
Impact of thermal modification on properties of tropical and temperate timber species

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IMPACT OF THERMAL MODIFICATION ON PROPERTIES OF TROPICAL AND TEMPERATE TIMBER SPECIES

ALEXANDRE BUCHE

**TRAVAIL DE FIN D'ÉTUDES PRÉSENTÉ EN VUE DE L'OBTENTION DU DIPLÔME DE
MASTER BIOINGÉNIEUR EN GESTION DES FORÊTS ET ESPACES NATURELS**

ANNÉE ACADÉMIQUE 2021-2022

CO-PROMOTEURS: PR. DE MIL TOM & PR. FRANCIS FRÉDÉRIC

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Abstract

Climate change is putting a lot of pressure on some of the most used production species in Wallonia. To compete against concrete and steel that are not sustainable in the climate change context, wood's natural durability against fungi and insects is important to consider for outdoor use. More durable tropical species are imported in Wallonia but tropical forests suffer from overexploitation of a couple timber species. Chemical treatments are found to make wood more durable but regulations and people mentality make chemical treatments hard to employ. To face those problems, it is important to find a way to add values and start using more Lesser Used Species (LUS). To make them more durable against fungi and insects and environmentally friendly when treated, thermal treatment has been developed. Four LUS (*Scyphocephalum manii*, *Acer pseudoplatanus* L., *Carpinus betulus* L. and *Alnus glutinosa* (L.) Gaertn) have been thermal treated (twice at 215 °C for 3 hours) with a VACWOOD® process. Sorro presents some heartwood colour differentiation, the impact of this trait is studied in this paper. The modulus of elasticity (MOE), the modulus of rupture (MOR) and the impact bending strength (IBS) have been tested in addition to their durability against fungi (*Coniophora puteana* and *Trametes versicolor*) and termites (*Reticulitermes flavipes*). The results show a global diminution of their physico-mechanical properties after treatment. The level of diminution can be correlated with the intensity of the treatment (time and temperature). *C. betulus* seems to be less impacted. An important increase of fungal durability has been noticed. Durability against termites' attacks decreased after thermal treatment for all species. Future studies should be focused on the intensity of the treatment to find a compromise between physico-mechanical properties loss and fungal durability increase. Sorro heartwood colour impact its MOE and MOR properties probably due to heartwood streaks.

Résumé

Le changement climatique exerce une forte pression sur certaines des espèces de production les plus utilisées en Wallonie. Pour concurrencer le béton et l'acier qui ne sont pas durables dans le contexte du changement climatique, la durabilité naturelle du bois contre les champignons et les insectes est importante à prendre en compte pour une utilisation en extérieur. Des espèces tropicales plus durables sont importées en Wallonie, mais les forêts tropicales souffrent de la surexploitation de quelques essences de bois. On constate que les traitements chimiques rendent le bois plus durable, mais les réglementations et la mentalité des gens font que les traitements chimiques ne sont plus aux goûts du jour. Pour faire face à ces problèmes, il est important de trouver un moyen d'ajouter de la valeur et de commencer à utiliser davantage d'espèces moins utilisées (LUS). Pour les rendre plus durables contre les champignons et les insectes et respectueux de l'environnement lorsqu'ils sont traités, le traitement thermique a été développé. Quatre LUS (*Scyphocephalum manii*, *Acer pseudoplatanus* L., *Carpinus betulus* L. et *Alnus glutinosa* (L.) Gaertn) ont été traités thermiquement (deux fois à 215 °C pendant 3 heures) avec un procédé VACWOOD®. Le sorro présente une différenciation de couleurs du duramen, l'impact de cette caractéristique est étudiée dans ce travail. Le module d'élasticité (MOE), le module de rupture (MOR) et la résistance aux chocs (IBS) ont été testés en plus de leur durabilité contre les champignons (*Coniophora puteana* et *Trametes versicolor*) et termites (*Reticulitermes flavipes*). Les résultats montrent une diminution globale de leurs propriétés physico-mécaniques après traitement. Le niveau de diminution ne peut être corrélé à l'intensité du traitement (durée et température). *C. betulus* semble être moins impacté. Une augmentation importante de la durabilité fongique a été constatée. La durabilité contre les attaques de termites a diminué après traitement thermique pour toutes les espèces. Les études futures devraient se concentrer sur l'intensité du traitement pour trouver un compromis entre la perte de propriétés physico-mécaniques et l'augmentation de la durabilité fongique. La couleur du duramen du sorro a un impact sur ses propriétés MOE et MOR, probablement en raison des veines grasses.

Table of contents

1.	INTRODUCTION	1
2.	STATE OF THE ART	2
A.	GLOBAL CLIMATE CHANGE.....	2
B.	EFFECTS ON WALLONIA’S FORESTS FUTURE	3
C.	CAN WOOD IN CONSTRUCTION HELP?.....	3
D.	WOOD.....	4
i.	<i>Physical properties</i>	4
ii.	<i>Natural durability</i>	6
E.	WOOD’S TREATMENT	8
i.	<i>Chemical treatment</i>	8
ii.	<i>Thermal treatment</i>	9
F.	LESSER USED SPECIES.....	13
3.	MATERIAL AND METHODS	15
A.	WOOD MATERIAL	15
i.	<i>Treatment</i>	15
B.	PHYSICO-MECHANICAL PROPERTIES.....	16
i.	<i>Sample preparation</i>	16
ii.	<i>Wood density</i>	19
iii.	<i>Modulus of elasticity (MOE)</i>	19
iv.	<i>Modulus of rupture (MOR)</i>	20
v.	<i>Impact bending strength (IBS)</i>	20
C.	DURABILITY SCREENING TESTS.....	20
i.	<i>Samples preparation</i>	21
ii.	<i>Fungal tests</i>	22
iii.	<i>Reticulitermes termite tests</i>	25
D.	DATA TREATMENT AND STATISTICS.....	27
i.	<i>Software</i>	27
ii.	<i>Descriptive statistics</i>	27
iii.	<i>Variance analyses</i>	28
4.	RESULTS AND DISCUSSION.....	29
A.	VISUAL ASPECT	29
B.	PHYSICAL PROPERTIES	30
i.	<i>Equilibrium moisture content</i>	30
ii.	<i>Wood density</i>	31
C.	MECHANICAL PROPERTIES.....	33
i.	<i>Modulus of elasticity</i>	33
ii.	<i>Modulus of rupture</i>	35
iii.	<i>Impact bending strength</i>	37
D.	IMPACT OF HEARTWOOD COLOUR IN SORRO	39
E.	FUNGI.....	41
F.	TERMITES’ TESTS.....	44
G.	ALL IN ONE	49
5.	CONCLUSION AND PERSPECTIVES	50
6.	ANNEXES	52
7.	BIBLIOGRAPHY.....	55

