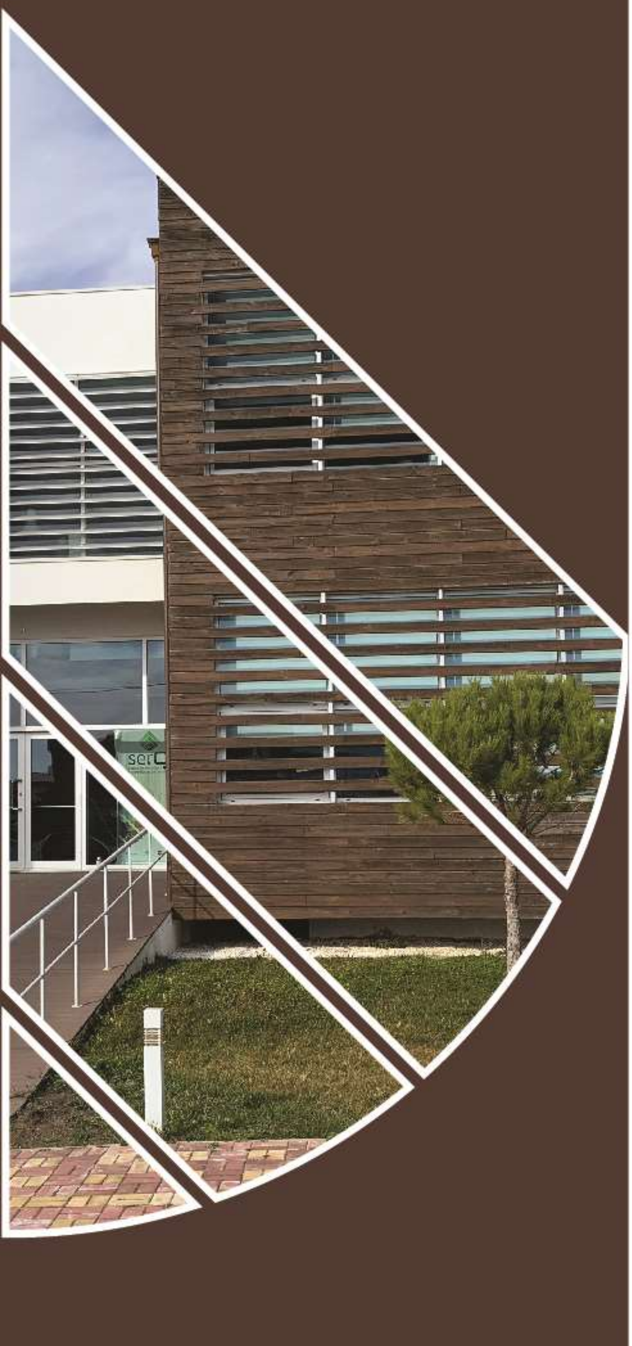

REPORT

STUDY OF DENSIFICATION OF SUGI WOOD THROUGH THE THERMO-HYDRO-MECHANICAL PROCESS

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DENSIFICATION OF SUGI TIMBER THROUGH THERMOHYDROMECHANICAL PROCESS

Abstract

This report presents the results of a study related to a thermo-hydro-mechanical treatment process of *Cryptomeria japonica* wood, as a way to increase the competitiveness of this wood in the construction market, for non-structural applications. The study included several treatment parameters (softening, pressing time, treatment temperature and densification rate), and the results obtained allow to admit this process as a viable way to support the application of sugi wood (densified) for use in flooring and in panelling for internal walls.

The study has a purely screening purpose, being at a level of TRL (Technology Readiness Level) 3 - Research supported by experimentation. The responsibility of full validation in the operational environment through tests and demonstrations (TRL 8) relies on the interested companies. This step will allow to reach the conditions necessary for its commercialization, thus reaching TRL 9 (real system finalized and qualified).

Keywords: Sugi / Thermo-Hydro-Mechanical process / Hardness / Density / Durability

ESTUDO DE DENSIFICAÇÃO DA MADEIRA DE CRIPTOMÉRIA POR MEIO DE PROCESSO TERMO-HIDRO-MECÂNICO

Resumo

O presente relatório apresenta os resultados de um estudo relativo a um processo de densificação termo-hidro-mecânico de madeira de *Cryptomeria japonica*, como forma de aumentar a competitividade desta madeira no mercado da construção, atendendo a aplicações não estruturais. O estudo incluiu diversos parâmetros do processo (amolecimento, tempo de prensagem, temperatura e taxa de densificação), sendo que os resultados obtidos permitem admitir este processo como forma viável de suportar a aplicação de madeira de criptoméria (densificada) em aplicações de revestimento de pisos ou de paredes interiores.

O estudo apresenta um caráter meramente de triagem, encontrando-se num nível de TRL (Technology Readiness Level) 3 – Investigação suportada por experimentação. Irá competir às empresas a responsabilidade da plena validação em ambiente operacional através de testes e demonstrações (TRL 8), permitindo reunir as condições necessárias para a sua comercialização, atingindo deste modo o TRL 9 (sistema real finalizado e qualificado).

Report: STUDY OF DENSIFICATION OF SUGI WOOD THROUGH THE THERMO-HYDRO-MECHANICAL PROCESS

Palavras-chave: Criptoméria / Processo termo-hidro-mecânico / Dureza / Massa volúmica / Durabilidade

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1 | Introduction

Within the scope of the project "Promotion of sugi wood in construction. New products, opportunities and markets", the wood qualification studies are carried out by the Forest Innovation and Competence Center (SerQ), through a contract established with the Sociedade de Gestão Ambiental e Conservação da Natureza,SA - Azorina , SA. Within the scope of contract a study related to the feasibility of increasing mechanical performance for non-structural purposes of sugi wood through densification was carried out, which is the subject of this report.

Densification of wood generally involves processes called thermo-hydro-mechanical (THM), or thermo-mechanical (TM), showing these processes the advantage of not incorporating the addition of substances to wood, but only incorporating energy and water in the process. In a scenario increasingly focused on the need to move towards a circular economy, this process does not imply the need for concern regarding the reuse of the material after the end of its service time.

The present study is, therefore, based on an attempt to solve the problems of sugi wood from Azores, derived from its low density and consequently low hardness, impact resistance, as well as other mechanical properties. The study comprised the design of a series of tests following a THM process, with changes being made to the process variables namely press time, press temperature or densification rate. The study and the results presented are intended merely to indicate the possibilities of the THM process as a way to increase the competitiveness of sugi wood in the construction market, given non-structural applications such as multilayer wood floor or wall covering. The study is at a level of TRL (Technology Readiness Level) 3 - Research supported by experimentation - being the responsibility of full validation in the operational environment through tests and demonstrations (TRL 8) of the necessary conditions for its commercialization of the companies that adopt this solution, thus reaching a TRL 9 (real system completed and qualified), figure 1.1.