TABLE OF FIGURES

FIGURE 1: THE THREE REFERENCES FORMING THE LIGNEOUS PLANE (MOUTEE, 2006)	5
FIGURE 2 : RETICULITERMES TERMITES; NYMPHS (CLEAR ABDOMEN WITH TWO WINGS), WORKERS (WITH A DARKER ABDOMEN) AND SOLDIERS (DARKER HEAD WITH RED MANDIBLES) (CREFFIELD,2005)	8
FIGURE 3: EVOLUTION OF THE TEMPERATURE OVER TIME FOR A THERMAL TREATMENT. SOURCE: HTTP://WWW.HEATWOOD.SE/	11
FIGURE 4 : COLOR DIFFERENCE BETWEEN (A) UNTREATED AND (B) HEAT TREATED ACER PSEUDOPLATANUS	12
FIGURE 5: UNTREATED SORRO WITH DARK HEARTWOOD AND HEARTWOOD STREAKS	13
FIGURE 6: UNTREATED HORNBEAM	13
FIGURE 7: UNTREATED ALDER.....	14
FIGURE 8: UNTREATED MAPLE	14
FIGURE 9: MASPEL OVEN MODEL TVS 6000 WDE MASPELL SRL, TERNI, ITALY) AT SCIDUS COMPANY, ETALLE, BELGIUM.	15
FIGURE 10: CUTTING SCHEME FOR THE PHYSICO-MECHANICAL TESTS BEFORE CONDITIONING	17
FIGURE 11: CUTTING SCHEME FOR MOE/MOR AND IMPACT BENDING STRENGTH TEST AND HUMIDITY SAMPLES (H)	18
FIGURE 12: CUTTING SCHEME OF TEMPERATE SPECIES BEFORE CONDITIONING	18
FIGURE 13: CUTTING SCHEME FOR MOE/MOR AND IMPACT BENDING STRENGTH TESTS AND HUMIDITY SAMPLES (H)	19
FIGURE 14: SAMPLES FOR DURABILITY TESTS (FUNGI AND TERMITES)	21
FIGURE 15: DURABILITY SAMPLES PREPARATION	21
FIGURE 16: DIFFERENCE BETWEEN AN L, R, T SAMPLE (TOP PICTURE) AND AN R, T, L SAMPLE (BOTTOM PICTURE).....	22
FIGURE 17: FUNGAL TEST PREPARATION. A: SOLUTION PREPARATION. B: FLASKS PLACED. HORIZONTALLY TO COOL DOWN. C, D: COTTON CAPS.	23
FIGURE 18: AUTOCLAVE PROCESS. A: CONTROL LABEL TO PROVE THAT THE SAMPLES HAVE BEEN AUTOCLAVED. B: AUTOCLAVE SYSTEM	23
FIGURE 19: FUNGAL TEST. FLASK PREPARATION WITH CONIOPHORA PUTEANA (CP). THE TOP PICTURE REPRESENTS A FUNGAL PREPARATION AND THE BOTTOM PICTURE SHOWS A FUNGAL TEST AFTER 8 WEEKS.....	25
FIGURE 20: TERMITES' STORAGE AT CIRAD, MONTPELLIER.....	25
FIGURE 21: TERMITES TESTS. A: CHOICE TEST. B: CONDITIONED ROOM WITH THE PETRI DISHES. C: NONCHOICE TEST WITH A TREATED SAMPLE. D: PETRI DISH WITH A CONTROL SAMPLE	26
FIGURE 22 : COMPARISON OF EACH SPECIES BEFORE (LEFT) AND AFTER TREATMENT (RIGHT). THE SPECIES ARE ORGANIZED FROM LEFT TO RIGHT ; HORNBEAM, ALDER, MAPLE AND SORRO.	29
FIGURE 23 : BOXPLOTS OF THE IMPACT OF SPECIES AND TREATMENT ON EQUILIBRIUM MOISTURE (%). WILCOXON TEST IS USED TO COMPARE THE SPECIES/TREATMENT. THE P-VALUE IS ADDED IF THERE IS A SIGNIFICANT DIFFERENCE BETWEEN TWO BOXPLOTS WITH THE DEGREE OF SIGNIFICANCE	30
FIGURE 24 : BOXPLOTS OF THE IMPACT OF SPECIES AND TREATMENT ON WOOD DENSITY (KG/M ³). WILCOXON TEST IS USED TO COMPARE THE SPECIES/TREATMENT. THE P-VALUE IS ADDED IF THERE IS A SIGNIFICANT DIFFERENCE BETWEEN TWO BOXPLOTS WITH THE DEGREE OF SIGNIFICANCE	32
FIGURE 25 : BOXPLOTS OF THE IMPACT OF SPECIES AND TREATMENT ON MODULUS OF ELASTICITY (MPA). WILCOXON TEST IS USED TO COMPARE THE SPECIES/TREATMENT. THE P-VALUE IS ADDED IF THERE IS A SIGNIFICANT DIFFERENCE BETWEEN TWO BOXPLOTS WITH THE DEGREE OF SIGNIFICANCE	34
FIGURE 26 : BOXPLOTS OF THE IMPACT OF SPECIES AND TREATMENT ON THE MODULUS OF RUPTURE (MPA). WILCOXON TEST IS USED TO COMPARE THE SPECIES/TREATMENT. THE P-VALUE IS ADDED IF THERE IS A SIGNIFICANT DIFFERENCE BETWEEN TWO BOXPLOTS WITH THE DEGREE OF SIGNIFICANCE.	36
FIGURE 27: BOXPLOTS OF THE IMPACT OF SPECIES AND TREATMENT ON THE IMPACT BENDING STRENGTH (KG/CM ²). WILCOXON TEST IS USED TO COMPARE THE SPECIES/TREATMENT. THE P-VALUE IS ADDED IF THERE IS A SIGNIFICANT DIFFERENCE BETWEEN TWO BOXPLOTS WITH THE DEGREE OF SIGNIFICANCE.....	38
FIGURE 28 : IMPACT OF HEARTWOOD COLOUR IN SORRO ON THE MODULUS OF ELASTICITY (MPA) ; C = CLEAR, D = DARK, M = MIXT.). WILCOXON TEST IS USED TO COMPARE THE COLOR OF HEARTWOOD. THE P-VALUE IS ADDED IF THERE IS A SIGNIFICANT DIFFERENCE BETWEEN TWO BOXPLOTS WITH THE DEGREE OF SIGNIFICANCE. THE MEDIAN HAS BEEN ADDED IN THE BOXPLOT ..	40
FIGURE 29 : IMPACT OF THE PRESENCE OF HEARTWOOD STREAKS IN SORRO ON THE MODULUS OF ELASTICITY (MPA) AND THE MODULUS OF RUPTURE (MPA); 0 = NO PRESENT, 1 = PRESENT.). WILCOXON TEST IS USED TO COMPARE THE PRESENCE OR NOT OF HEARTWOOD STREAKS. THE P-VALUE IS ADDED IF THERE IS A SIGNIFICANT DIFFERENCE BETWEEN TWO BOXPLOTS WITH THE DEGREE OF SIGNIFICANCE. THE MEDIAN HAS BEEN ADDED IN THE BOXPLOT	40
FIGURE 30 : IMPACT OF THERMAL TREATMENT ON THE RESISTANCE TO TRAMETES VERSICOLOR EXPRESS IN MASS LOSS (%) FOR EACH SPECIES	43
FIGURE 31 : IMPACT OF THERMAL TREATMENT ON THE RESISTANCE TO CONIOPHORA PUTEANA EXPRESS IN MASS LOSS (%) FOR EACH SPECIES	43

List of tables

TABLE 1: THERMAL CHARACTERISTICS AND EE OF BUILDING MATERIALS (CABRAL AND BLANCHET, 2021)	4
TABLE 2: RELATION BETWEEN THE MASS LOSS (%) AND THE DURABILITY CLASS FROM THE EN 350-1 (1994) STANDARD.....	24
TABLE 3: CLASSES OF DURABILITY OF WOOD SPECIES AND WOOD-BASED MATERIALS TO TERMITE ATTACK BASED ON EN 350 (2016) RATINGS	27
TABLE 4 : RELATIVE DIFFERENCE (Δ) OF THE MEDIAN VALUES OF THE EQUILIBRIUM MOISTURE (%) OF TREATED SPECIMENS COMPARED TO CONTROL SPECIMENS (%).....	31
TABLE 5 : RELATIVE DIFFERENCE (Δ) OF THE MEDIAN VALUES OF THE WOOD DENSITY OF TREATED SPECIMENS COMPARED TO CONTROL SPECIMENS (%)	32
TABLE 6 : RELATIVE DIFFERENCE (Δ) OF THE MEDIAN VALUES OF THE MOE (MPA) AND THE MEAN OF MOE RATING OF TREATED SPECIMENS COMPARED TO CONTROL SPECIMENS (%)	34
TABLE 7 : RELATIVE DIFFERENCE (Δ) OF THE MEDIAN VALUES OF THE MOR (MPA) AND THE MEAN OF MOR RATING OF TREATED SPECIMENS COMPARED TO CONTROL SPECIMENS (%)	36
TABLE 8 : RELATIVE DIFFERENCE (Δ) OF THE MEDIAN VALUES OF THE IMPACT BENDING STRENGTH (IBS) (KG/CM ²) AND THE MEAN OF IMPACT BENDING STRENGTH RATING OF TREATED SPECIMENS COMPARED TO CONTROL SPECIMENS (%)	38
TABLE 9 : SUMMARY OF FUNGAL TESTING FOR THE FOUR SPECIES TREATED AND UNTREAD WITH THEIR MEDIAN OF MASS LOSS (%) AND THEIR DURABILITY CLASS BASED ON THE RAPPORT BETWEEN SAMPLES MASS LOSS (s) AND THE CONTROL SAMPLE MASS LOSS (c) ACCORD TO THE EN 350-1 (1994) STANDARD.....	44
TABLE 10 : RESULTS OF THE TERMITE NON-CHOICE TEST FOR EACH SPECIES/TREATMENT	46
TABLE 11 : RESULTS OF THE FIRST CHOICE TEST FOR EACH UNTREATED SPECIES	47
TABLE 12 : RESULTS OF THE SECOND CHOICE TEST FOR EACH TREATED SPECIES.....	48
TABLE 13 : RESULTS OF THE THIRD CHOICE TEST FOR EACH SPECIES (UNTREAD AND TREATED IN THE SAME PETRI DISH)	48