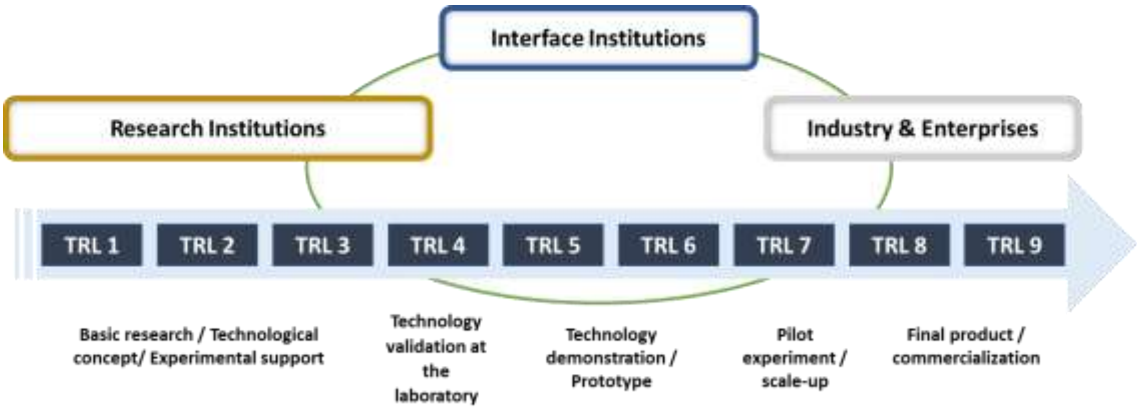


Figure 1.1 – Technology readiness levels (TRL)

2 | Wood modification processes

2.1 Generalities

The modification processes can be divided according to their objective to those that are intended for: increasing the durability of the wood; increasing its dimensional stability; or increasing / changing its physical or mechanical properties, figure 2.1.

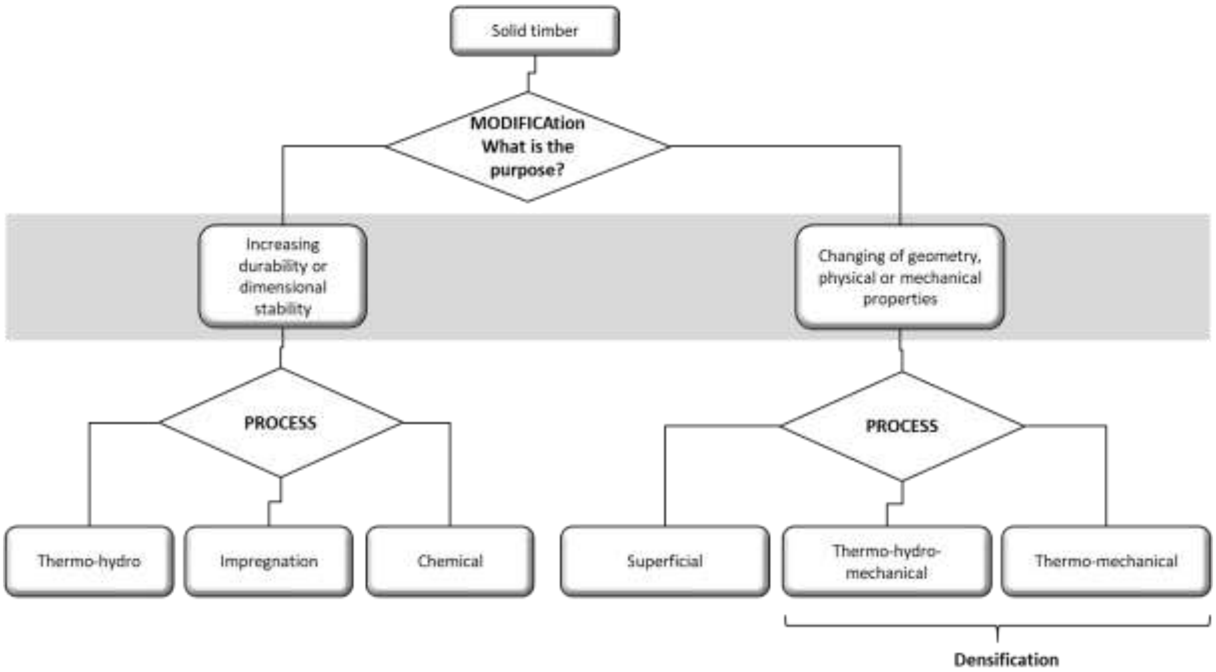


Figure 2.1 – Modification processes according to the objective

The existing densification processes can be divided between two groups, thermo-hydro-mechanical (THM) or thermo-mechanical (TM) processes, depending on whether there is a softening phase or not, respectively. A more detailed description of the advantages and disadvantages of different wood modification processes can be found in Esteves and Pereira (2014) and Sandberg *et al.* (2013). Densification has in general the objective of enabling low to medium density wood to improve its mechanical properties, constituting alternatives to denser wood, namely tropical, usually for non-structural applications.

The densification processes generally comprise four phases:

1. Softening / plasticizing.
2. Compression.
3. Cooling and drying at the deformed state.
4. Fixation of the deformed state

3 | Densification of sugi wood by a Thermo-hydro-mechanical process

3.1 Objectives

Sugi wood has low density values (average value around 350 kg/m^3), which implies a low hardness, below the lower specification limit of 10 N/mm^2 requirement for application in wood floor coverings, according to the European standardization.

In order to overcome this obstacle, a test program was defined comprising:

- Different densification cycles (thermo-hydro-mechanical process).
- Different pattern of growth rings to compression load (flat sawn or quarter sawn).

Quarter sawn   Flat sawn

The setup program constitutes a set of screening tests, covering several options regarding process parameters, and should be complemented in the future by companies interested in obtaining a final product, by an extended testing program including calibration and validation of results (product-type tests[1]).

3.2 Densification processes carried out

3.2.1 Material and methods

3.2.1.1 Densification

The processes carried out included densification with the objective of obtaining elements for use in wall coverings (TMH A) and obtaining top layers (thickness ≈ 3mm), for application in multilayer parquet elements (TMH B).

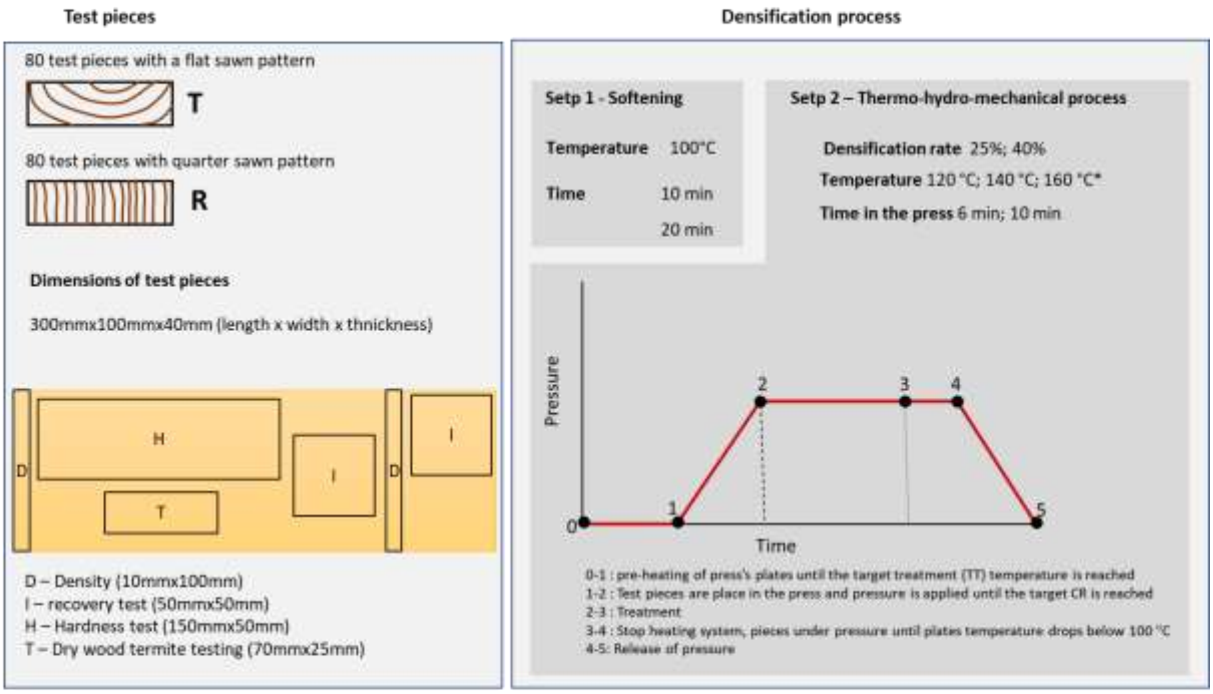
In the case of elements for application in wall coverings, the tests comprised a set of test pieces of dimensions 300mmx100mmx40mm, presenting half a flat sawn (compression applied in the radial direction) and the other half a quarter sawn (compression applied in the tangential direction) growth ring pattern. The specimens were subjected to several processes, varying the parameters established for the softening step (time) and press phase (densification rate, temperature and press time), figure 3.1. Table 3.1 shows the type and number of test pieces involved in the tests.

[1] Set of levels or classes of performance representative of a construction product, corresponding to its essential characteristics, obtained from a certain combination of raw materials or other elements according to a specific manufacturing process.

Table 3.1 – Dimensions and number of densified test pieces

Densification process	Test pieces (mm)	Number of test pieces
TMH A	300 x 100 x 40	160 1)
TMH B	200 x 100 x 6	20

1) Half of the test pieces with a flat sawn and the other half with a quarter sawn growth ring pattern



* The 160 ºC process only included 5 test pieces T with softening of 20 min, densification rate of 40% and time under pressure of 10 min

Figure 3.1 – THM A process – Test pieces and identification of the parameters of the densification process

In the case of elements for application in floor coverings, the tests comprised a set of test pieces of dimensions 300mmx100mmx40mm, presenting half a growth ring pattern of flat sawn and the other half quarter sawn. The test species were subjected to a densification process being the parameters indicated in figure 3.2.

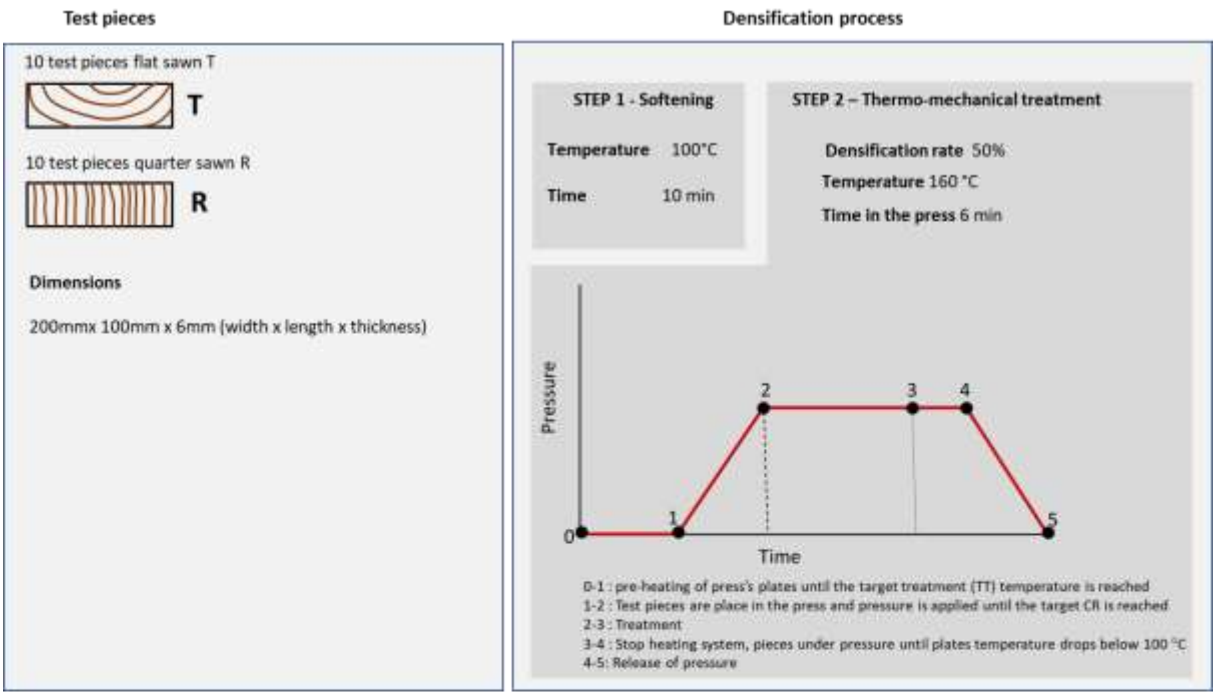


Figure 3.2 – THM B process - Test pieces and identification of the parameters of the densification process

The test pieces before being densified were initially placed in water immersion at 20°C between 6 hours to 15 hours (allowing to restore water content conditions above the fiber saturation point - green wood), and then subjected to a softening process by immersion in boiling water (100°C), using equipment with bath temperature control (± 1°C).

Figure 3.3 – a) Piece inserted in the bath; b) Equipment used for the softening of test pieces

After softening, the test pieces were placed in a hot press with a pressure capacity of 30MPa, with independent temperature control of the two plates and an accuracy of ± 2°C, figure 3.4. After the established press time the heating of the plates was turned off and the test pieces were left under pressure until the temperature of the plates dropped below 100°C.

Figure 3.4 – a) Pressing and control equipment; b) Placement of thermocouples on the test piece; c) Placing the specimen in the press; d) Pressing to the target thickness

Four thermocouples were inserted in each test piece subject to the THM A process. One next to each surface and two at the central point, figure 3.5, using a reading frequency of 17Hz. In the case of the THM B process, given the small thickness of the test pieces, no thermocouples were used.

After densification, a reading was performed with a handheld wood moisture content meter at a depth of about 0.3 x thickness. Afterwards the test pieces were placed in a conditioned room (20°C ± 2°C of temperature and 65% ± 5% of relative humidity) and the moisture content periodically controlled until it reached a value ≤ 15%, at which time the test pieces for the various scheduled tests were cut, figures 3.1 and 3.2.

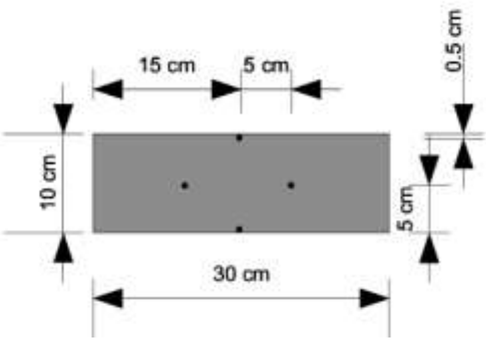


Figure 3.5 – Position of thermocouples on test pieces subject to the THM A densification process

Figure 3.6 presents the variables and combination of variables considered in the present study.