List of equations

EQUATION 1: DENSITY (KG/M³) WITH M, THE MASS AND V, THE VOLUME AT 12% MC..... 6

EQUATION 2: MODULUS OF ELASTICITY (MPA) 19

EQUATION 3: MODULUS OF RUPTURE (MPA)..... 20

EQUATION 4: IMPACT BENDING STRENGTH (KG/CM²)..... 20

EQUATION 5: MASS LOSS FOR FUNGAL TEST 24

EQUATION 6: MOE RATING 33

EQUATION 7: MOR RATING 35

EQUATION 8: IMPACT BENDING STRENGTH RATING 37

1. Introduction

Wood has always been a construction material for humankind (Asdrubali, 2017). At first, it was mainly used because of its ease to find in the natural environment (Salman, 2017). The use of wood decreased due to the appearance and abundance of cheaper material such as concrete and steel on the construction market (Asdrubali, 2017). But for the past few years the interest for using wood in construction has been rising. This wave of interest comes from people realizing that the climate is changing and that building materials requiring a lot of fossil energy are not durable in the long term (Breyer, 2015). Concrete and steel impact the planet negatively and release large quantities of carbon dioxide (CO₂) in the atmosphere. If the floor area per capita approaches the global maximum (79 m² per capita in 2020) emissions from mineral-based construction material (steel and concrete) may reach 19 Giga tons of carbone (GtC) which is 20% of the carbon budget remaining for 2020–2050 (Churkina and al., 2020). Concrete alone causes the release of 4 to 8% of CO₂ in the atmosphere (Lehne and Presto, 2018). On the opposite of those materials, wood is a carbon-neutral building supply: to grow, a tree absorbs CO₂ and it is sequestered in the building made from wood (Buchanan and Levine, 1999). Simultaneously, wood has some attractive properties (Asdrubali, 2017). Wood presents a low conductivity either acoustic, electric or thermic. Those properties depend on the wood structure. Most of the anatomical constituents of the wood are filled with air which is a highly insulating element. Density is the main property for thermal performance which gives a clear advantage to wood material over the other ones (concrete, steel, glass, etc.) (Asdrubali and al., 2017).

Global climate change is reaching a critical point and Wallonia's forests are not spared. The changes induced by climate change are impacting those complex ecosystems. A large panel of species that were adapted to our climate are now in danger. Spruce is one of those. It is now declining due to warmer temperature in summer and various natural disasters (storms, insects, fungi) (fichier écologique des essences, 2022). For example, a massive crisis well known in Belgium is the bark beetle (*Ips typographus*). In public forests, the Department of Nature and Forest (DNF) marked more than 190,000 m³ of wood impacted by the bark beetle from April 2018 to April 2019, more than 395,000 m³ for the 2019 season and 282,600 m³ for the 2020 season. The DNF estimates the wood that still needs to be collected around 100,000 m³ at this state (Parlement de Wallonie, 2022).

When used in construction, wood may be affected by fungi or insects. To cope with the lack of durability of the untreated native wood species in Belgium, tropical species are imported or native species are treated. Belgium imports a lot of tropical species for their durability in outdoor construction (Langbour and Gerard, 2019). Tropical forests are mostly used for a few highly priced timber species which are only a small fraction of the species usable (Effah and al., 2013). The increase of the demand results in the over-exploitation of the traditional tropical traded species. Other species are usually being left out. They are not harvested mainly due to lack of knowledge about their properties. Roughly 420 of these species have a potential economic value because they can reach a timber size and about 126 of them are in sufficient quantity to be considered as an exploitable timber species (Effah and al., 2013; Poku and al., 2001). Traditional tropical timber prices are increasing due to resource depletion or countries' regulations. Producers only have little options to compensate for this price augmentation but using those remaining species is a good way to. Those species are the future of tropical forests if manufacturers want to stay competitive on the market (Effah and al., 2013).

Forest production needs to follow wood demand without being overexploited. The public is asking for more multifunctional forests to cope with the future. Mono specific forests are facing a large panel of problems due to their weakness against natural disaster, as previously cited as an example, the Spruce Beetle Crisis. Mixed forests are more resilient (Thompson, 2009). It requires a way to promote species even if they have a weaker natural durability.

Only a few native species from Wallonia are well adapted for outdoor use thanks to their good natural durability towards fungi such as: *Robinia pseudoacacia* L. (Durability Class (DC) 1), *Quercus spp.* (DC 2), *Castanea sativa* Miller (DC 2) (Salman, 2017). Nevertheless, those timber species are more expensive than the others which makes them less accessible. Different techniques exist to improve outdoor durability of wood. Nowadays, an argument to find out new preservation techniques is the ecological aspect of the products and the regulation of biocides established by the directive Registration Evaluation, Authorization and Restriction of Chemicals (REACH) (ECHA, 2022). Many preservatives have been proposed on the market but many of them were not eco-friendly. An alternative to biocide products that makes its place on the market is the thermal treatment (Repellin, 2006; Gerardin, 2016). This master thesis will be focused on the thermal treatment of lesser used forest species.

Thermal treatment can improve timber durability for construction. The material is cooked in an oven at a temperature between 160 °C and 220 °C. This will degrade different components of the cell, changing properties in the wood composition (Repellin, 2006). Heat treatment is a good way to promote less durable species and add value to the timber (Korkut, 2012).

The topic of this master thesis will be focused on the prospect of thermal treatment effects on three temperate and one tropical Lesser Used Species (LUS). The selected species are the following ones: (*Acer pseudoplatanus* L.), hornbeam (*Carpinus betulus* L.) and alder (*Alnus glutinosa* (L.) Gaertn), sorro (*Scyphocephalum manii*). The 5 research objectives that will guide this master thesis are the following one:

- A. Impact of thermal treatment on mechanical properties
- B. The impact of the heartwood colour of sorro on its mechanical properties
- C. Impact of thermal treatment on its durability against wood-decay fungi
- D. Impact of thermal treatment on its durability against termites
- E. Prospects investigation in a context of a future research

2. State of the art

A. Global climate change

Humankind is facing an important turn in history. Climate is changing and being disrupted. Originally, climate change is a natural event that makes temperature, precipitation and other natural phenomena fluctuate over various periods of time but mostly more than 10 years. Over millions of years, earth had colder and warmer periods than now but the human impact on earth is dramatically increasing the temperature. Greenhouse gases are creating a layer in the atmosphere that blocks the sunbeam on earth. The two main greenhouse gases are methane and CO₂. Fossil fuel combustion in the industry, the energy sector and transportation is the main reason for the liberation of gas in the atmosphere but agriculture and waste elimination is also two important sources. Greenhouse gases has recently reached their highest level in the last 2 million years and it keeps increasing. The temperature around the world has increased by 1.1 °C compared to the temperature registered in 1800. The last decade has been the warmest.