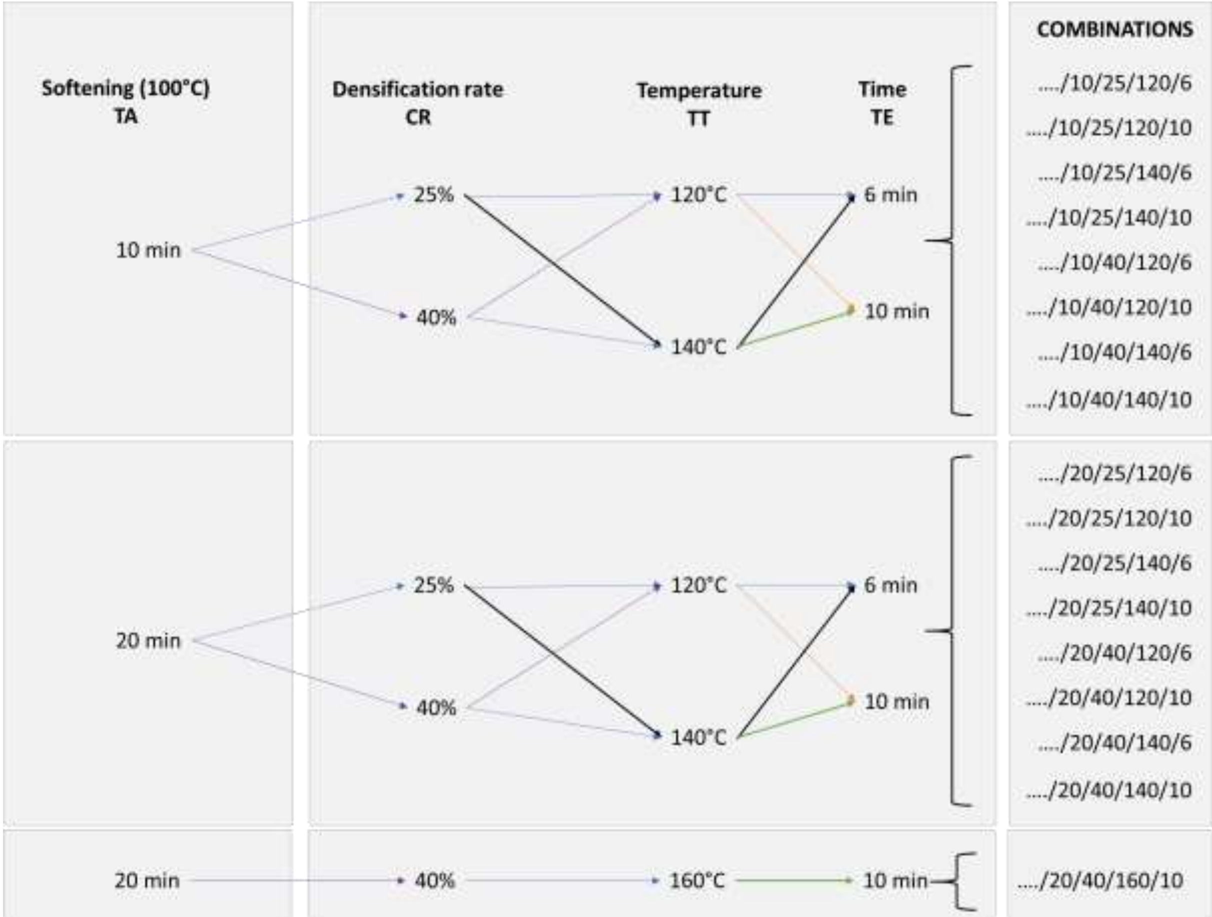


Figure 3.6 – Combination of variables studied (THM A)

3.2.1.2 Determination of density

From each initial test piece subject to the THM A process, two test species were cut to determine the density. The density was determined on test pieces of dimensions 10mmx100mmx thickness after densification, following the procedure described in ISO 13061-2.

3.2.1.3 Thickness recovery test

From the initial test pieces subject to the THM A process, two test pieces with dimensions of 50mmx50mmx thickness (variable depending on the densification rate) were cut, the tops (cross section) being sealed with epoxy resin in order to avoid the effect of water absorption by the tops, figure 3.7 .

Figure 3.7 – Test piece prepared for the thickness recovery test

The test pieces were initially subjected to the test conditions relate to application in indoor environments, adapting the procedure described in standard EN 1910. This standard establishes the prior conditioning of the test pieces in an environment of 20ºC ± 2ºC of temperature and 65% ± 5% of relative humidity of the air, until they show a weight variation of less than 0.1% in a 24-hour time interval. Then the test pieces were subjected to a dry and humid environment, being exposed to each environment for a period of no less than 28 days and until they presented a weight variation of less than 0.1% in a 24-hour time interval. Finally, the test pieces were immersed in water for 24 hours and dried in an oven at 40°C for 24 hours. This last step was adapted from the study conducted by Laine *et al.* (2016), called "Set recovery".

Table 3.2 – Exposition conditions

Exposition steps		**Conditions**	**Duration**
1	Dry environment	Air temperature - 20 °C±2 °C Air relative humidity – 30 %±5 %	Minimum 28 days and until weight stabilization
2	Humid environment	Air temperature - 20 °C±2 °C Air relative humidity – 85 %±5 %	Minimum 28 days and until weight stabilization
3	Immersion in water	Temperature of water - 20 °C±2 °C	24 hours
	Oven-dry	Temperature of air - 40 °C±2 °C	24 hours

The recovery (R) in steps 1 and 2 is calculated according to equation 1, being that of step 3 calculated according to equation 2.

$$R_{1,2} = \frac{E_i - E_{i-1}}{E_{i-1}} \times 100\ (\%) \qquad (1)$$

$$R_3 = \frac{E_i - E_d}{E_0 - E_d} \times 100\ (\%) \qquad (2)$$

Where:

E_i – thickness after step i

E_{i-1} – thickness after step i-1

E_d – thickness after densification

E_0 – thickness after densification

3.2.1.4 Hardness determination

From each densified test piece (THM A) a test piece of dimensions 150mmx50mmx thickness was obtained, being these test pieces initially being conditioned in an environment with 20°C ± 2°C of temperature and 65% ± 5% of relative air humidity, until they presented a mass variation of less than 0.1% between consecutive weighings (24 hour interval). Then the test pieces were subjected to Brinell hardness test in accordance with EN 1534, and a test was performed on one side and another test on the opposite side (minimizing the variability normally found in a piece of wood). The test pieces were then conditioned in an environment with 20°C ± 2°C of temperature and 85% ± 5% of relative humidity, until presenting again a mass variation of less than 0.1% between consecutive weighings (24 hours interval). As in the previous stage, the test pieces were subjected to a hardness test, once again a test on one surface and other on the opposite surface.

The test consisted of applying a force of 1kN on a sphere with a diameter equal to 10mm supported on the face of the specimen, figure 3.8a). After removing the force, and after a period of not less than 3 minutes, the measurement of the residual dent produced on the face of the test piece was carried out, using a binocular magnifying glass, figure 3.8b). The hardness was determined according to equation 3.

$$HB = \frac{2F}{\pi.D\left(D-\sqrt{D^2-d^2}\right)} \quad (3)$$

Where:

π – is a mathematical constant - Pi (≈ 3,14);

F – is the nominal load, in Newtons;

D – is the diameter of the sphere, in millimetre;

d – is the diameter of the residual dent, in millimetre.

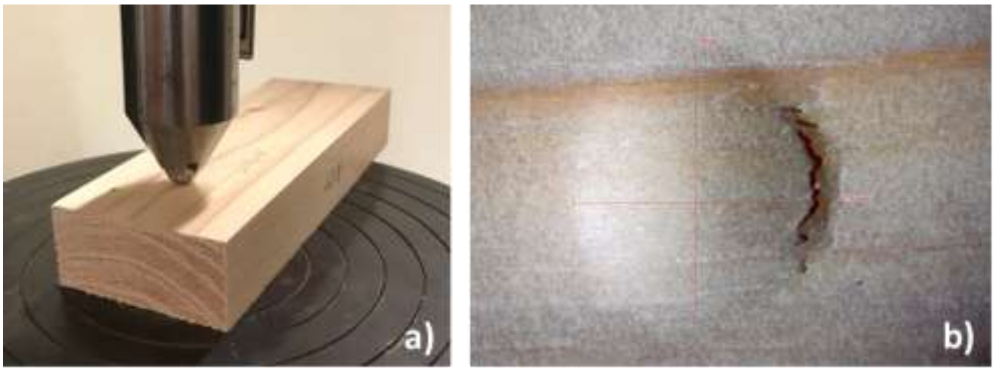


Figure 3.8 – Hardness test: a) Introduction of a sphere with a diameter equal to 10mm in the face of the specimen; b) Measurement of the indentation using a binocular magnifying glass

In order to evaluate the application in multilayer wood flooring, sheets of densified wood (≈ 3mm) were produced through the THM B process, figures 3.2 and 3.9. The sheets of densified wood produced were glued to a support of particleboard with a density greater than 700kg/m^3.

Figure 3.9 – Test piece composed of a densified wooden sheet (≈3mm) glued to a support in particleboard - hardness test

3.2.1.5 Resistance to dry wood termites

In order to verify if the densification of the material could influence the durability of sugi wood against dry wood termites (*Cryptotermes brevis* (Walker)) tests were carried out in the Autonomous Region of the Azores (Wallon et al., 2019), having been followed a method adapted from IPT (São Paulo Research Institute), 1980 - Natural and acquired durability as described in Guerreiro, O. (2015) [1]. The test comprised thirty-four (34) specimens of densified cryptomeria with the approximate dimensions: 70x25x6mm (2 specimens for each test connected by glue tape). The thickness of the specimens varied due to the densification conditions (i.e., densification rate). Three (3) maritime pine specimens (*Pinus pinaster* Aiton) and three (3) sugi specimens without densification were also used as reference. All these specimens showed dimensions like the densified specimens tested.

A PVC tube (height 4 cm, diameter 4 cm) was fixed to one face of the wood specimen. Then, inside the PVC tube (in direct contact with the wood) it was placed 30 termites of the dominant caste, pseudo-workers (figure 3.10).

Figure 3.10 – Photograph of a part of the test pieces at the beginning of the test (© S. Wallon).

The daily count of pseudo-worker individuals was carried out to identify dead and alive individuals. A weekly count of excrements (pellets) was also carried out for 41 days.

Damage was assessed each day using a qualitative scale (0 - no damage; 1 - superficial damage; 2 - moderate damage; 3 - severe damage; 4 - deep damage, just like the witness) and counting the number of holes.

3.3 Analyse and discussion of results

Throughout this point, the results will be presented in boxplot charts, showing the extent to which the data are distributed in the sample. The meaning of these diagrams are shown in figure 3.11.

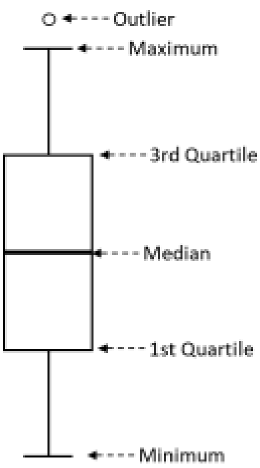


Figure 3.11 – Boxplot chart – distribution of values: 1st quartile – value equal or superior to 25% of the total values observed; Median – value equal or superior to 50% of the total values observed; 3rd quartile – value equal or superior to 75% of the total values observed; Outlier – abnormal value

3.3.1 Density

Figure 3.12 shows the results of density, and its dispersion, for the various variables considered in the THM A densification process. In the figure there is a significant increase in density for the specimens subject to a density rate of 40%, although it is worth noting an evident variability that is also observed in non-densified specimens, with the exception of the results obtained in the 160°C process.

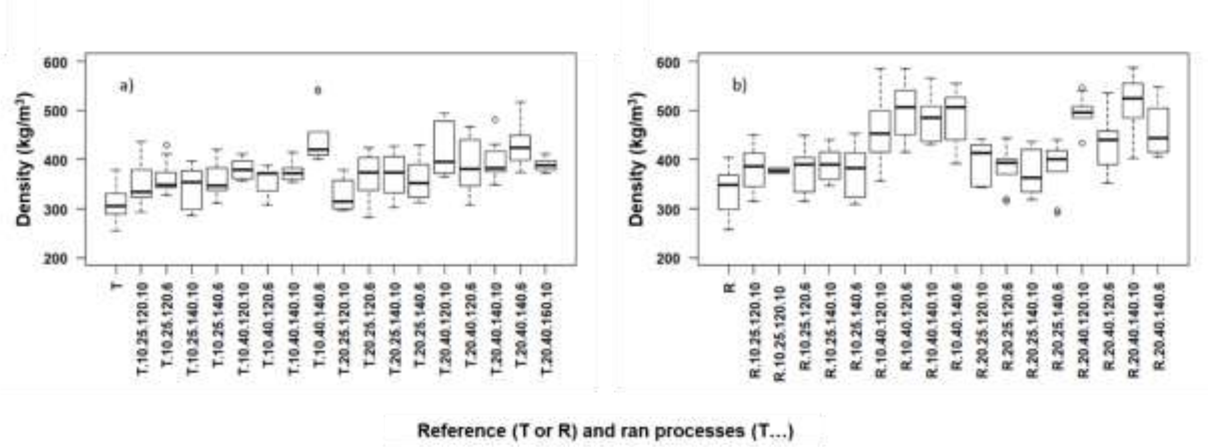


Figure 3.12 – Variation of density for the different densification processes and comparison with non-densification specimens (reference)

Figure 3.13 shows that with densification rates of 40%, the density value reaches values of approximately 500 kg/m^3, particularly when the tests are carried out on test pieces with quarter sawn growth ring pattern (load applied in the tangential direction - R test pieces).

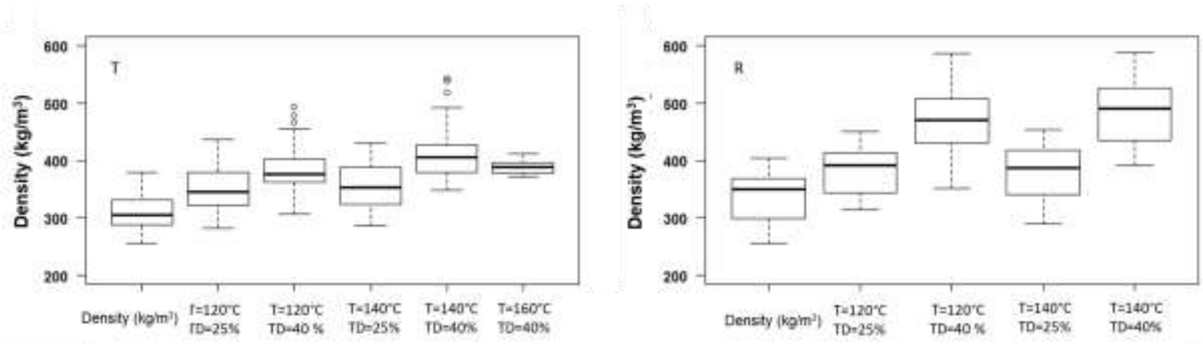


Figure 3.13 – Density variation according to the densification temperature (T) and densification rate (TD) for the two types of specimen (Flat sawn - T or quarter sawn - R)

The results obtained allow us to admit that in the development of the final product, a series of tests should be carried out, covering a temperature range from 160°C to 180°C and a densification rate of 40% to 60%.