Climate change does not just have an impact on temperature. It also accentuates drought periods, wildfire frequency, sea level rising, floods, bigger storms and loss of biodiversity. In the 2018 report, scientists agreed that the temperature should not increase by more than 1.5 °C to avoid more serious consequences of climate change. Now, they are expecting an increase of 4.4 °C by the end of the century. In 2019, carbon dioxide level represented 148% preindustrial levels (United Nations, 2022).

B. Effects on Wallonia's forests future

Spruce is the most spread softwood species in Wallonia with a volume of 46,73106 m³ in 2016 (IPRF Wallonie, 2022). Climate change has a negative impact on the spruce. Summers tend to be dryer and hotter. Spruce has a high drought sensitivity. It has a superficial rooting which makes it more sensitive to storms that are happening more often with the climate change. (Fichier écologique des essences, 2022.) Spruce is our main production softwood species so if it disappears it will be problematic. The bark beetle crisis has already shown the impact of a large-scale crisis.

Oak trees (*Quercus robur*) and beech (*Fagus sylvatica*) trees are the main hardwood species with 25,19106 m³ and 15,79106 m³ in 2016 (IPRF Wallonie, 2022). Oak trees are not going to face as many problems as spruce trees but their production is a lot slower than spruce or beech trees. Beech trees will confront climatic problems. It is highly sensitive to humid soils. It also has a low resistance to dryness period, warm temperature and lack of water in summer. Beech has a superficial rooting which makes it sensitive to storms and heavy wind (Fichier écologique des essences, 2022). In this study, a focus has been made on LUS that have significant volume and that could be valued in the future. The tree species are *Scyphocephalum manii*. (sorro), *Acer pseudoplatanus* L. (maple), *Carpinus betulus* L. (hornbeam) and *Alnus glutinosa* (L.) Gaertn (alder). The estimated volumes for those species in Wallonia are respectively: 32,04103 m³, 34,94103 m³ and 11,82103 m³ (IPRF Wallonie, 2022). Some LUS represent a good adaptation to climate change. Hornbeam is highly resistant to the dryer periods and its rooting is strong and can resist heavy wind. Maple is also resistant to storms with strong rooting. Its weakness is its need in water. An increase of the temperature could be beneficial to alder but its weakness is the lack of water (Fichier écologique des essences, 2022).

C. Can wood in construction help?

Urbanization has been increasing in the past few years. The demand for housing, buildings, transportation and energy are intensifying. Construction in cities has been one of the biggest concerns (Selby and Desouza, 2019). An increase of 2.3% in energy consumption has been reported in 2018 by the IEA (International Energy Agency) which is twice the average growth rate since 2010 (IEA, 2019). Energy efficiency technology has the objective to use less energy to produce the same quantity of services. There are two categories of energy in buildings. Energy that comes directly from electricity or natural gas, in other terms, energy that is consumed directly from energy sources is called operational energy. The second category, the Embodied Energy (EE), is the energy that is indirectly used through construction material (Patterson, 1996). The fabrication and operation of a building require much energy. This energy does not only consider the construction but the entire existence of a building. In terms of materials used for building construction, it is estimated that it uses 40% of untreated material

extracted for it and 25% of virgin wood. Construction generates about 25% of worldwide residues. Buildings EE shows that untreated material extraction and manufactured products have a massive impact on the environment, especially for traditional construction materials such as concrete and steel. EE is lower for wood than for common building materials. Wood's thermal conductivity is also lower. This means that the operational energy needs of the building will be lower because of the better thermal insulation of wood (table 1). Less heating in winter or cooling in summer will be needed (Cabral and Blanchet, 2021).

Table 1: thermal characteristics and EE of building materials (Cabral and Blanchet, 2021)

Material	Density (kg/m ³)	Thermal Conductivity (W/m°C)	Typical Applications	Embodied Energy (MJ/kg)
Timber—softwood	450	0.12–0.14	Studs, trimmers, cripplers, other structural elements of wood frames	0.30–13.00
Timber—hardwood	700	0.17–0.23		7.00–18.00
Oriented strand boards (OSB)	650	0.13–0.24	Sub-flooring, single-layer flooring, wall and roof sheathing, ceilings/decks, structural insulated panels, webs for wood i-joists, industrial containers, mezzanines	10.00–15.00
Hardboard	1000	0.12–0.29		16.00–35.00
Particleboard	600	0.12–0.17		4.00–15.00
Medium density fiberboards (MDF)	600	0.011–0.14		8.90–11.00
Plywood	700	0.12–0.15		10.00–20.00
Cross laminated timber (CLT)	485	0.13–0.10	Floors, walls, roofing	4.90–10.00
Glulam	600	0.12–0.13	Beams, columns	8.00–14.00
Gypsum board	900	0.25–0.80	Heavy-wear locations where durability and resistance to abrasions are required	3.48–6.75
Cement-bonded board	1200	0.23–0.80	Sub-flooring, single-layer flooring, walls, ceiling/deck sheathing	4.80–6.75
Concrete	1600	0.40–0.57	Sub-flooring, beams, columns	1.70–23.90
Steel	7850	50.00–64.00	—	25.00–45.68

Hygroscopy of the wood makes it difficult to use. Therefore, wood needs to be employed in areas protected from high moisture levels. This is what this work is focusing on. Trying to find new products with better durability and stability properties that make them more versatile to use outdoor by thermal treatment of the untreated material (Kordziel and al., 2019; Voulpiotis and al., 2021).

D. Wood

i. Physical properties

Wood is a natural material which averages a composition of 50% carbon, 42% oxygen, 6% hydrogen, 1% nitrogen and 1% other components. Its main organic compounds are cellulose (38–46%), hemicellulose (13 à 29 %), lignin (25 à 33 %) and other compounds like resin and tannins (Gérard and al., 2019).

Wood is an anisotropic material. Its properties vary depending on the orientation of the fibers; longitudinal (parallel to the fibers), tangential (perpendicular to the fibers), radial (perpendicular to the fibers) (figure 1) (Salman, 2019).

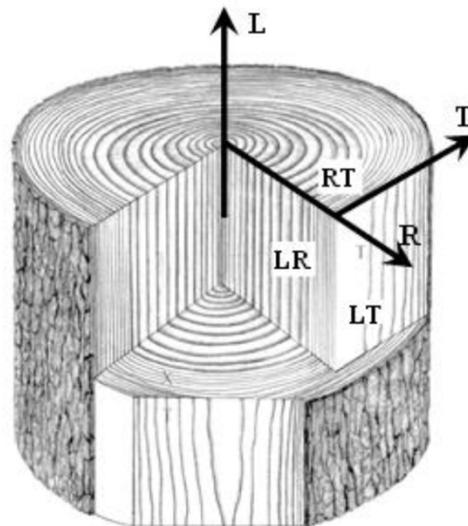


Figure 1: The three references forming the ligneous plane (Moutee, 2006)

Moisture content (MC) varies in a range between 25–250% for natural green wood, between 10–20% for naturally dried wood and 10% or less for artificially dried wood. MC is higher in sapwood than in heartwood (Almeida, 2006). MC impacts wood's natural durability. Fungi and insects need different ranges of humidity to attack the material (Fojutowski, 2009).

Water can be found in two forms, free water and bounded water. Free water is in vessels and intercellular voids. Evacuating this water when drying the wood will have an impact on its density. Density decreases when free and bounded water decreases. The bounded water saturates the fibers at $30 \pm 5\%$, it is called the Fiber Saturation (FSP) (Derbal and al., 2015). It is this water that provokes the shrinkage when drying the wood. Under the FSP, deformations and ruptures can appear (Repellin, 2006).