3.3.2 Thickness recovery

The recovery movement observed between the densification phase and the stabilization of the specimens in an environment of 65% relative humidity (normalized environment) was insignificant. Figures 3.14 to 3.16 show the movement observed between the conditioning step and step 1 (conditioned environment → dry environment), between steps 1 and 2 (dry environment → humid environment) and in the immersion-drying test (external exposure), called Set-recovery.

The results obtained allow to observe a variation of properties between indoor environments (relative humidity of 30%, 65% and 80%) which is very similar between the different processes, figures 3.14 and 3.15. The application as an interior covering (floor or walls) may show inferior results since this type of covering is usually applied with a surface finishing product (e.g. varnish), which increases the resistance of wood to the adsorption / desorption of water molecules from the interior environment .

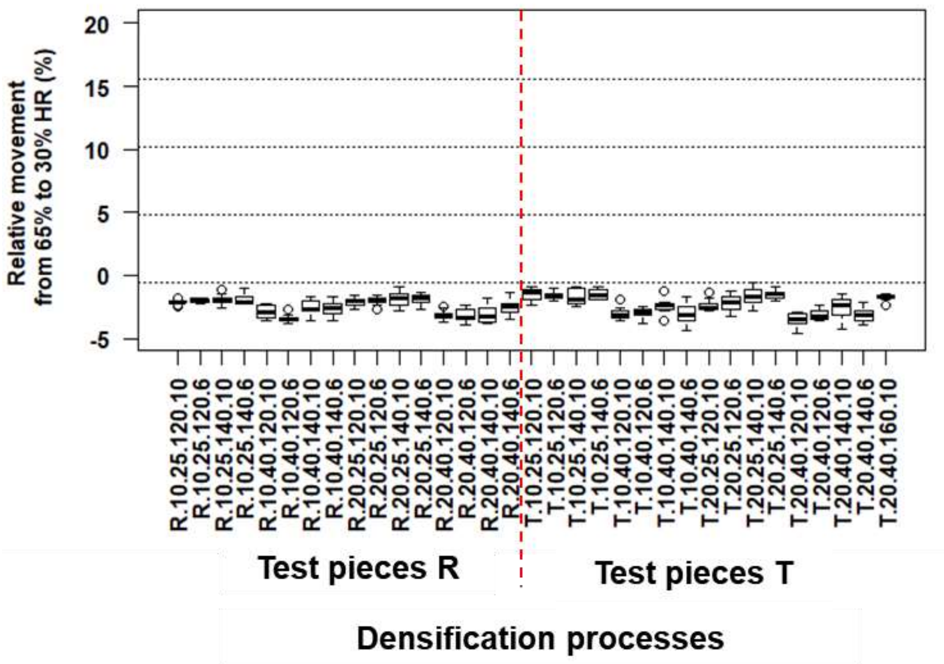


Figure 3.14 – Recovery observed between the 65% environment (standardized environment) and the 30% environment (dry interior)

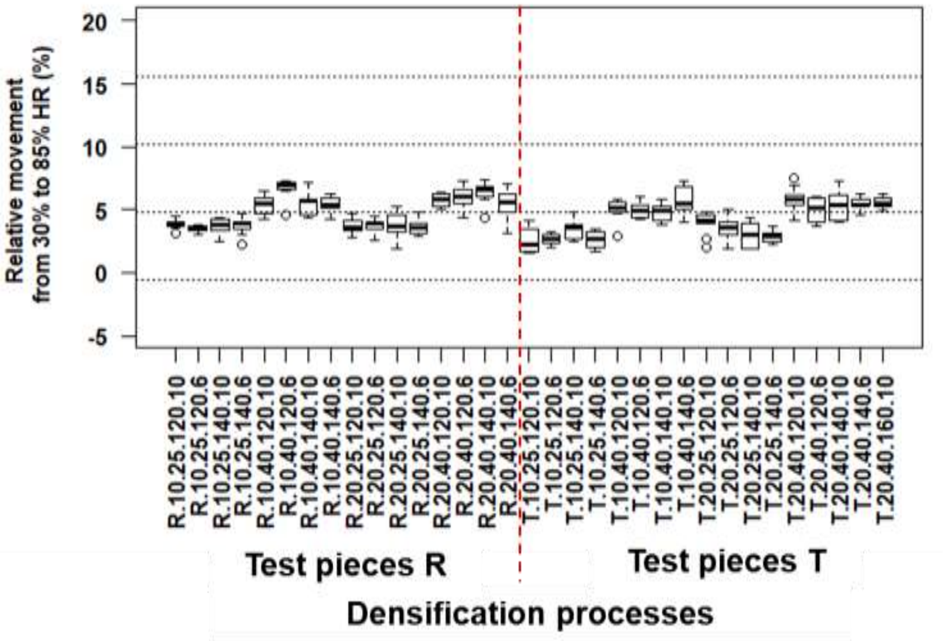


Figure 3.15 – Recovery observed between the 30% environment (dry interior) and the 85% environment (humid interior)

Regarding a more severe exposure, figure 3.16, the variation in dimensions is more evident in the case of the process carried out at 160°C (T.20.40.160.10). This result is unexpected considering that the processe present identical densification conditions as those conducted at 140°C (T.20.40.140.10), figure 3.17, aside temperature.

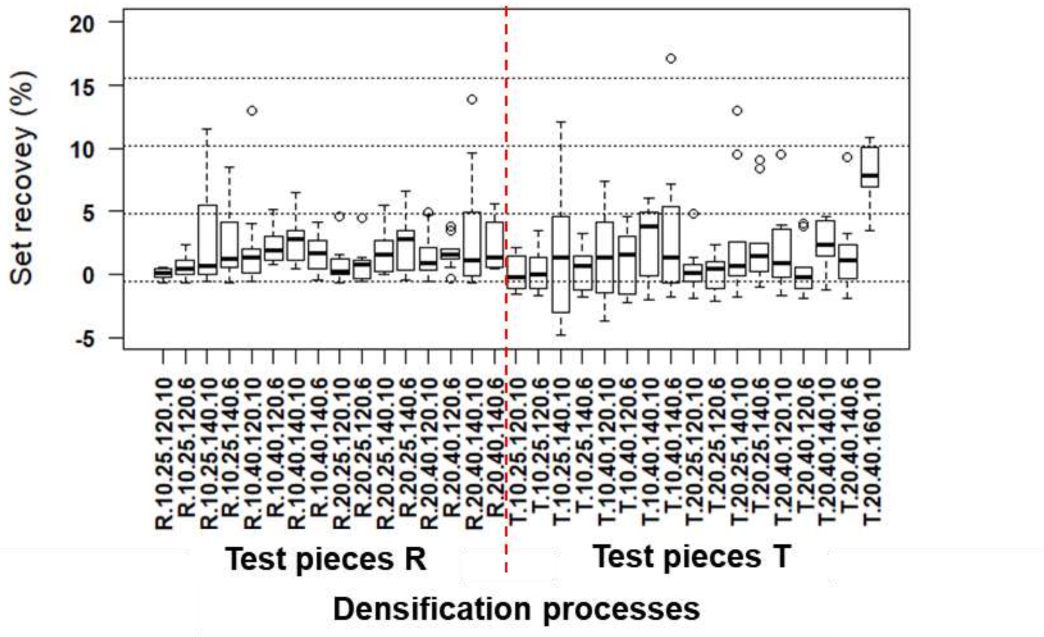


Figure 3.16 – Set recovery results

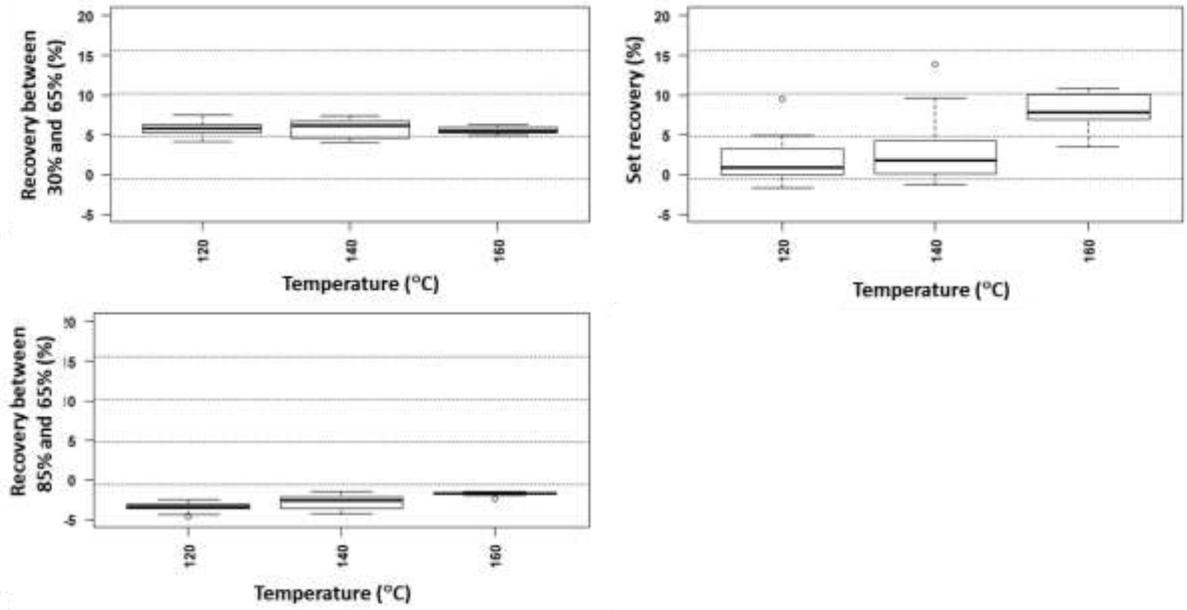


Figure 3.17 – Processes carry out with different temperatures keeping the rest of the parameters constant (TA = 20, TD = 40 and TE = 10, figure 3.6)

The results obtained tend to confirm that the recovery of wood may be a problem when applied in situations of extreme variations of the relative humidity of the air, assuming, however, that the application of coating products (e.g. paints, varnishes or stain) may result in a significant decrease in the movement of the wood.

The existing laboratory facilities available did not allow to verify the ability to obtain good results in the temperature range between 160ºC and 180ºC, through a softening phase at temperatures above 100ºC through the application of superheated steam (Kutnar and Kamke, 2012).

3.3.3 Hardness

The test on non-densified sugi test pieces was not possible according to the EN 1534 standard, since it was not possible to reach the minimum strength of 1kN required, being, therefore, only possible to state that the hardness of the sugi wood without densification is less than 6.4N/mm^2.

The tests carried out on test pieces with different pattern of orientation of the growth ring in relation to its cross section (T and R), figure 3.18, show that although the hardness values rise significantly, nevertheless present a high dispersion, not ensuring a quartile of 5% of at least 10N/mm^2 (that is, less than 5% of the values are below this value).

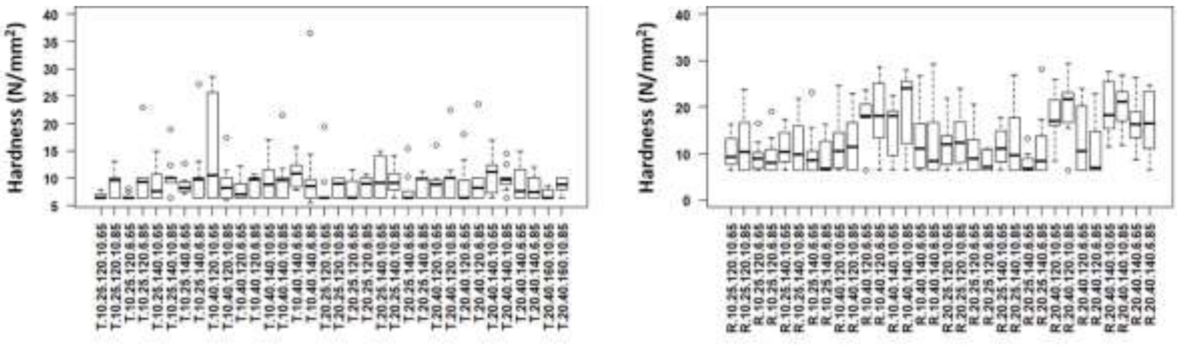


Figure 3.18 – Variation of hardness for the various densification process variables

Comparing the values obtained in the two environments, they do not present significant differences, suggesting a low rate of wood recovery. Figure 3.19 shows this result for the flat sawn growth ring pattern (T test pieces).

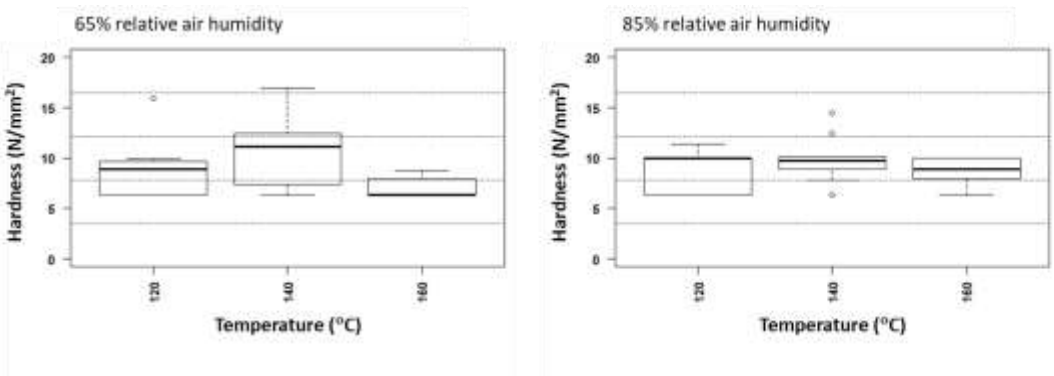


Figure 3.19 – Hardness of T test pieces as function of processes conducted with different temperatures and conditioning environments and keeping the remaining variables constant (TA = 20, TD = 40 and TE = 10)

Figure 3.20 shows that a densification rate of 40% allows, in the case of test pieces of type R, to admit the possibility of overcoming the lower hardness limit of 10N/mm^2. In the case of T-type test pieces, this possibility is not achieved, and it is necessary in future developments to analyse this difference in results dependent on the orientation of the pattern of growth rings by looking at the variation of the density profile along the thickness of the specimen.