Hygroscopy is also a major characteristic in the wood. Wood's dimensions are stable above the FSP. Its way to absorb and resorb water is important to know the behaviour of the material. It can change its weight and volume by absorbing water or rejecting it (Salman, 2017). It rules the shrinkage and swelling of the cells which impact its physico-mechanical properties (Chung Chian, 2006). Under this FSP (30% of humidity in the fibers), wood starts experiencing deformation: shrinks when it loses water and swells when it gains water. It is due to the loss or gain of water in the cellular membranes. By virtue of anisotropy, shrinkage and swelling vary depending on the orientation. Tangential variations are between 5–10%, radial variations are between 2–9% and finally the least impacted is the longitudinal plan with variations between 0.1 - 0.3%. As a correlation between wood density and shrinkage has been highlighted. Denser woods roughly shrink more than light woods (Cave, 1972).

By convention, the density of a wood species is taken when it is at 12% MC. Wood density (kg/m^3) at 12% MC is the relation between mass (kg) and the volume (m^3) both measured at 12% MC. The wood density (kg/m^3) can be found with the equation 1. Most of the species has a density between 320 and 720 kg/m^3 . It can be as low as 160 kg/m^3 for Balsa wood (*Ochroma pyramidal*) and as high as 1040 kg/m^3 for a couple of tropical species (Borrega and al., 2015; Reyes and al., 1992). Generally, softwoods are less dense than hardwoods, but there are a couple of exceptions. Density variation depends on the wood structure and composition. In the case of hardwoods, it will mostly be related to the quantity of fibers and vessels. More vessels there are and bigger they are, the lower the density will be (Popescu and al., 2011).

Equation 1: Density (kg/m³) with m , the mass and V , the volume at 12% MC

$$\rho_{(12)} = \frac{m_{(12)}}{V_{(12)}}$$

Lastly, wood can have varying visual aspects due to its colour or its texture. This will have a role in construction when the material is visible or in carpentry to build furniture (Srinivas and Panday, 2012).

ii. Natural durability

Natural durability is characterized by the wood's resistance against external aggression such as organisms (fungi, insects), physical stress (humidity, light, cold, mechanical erosion) or chemical degradation (acid rain). Such deterioration impacts wood's structure. Each specie has its own natural durability against abiotic and biotic external factors. There is durability variation in the tree, e.g. heartwood and sapwood do not have the same resistance against external aggression. Heartwood is far more durable than sapwood. The durability can be artificially improved by applying chemical or physical treatment to the wood, it is called the conferred durability (Ross, 2021).

a) Fungi

(1) Molds and discolouration fungi

Molds (Ascomycetes, Deuteromycetes) grow on humid wood. Molds appear as coloured stain on the wood (mostly black). Molds feed on simple sugars but do not degrade lignin nor polysaccharides. They do not impact the mechanical properties of the wood. A simple drying of the wood can make those molds disappear (Ross, 2021).

(2) Basidiomycete

A Basidiomycetes attack represents the biggest risk for wood because it degrades the structural polymers of the wood. Those degradation generates mechanical properties, colour and smell change. The fungus produces an enzyme that depolymerizes the ligno-cellulosic components into simple molecules that it can absorb.

Basidiomycetes regroup brown/cubic rot and white/fibrous rot that differentiate themselves by the way they degrade the wood and their preference of the substrate. These brown and white rots can cause wood degradation both in the ground and above the ground.

Brown rots such as *Coniophora puteana* (Schumach.) P. Karst. attacks the polysaccharides (cellulose and hemicellulose) using complex reductase enzymes. Brown rot cannot degrade lignin. That rot degrades mostly softwoods and rarely hardwoods. Degraded woods show a brown aspect with cubic separations.

White rots such as *Trametes versicolor* (L.) Lloyd have the capacity to degrade all the polymers of the wood. They mostly develop on hardwood that has a high content of lignin and sometimes on softwoods. *T. versicolor* degrades lignin, cellulose and hemicellulose in the same proportion.

Other white rots sometimes degrade more the lignin and hemicellulose. In every case, the lignin is degraded faster than cellulose, leaving white cellulose fibrous exposed (Salman, 2019).

(3) Soft rot

Soft rot attacks mostly the cellulose of hardwoods and softwoods too. It degrades the wood with a high MC (ground/water contact). The degraded wood is grey/black and soft. Those fungi can either be Ascomycete or Deuteromycete. Soft rot feeds on the polysaccharides present in the S2 of the secondary wall only. The S3 of the secondary wall that is rich in lignin stays intact after a soft rot attack (Salman, 2019).

b) Insects

Insects can have a negative impact either on green wood or dry timber depending on the species. A felled tree has a high rate of moisture. The category of insects that prefer high MC wood is called fresh wood insects. They affect both softwood and hardwood. They also attack trees that are still standing up but are unhealthy because of a pathogen. The problem linked to this kind of insect is that he can transport fungi with it that can attack healthy trees.

Other species go toward dry wood. Termites and some beetles are xylophagous species. Beetles degrade wood only at the larval stage to eat starch reserves but some can also have a slight cellulose and lignin degradation activity (Scully and al., 2013).

Reticulitermes termites are part of the *Blattodea* order. There are three categories of termites. First, there are the inferior termites with the four families: *Mastotermitidae*, *Kalotermitidae*, *Hodotermitidae* and *Termopsidae*. Secondly, there are the superior termites with two families: *Termitidae* and *Rhinotermitidae*. Lastly, there is the fungus termites (Zaremski and Louppe, 2016). This classification is mainly based on their diet. The inferior termites have microorganisms in their digestive tract, some flagella protozoa and bacteria. Superior termites' digestive tubes produce cellulite enzyme or cellulase. Cellulose is being cut down into simpler sugar that can easily be assimilated by termites. They can also feed on hemicellulose and starch but are unable to digest lignin. Lignin is being rejected in their excrement and used to build the termite mound. The fungus termites prefer feeding on wood that has been previously digested by fungi. Some basidiomycete breaks down the lignin and releases the cellulose and makes it available for the termites (Zaremski, 2009).

Termites are organized in different castes: workers, soldiers, nymphs, breeders, kings, queens and larvae (figure 2). They all live and communicate with a complex organization. Workers represent 90–95% of the individuals of a colony. They assure a variety of tasks: they take care of the eggs, build the termite mound and find food for the colony. Soldiers represent less than 10% of the colony and assure the protection of the colony. Breeders are in small numbers in the colony. Their unique function is to reproduce. There are two kinds of breeders, primary and secondary breeders. They can either reproduce to expand the colony or travel to disseminate the species. If one of the original sexuate individuals of the colony dies, it is replaced by a secondary breeder (Salman, 2019; Zaremski and al, 2009).

This pest is a major economic issue for timber. Termites attack all kinds of species and do not make any difference between heartwood and sapwood. Only a few exceptions of tropical species hardwood are not being eaten because they are too dense. They mostly feed on

earlywood due to its higher concentration in starch (Salman 2019). Cellulose is a hard polymer to digest for insects (Zaremski and al, 2009).

Reticulitermes termites can be found in the United States, Japan, Southern Africa and Europe (Verma and al., 2009). In Europe termites are mainly situated in the forest in the south of the continent (Spain, Portugal, Andorra, Gibraltar, France, Italy and the Balkans). In urban area, they are more widely spread. In 1953, about 10 French departments were impacted but in 1989 it went up to 52 (Clement and al., 2001). This spread has 3 origins:

- First, urbanization and the generalization of heating in households have facilitated the conditions for termites to spread and survive over winter;
- Secondly, general trade between countries has eased the transport of termites from infected areas to safe ones (Zaremski and Louppe, 2016);
- Thirdly, global climate change makes temperature rise. This augmentation spread livable conditions for termites across Europe with higher temperature in summer and softer winter.



Figure 2 : *Reticulitermes* termites; nymphs (clear abdomen with two wings), workers (with a darker abdomen) and soldiers (darker head with red mandibles) (Creffield, 2005)

E. Wood's treatment

i. Chemical treatment

Wood protection can be biocidal treatment or non-biocidal treatment (Gerardin, 2016). There are oils, water-soluble products and products soluble in volatile organic solvent. The most used products are creosote, pentachlorophenol (PCP) and the Chromium—Copper—Arsenic (CCA). Biocidal products are under restriction due to the Biocide and REACH directives. CCA is banned in Europe since 2004. PCP and Creosote are under scrutiny and under very strict regulations. Wood protection systems based on biocide solutions/products/formulations, cause problems on the manufacture sites, during treatment process, and once impregnated in the wood as they can be leached out (with the rain for outdoor uses) and/or cause other emissions. At the end of its service life, biocide treated wood is a problematic waste (ECHA, 2022).

According to Repellin (2006), there are a couple of treatments to cite:

- Acetylation increases dimensional stability by a chemical reaction with the hydroxyl group. Hydroxyl is being replaced by acetyl groups. Those groups are bigger than hydroxyl which is translated into a mass augmentation (up to 18% for hardwoods and to 25% for softwoods);
- Furfurylation is a treatment that uses alcohol furfuryl. Those molecules are small enough to penetrate the membrane and polymerize by an augmentation of the temperature. The composite wood resulting from the polymerization is exceptionally stable and resistant to acid and base;
- Lastly, there is the polyethylene treatment (PEG) that can be applied to historical wooden objects for curative treatment. The PEG replaces all the water and keeps the cells swollen.

ii. Thermal treatment

To answer the environmental problem of chemical treatment, thermal has been developed (Gerardin, 2016)

a) Historic

Thermal treatment has already been used at the prehistoric age for arrowheads. They were burned to give them stiffness and durability (Salman, 2019). First scientific research has been conducted in the United States with Stamm in 1937 and White in 1944. In Germany similar research was in development with Bavendan in 1944, Runkel in 1951 and Buro between 1954 and 1955. Research began in 1985 in the national school of mines in Saint-Etienne to reach the first objective of obtaining torrefied wood for the mine industry. Later, the process of “Retified Wood®” has been developed in that laboratory (Candelier and al., 2016).

At the beginning, heat treatment of wood suffered from a lack of data and knowledge about the degradation of the wood linked to many different processes and the difficulty to control the process to obtain a homogenous product at the end. People were being cautious in the use of those products. Treated wood volume only represents a couple thousand cubic meters in the north of Europe. Finland is the biggest producer due to their number of installations. Thermally modified wood only represents a small part of the market even if the environmental arguments are in its favour compared to chemically treated wood (Cao and al., 2022; Esteves and Pereira, 2009).

b) Technical

Many processes exist on the industrial market. Most of them have been developed in the past 20 years. Those technologies are different depending on the product wanted (heart or surface treatment), the heat-carrying environment or the way to reduce oxygen in the oven. All the processes have the goal to either give more durability, stability and colour to the wood. The main difference is the atmosphere used to reduce oxygen in the oven (nitrogen, water vapour, void, oil, smoke). Wood is dried before going in the oven and needs an approximative MC of 12%. The main different technics are:

- **Retification®**: this French process comes from the mining school of Saint-Etienne. It is a controlled pyrolyse with a nitrogen convection. It is separated into three different phases. The first one consists in drying the wood between 20 °C and 120 °C, the atmosphere is charged in water vapour and CO₂. After that, a relaxation phase begins at a temperature between 150 °C and 200 °C which is followed by the depolymerization of the polysaccharides. Azote is then added when the temperature reaches 160 °C. The last phase aims to degrade hemicellulose and to modify lignin. It happens when the temperature is between 200 °C and 250 °C. The duration of all processes is around 25 hours.
- **Perdure®**: this process is French but its similar process in Canada is called Pluricap Inc. It is a controlled pyrolysis under a smoke atmosphere. The wood is heated under air using natural gas burners. The released gases from the wood decomposition are reused to control the atmosphere of the oven.
- **VTT®**: Thermowood technologies have been created from the Finnish research center VTT. It is a controlled pyrolysis under water vapour atmosphere. The temperature is superior to 180 °C with water vapour. Steam presence is a way to reduce the temperature difference between wood and atmosphere. There are 3 phases to this process. First, the temperature rises rapidly to 100 °C then slowly to 130 °C with steam to dry the wood to obtain an anhydrous material. The second phase's temperature rises to 185 °C and 215 °C for 2 or 3 hours. The chamber is saturated in pyrolysis gases and steam. To end the process, the cool down requires a cold water pulverization to lower the temperature of the wood.
- **Under Vacuum**: The Prodéo® process heats the wood by conduction under vacuum to obtain a homogenous product, durable and dimensionally stable. The conduction is facilitated by metal plates heated with vegetable oils. The wood pieces are being placed in the chamber with metal plated inserted between wood planks. The process is under vacuum to limit the oxygen presence. The wood is dried until a mass stabilization, then a thermal treatment at a temperature between 200 °C and 240 °C and finally a cool down. The total duration is around 30 hours. The Swiss company Prodéo® has closed and has been bought by the German company Timura.
- **Oil bath**: This process is different from the other ones because in the final product, there is a presence of residual oil into it. Its origin is German from OHT-Menz Holz. Dry wood is placed into a cylinder that contains oil heated at a temperature between 180 °C and 200 °C. After oil evacuation, the wood is air dried, so the oil is hardening. This process is mostly used for little impregnable softwood. The oleo-thermy process has been developed at the CIRAD. Green wood is submerged into a first bath of warm oil (<150 °C). It generates steam and suppresses water into the material. It is submerged in a second oil bath (<100 °C). Steam is condensed and it creates a depression in the material. There is an aspiration of the surrounding fluid. The wood is finally dried to finish the impregnation (Chaouch, 2018; CRIQ, 2003).
- **VACWOOD®**: This process has been developed by WDE-Maspell and VacWood®. After being placed in the oven, the wood is dried during a preheating phase. This is operated under atmospheric pressure. Ventilators produce an airflow that passes through the heater to dry the wood (phase 1). After that the thermal treatment starts. The vacuum evacuates the air from the oven until there are no more air in it. At the same time, the ventilators and the heating systems keep giving the necessary energy to stay at the temperature of the program. It can reach the thermal treatment program temperature requested. The vapors and gas from the wood are vacuumed and condensed out of the oven (phase 2). This phase lasts until a certain mass loss in the wood is reached. Finally,

a cooling phase starts. The heating system stops but the ventilators keep ventilating the wood (phase 3) (figure 3).

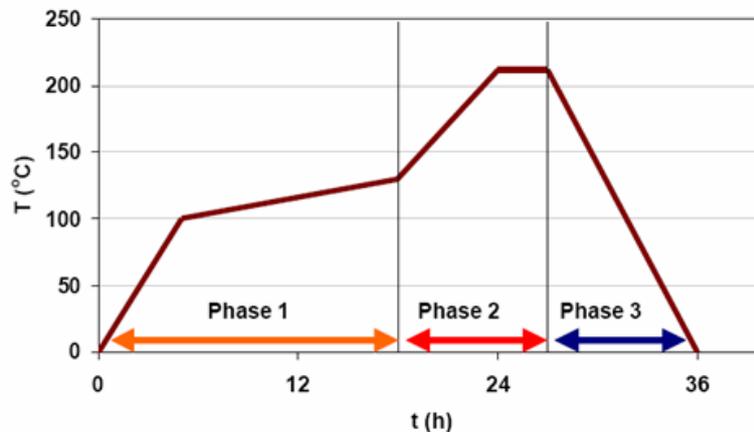


Figure 3: Evolution of the temperature over time for a thermal treatment. Source: <http://www.heatwood.se/>

c) Effects

Wood is treated in his mass. It is possible to cut or plane the plank afterward without losing any benefits given by the treatment. Timber loses mass. The longer the treatment is the darker the wood will be (figure 7). the two factors to have a control of the duration of the treatment are the colour and the mass loss.