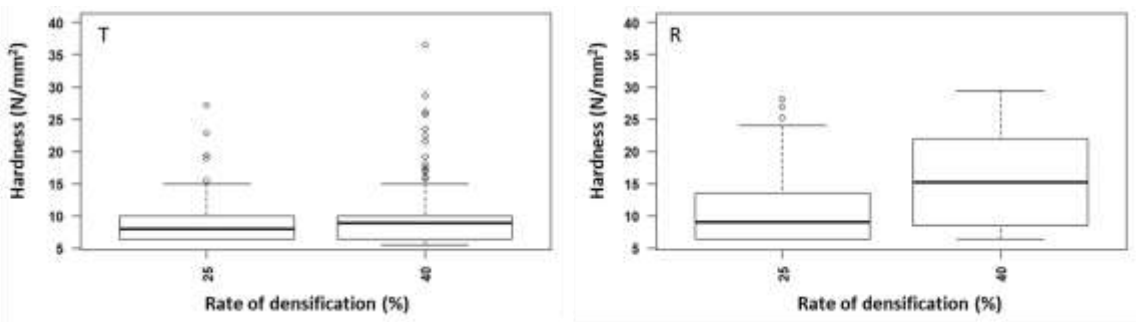


Figure 3.20 – Variation of hardness according to the rate of densification and type of test piece (Flat sawn - T or quarter sawn - R)

Regarding the application in multilayer floor elements, the results obtained, figure 3.21, indicate an increase in hardness to values significantly above the recommended minimum value of 10N/mm^2. Thus, the feasibility of applying wood sheets of densified sugi (thickness between 3 and 4 mm) in multilayer floor coverings is admitted.

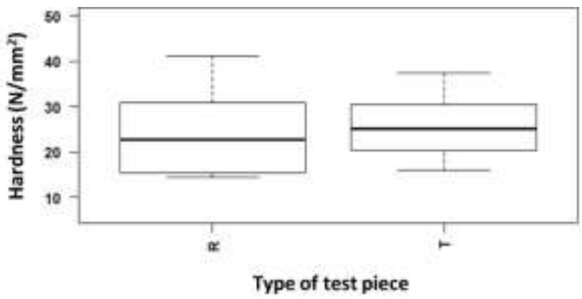


Figure 3.21 – Hardness obtained for multilayer floor elements

Figure 3.22 – Hardness test on multilayer floor element using a densified sugi layer as upper layer

Analysing the graphs of temperature readings provided by the thermocouples place near the surface and the central area of the test pieces, it noticed that there is a gap between the temperature of the plates and the surface area of about 20ºC and between the surface area and the center of the specimens of between 20ºC and 40ºC. Future tests should try to check the possibility of a higher temperature uniformity in the test pieces, through a longer press time (up to 30 minutes) or through the use of a preheating period (test piece placed in the press without pressure being applied)), following the work of Wang and Cooper (2005). However, the same study indicates that this step may result in lower densification close to the surface, which may compromise the objective of obtaining greater surface hardness.

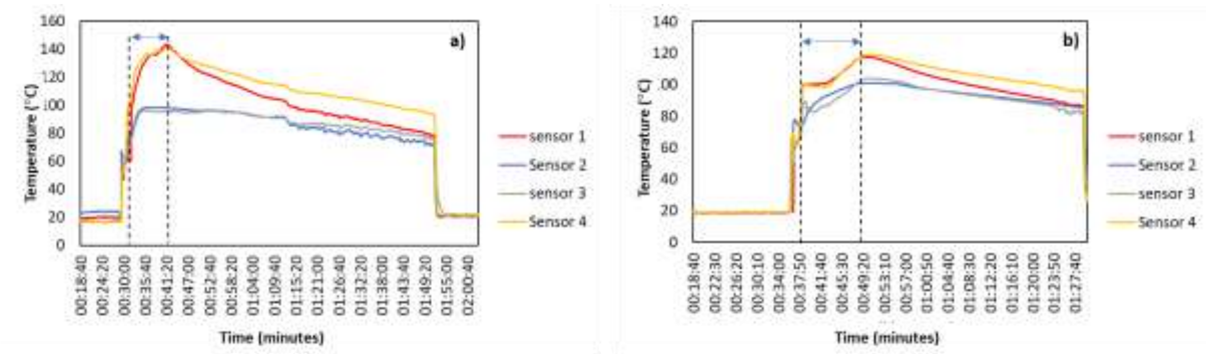


Figure 3.23 – Thermocouple reading record: a) 40% densified test piece at a temperature of 160 ℃; b) test piece densified at a temperature of 140 ℃ (arrows showing phase 2-3, see figures 3.1 and 3.2)

3.3.4 Resistance to dry wood termites

The results obtained from the screening tests indicate the apparent absence of significant differences in resistance of the wood of *Criptomeria japonica* with different densification rates, against attack by dry wood termites *Cryptotermes brevis*. However, the results indicate an apparent tendency that a longer softening time (20 min), a higher densification rate (> 40%) and a high temperature (160ºC), may influence the resistance of sugi wood to attack by *Cryptotermes brevis* dry wood termites.

Therefore, further studies related to product development tests, in case of the durability against termites, should establish as minimum densification process conditions those mentioned above.

3.4 Costs associated to production

The implementation of a densified wood production line implies a set of costs, divided by costs related to the development of a final product (including supplementary laboratory tests, product definition and product type report), costs associated with the acquisition and installation / alteration of a production line and costs associated with production (material and processing costs). The costs associated with each of these phases are dependent on market factors, which are difficult to quantify precisely at the present moment, and therefore the costs mentioned in this point are only indicative.

The type of product and intended use (including use environment) will condition the studies to be undertaken in order to the exact definition of the production parameters and the consequent product-type report (understood as the product's technical sheet). It is estimated that the costs of this phase can be between 5 to 10 k€.

The specification of the densification equipment depends on the additional studies mentioned above. However, but taking into account the conclusions of the present study and other studies carried out for other wood species, it is admitted the need to purchase a hot plate press, with a minimum 60kN and with plate cooling capacity. Equipment of this type, always dependent on the dimensions of the press, may have an estimated minimum cost of 150k€.

Regarding manufacturing costs, attention should be paid to the possibility of using the source of geothermal energy available in the Autonomous Region of the Azores as a way to reduce the energy costs associated with production, namely during the softening phase at temperatures above 100ºC through the application of superheated steam.

4. Conclusions

The present study incorporates preliminary results involving the densification of sugi wood by means of a thermo-hydro-mechanical process (THM). Several process parameters were studied, involving the softening, compression and cooling phases. The application of a specific post-treatment to increase the stability of the process has not been carried out and should be considered in future studies.

The aim of the study was to assess the feasibility of enhancing the use of this wood for flooring and interior wall cladding applications. The results obtained are promising, indicating the possibility of obtaining elements presenting a significantly higher density (≈ 500kg/m^3) than the average value associated with sugi wood (≈ 350kg/m^3), and making its use feasible in applications where a certain minimum level of hardness is required (e.g. floor coverings).

The study conducted had only in mind a preliminarily assessment of the potential of densification. The results showed that it is needed namely the decrease the variability found and increase of the stability of the process, in order to reduce the recovery of the densified material when exposed to humid conditions. The present study should be used as basis for more conclusive studies.

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