Thermic treatment provokes a hemicellulose degradation into the wood structure. A study of poplar and pine tree's Nuclear Magnetic Resonance (NMR) spectroscopy shows a xylan degradation. Hemicellulose is the most hydrophilic compound. Their diminution is considered as the principal cause of hygrosopicity reduction in heat-treated wood (Cao and al., 2022; Candelier and al., 2016). Cellulose is impacted with an augmentation of the crystallization (Bhuiyan and al., 2000). Treatment also impacts lignin compounds. There is a reticulation of the lignin. Infrared spectrometry with diffuse reflection shows peaks that represent linkage between wood molecules. Natural wood and reticulated wood spectrum are close which means a chemical structure almost similar. For pine wood, there is a diminution is C=O links in lignin and an augmentation of C-O-C with temperature augmentation (up to 240 °C). Ether linkage formation (C-O-C) between wood compounds happens because of the carbonyl group opening (C=O). Lignin degradation is associated with methanol emission from the loss of methoxyl groups (Repellin, 2006).

Due to the diminution of hemicellulose, the density decreases (Hannouz and al., 2015). Heat modified wood is close to fragile material and breaks faster than untreated wood. (Repellin, 2006). The changing degree depends on the species and the temperature of the applied treated processes (Van Blokland and al., 2018). Mechanical properties are impacted by the modification of the cell wall chemistry structure with heat treatment. Due to hemicelluloses reduction, the depolymerization of cellulose, the increase of crystallization and other changes in the polymeric component of the cell's walls, the mechanical properties are affected in diverse degrees (Van Blokland and al., 2018). Hannouz and al. (2015) showed in his study that mechanical properties are modified when wood is heat treated. All mechanical properties are reduced except compression parallels that increase by 11%. The most constraining is the shear

strength with a decrease of 74%. The modulus of elasticity parallel to grain does not change given the uncertainty of the measurement. MOE (modulus of elasticity) often increases when the heat treatment is light while it decreases when the heat treatment is stronger (Militz, 2008).

Wood fungal durability is increased when a thermal treatment is applied. Impact of brown and white rot attacks is highly decreased after treatment. The level of resistance to fungi depends on the treatment process, the specie and the intensity of the process (Kandem and al., 2002). There are four origins of this increasing resistance:

- The hydrophobicity of thermal treated wood reduces the moisture content of the wood and block the development of fungi due to a lack of water (Hakkou and al., 2006);
- The development of toxic molecules that block the propagation of the fungi (Peters and al., 2009);
- The modification of the polymers of the wood prevents the enzymatic system of the fungi to degrade the polymers (Lekounougou and al., 2009);
- The degradation of the hemicelluloses makes the feeding of the fungus difficult (Hakkou and al., 2006).

The thermal treatment decreases the resistance of wood against termites by producing more substances more attractive for termites. Polysaccharides degradation produces light molecular weight sugar that can be easily eaten by termites (Doi and al., 1999). In the opposite, when treated, some species, such as *Pinus pinaster Aiton* produces substances that are toxic for termites and a high mortality is observed (Surini and al., 2012).

The colour of the material is also impacted (figure 4). The longer the treatment is the darker the material will be. Also, the higher the temperature is and the darker the wood will be (Repellin, 2006). The colour does not impact the mechanical properties but is an important factor for wood employment in construction when the material is visible.



Figure 4 : Color difference between (a) untreated and (b) heat treated *Acer pseudoplatanus*

F. Lesser Used Species

Sorro belongs to the *Myristicaceae* family and its scientific name is *Scyphocephalum mannii*. Different varieties of Sorro exist but they are mostly named under the name *Scyphocephalum*. It can be found in Cameroon under the appellation of “Akurna” or “Eboukzok” and in Gabon by the name “N’suku”, “Sorro” and “Ossoko”. Its wood is brown. Sorro wood has two colours of heartwood, one is clear and the other one is darker. It is a problematic for sawmills in Gabon to sell sorro wood at a good price because clear heartwood can mingle with sapwood even if it is heartwood. This makes the value of the wood drop. The differences between clear and dark heartwood are not known yet but it is an important question to answer. Another visual aspect is the heartwood streaks (figure 5). Those purple lines are mostly situated between clear and dark heartwood but not only. Their origins are unknown yet. Its grain is medium and straight with a small portion of interlocked grain. The wood is nondurable against fungi (DC 5). It is nondurable against termites (DC S). This wood species can be utilized in Use Class 1 (UC 1), inside, with no risk link to humidity (Tropix, 2022).

Sorro is the 6th most abundant species in the Congo Basin after *Aucoumea klaineana*, *Entandrophragma cylindricum*, *Terminalia superba*, *Triplochiton scleroxylon* and *Triplochiton scleroxylon*. Even with its mobilizable volume, it is not exploited as much as the other species. It has a crucial importance strategy of diversification in central Africa (Sherif and al., 2018).



Figure 5: Untreated sorro with dark heartwood and heartwood streaks

European hornbeam belongs to the *Betulaceae* family and its scientific name is *Carpinus betulus* L. Its wood’s colour is nearly white with pale yellowish-brown heartwood (figure 6). Heartwood and sapwood are unclearly demarcated. Hornbeam sapwood is very thick. The grain is straight and sometimes slightly interlocked and it has a fine texture. Hornbeam is rated as nondurable to fungi attacks (DC 5) and it is sensitive (DC S) to insect attacks. This timber species is also employed for indoor (UC 1) uses such as flooring or handles. It is mostly found in Europe and western Asia (EN350, 2016).



Figure 6: Untreated hornbeam

Black alder belongs to the *Betulaceae* family and its scientific name is *Alnus glutinosa* (L.) Gaertn. Its wood colour tends to be light tan to reddish brown (figure 7). There is no visible distinction between heartwood and sapwood. Aggregate rays can appear as small streaks and can be mistaken as wood's defects. Generally, grain is straight, but some individuals can present wild and irregular grain. It has a fine texture. Alder is rated as nondurable to fungi (DC 5) and sensitive insects (DC S). It is also employed for indoor uses (UC 1) such as plywood or turned items. It can be found in western Europe (EN350, 2016).



Figure 7: Untreated alder

Sycamore maple is a *Sapindaceae*. Its scientific name is *Acer pseudoplatanus* L. It is a white wood yellowish sheen and there is no differentiation between hardwood and sapwood (figure 8). Grain is straight and it is not interlocked. Maple wood is nondurable towards fungi (DC 5) and it is sensible to termites (DC S). Maple timber is used for indoor (UC 1) use such as cabinetmaking or wooden floor. Sycamore maple is present in Europe and southern Asia (EN350, 2016).



Figure 8: Untreated maple

3. Material and methods

All physico-mechanical tests have been run in the wood technology laboratory (LTB) from the Agronomic Research Center of Wallonia (CRa-W) at the public services of Wallonia (SPW) in Gembloux. Durability tests have been run in the research unit BiowooEB at the Center of international cooperation in agronomic research for development (CIRAD) in Montpellier.

A. Wood Material

Sorro wood was imported from the FSC certified logging concession “Compagnie Equatoriale des bois Tropicaux – Precious Wood Gabon” (CEB-PWG). This company is located at Bambidie, Lastourville (Gabon) (0°41.65' S - 12°59.01' E). The average temperature and precipitation are 25 °C and 1700 mm, respectively TerEA (Terre Environnement Aménagement) (Precious Wood Gabon, 2007). Sorro material comes from four different trees.

Hornbeam, Maple and Alder have been bought at Houthandel Vercruysse¹. Each species was coming from one individual due to the availability of dried wood. All the material was already naturally dried and did not need any artificial drying before thermal treatment and use for testing.

i. Treatment

Treated samples have received a VACWOOD® treatment (previously detailed in the state of the art) in a Maspel oven (Model TVS 6000 WDE Maspell srl, Terni, Italy) in Scidus (Etalle, Belgium) (figure 9). The four species have received two treatments identical as a treatment applied to *Triplochiton scleroxylon*. The temperature of the treatment progressively reaches 215 °C and when arrived at this temperature it stabilized for three hours (Gennari and al., 2020). After the three hours, the wood is cooled down. The species were all placed together in the same oven.



Figure 9: Maspel oven Model TVS 6000 WDE Maspell srl, Terni, Italy) at Scidus company, Etalle, Belgium.

¹ <https://www.houtvercruysse.be/nl/houthandel>

B. Physico-mechanical properties

Physico-mechanical tests will be conducted to characterize the different species and the treatment effect. The different tests are following standards to find differences or patterns through the results. Due to a limit of time, all recommended physico-mechanical test will not be done. A selection of tests has been made at the beginning of the master thesis.

i. Sample preparation

The samples for the physico-mechanical tests were cut and prepared following the ISO 3349:1975 (wood—determination of the elasticity modulus in static flexion), ISO 3133:1975 (wood—determination of the resistance to static flexion) and ISO 3348:1975 (wood—determination of the resilience to flexion) standard. Defect-free samples were selected according to the ISO 3129:2012 standard (Wood—Sampling methods and general requirements for physical and mechanical testing of small clear wood specimens), considering essentially samples:

- quarter-sawed
- straight grain
- no defect (knot, slot, etc.)

The sorro will have 59 untreated samples tested and 26 treated samples for both MOE/MOR and impact bending strength. For each temperate specie, 10 samples will be tested for the MOE and MOR and 10 for the impact bending strength.

a) Sorro cutting process

27 boards of 30*110*2200 mm³ (R,T,L) were received in Gembloux (annex 3). Figure 10 presents the cutting process of the 27 boards. Each board of 2200 mm was cut into 5 pieces of 430 mm in length. Two pieces out of five were sent to Scidus to be thermally treated in their ovens.

It was then brought back to the wood technology laboratory (LTB) to follow the same cutting process as the rest of the untreated pieces. All the pieces were planned on one face and one field then the second face was planned with a planer to attain a thickness of 27 mm. Each piece was cut into 4 bars of 27*30*430 mm³ (R,T,L) using a circular saw. For each initial board, 3 pieces of each modality (treated and untreated) that respects the criteria of the standards will be selected (figure 10).

It makes a total of 6 bars selected per board (3 treated bars and 3 untreated bars). Those bars were progressively planned to arrive at the dimension of 27*27*430 mm³ (R,T,L) using a planer. The 162 bars (6*27 planks) were placed in a conditioned room at 20 °C and 65% relative humidity conformed to the ISO 3129:2012 standard for 4 weeks (figure 10).

The figure 11 shows the label to trace each bar. The first step consists of writing the identification number of the tree and the radial position. Then each piece cut from the plank gets a letter. Finally, each bar gets another number to identify it.

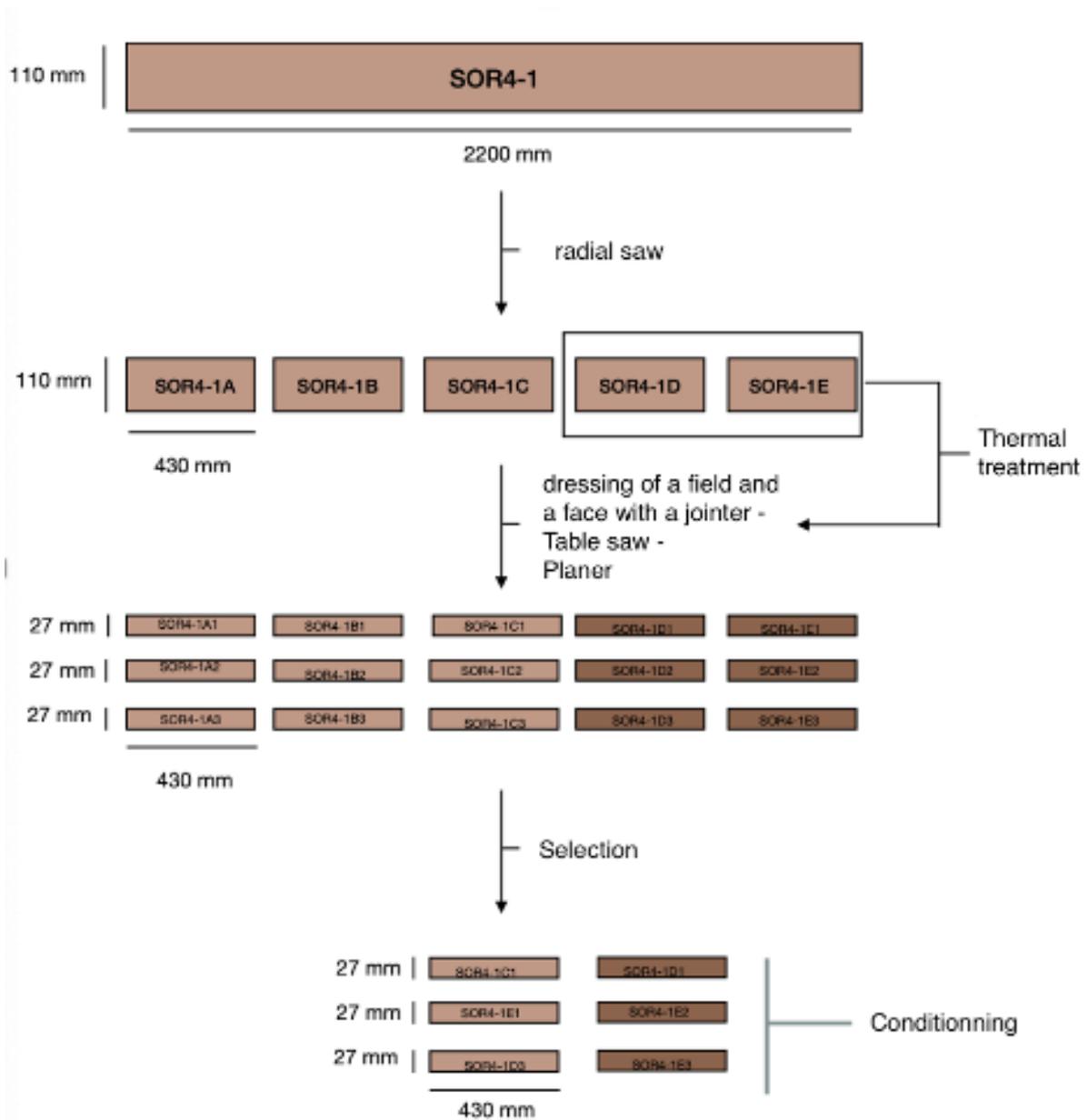


Figure 10: Cutting scheme for the physico-mechanical tests before conditioning

When the samples reach the equilibrium at 20 °C and 65% relative humidity, the bars can be cut to final dimensions. The bars for MOE, and impact bending strength will be planned to 20*20 mm² (R,T) making sure they stay quarter sawed. The bars for MOE and MOR will be cut to 3600 mm length. The bars for the impact bending strength will be cut to 3000 mm in length. On each bar, after being tested, a sample of 20*20*20 mm³ (R, T, L) will be collected to calculate the MC of the bar and an “H” will be added to those humidity samples (figure 11). Moisture content has been measured following the standard NBN EN 13183-1:2002 (moisture measurement by desiccation).



Figure 11: Cutting scheme for MOE/MOR and impact bending strength test and humidity samples (H)

b) Temperate species cutting process

The tree was cut plain sawed then cut into 450 mm of length planks using a chainsaw. After that, every plank has been cut and planed into $30 \times 30 \times 450 \text{ mm}^3$ (R,T,L) bars before being placed into a conditioning room at 20 °C and 65% relative humidity conformed to the ISO 3129:2012 standard for 4 weeks. Each plank got a number with the first letter of the French tree name starting on the plain (figure 12). The second number was attributed to each bar cut in every plank. A “T” was added to the thermal modified wood bars. For example, C1-4T comes from the first plank cut and it is the 4th bar cut into that plank, then the bar has been heat treated.

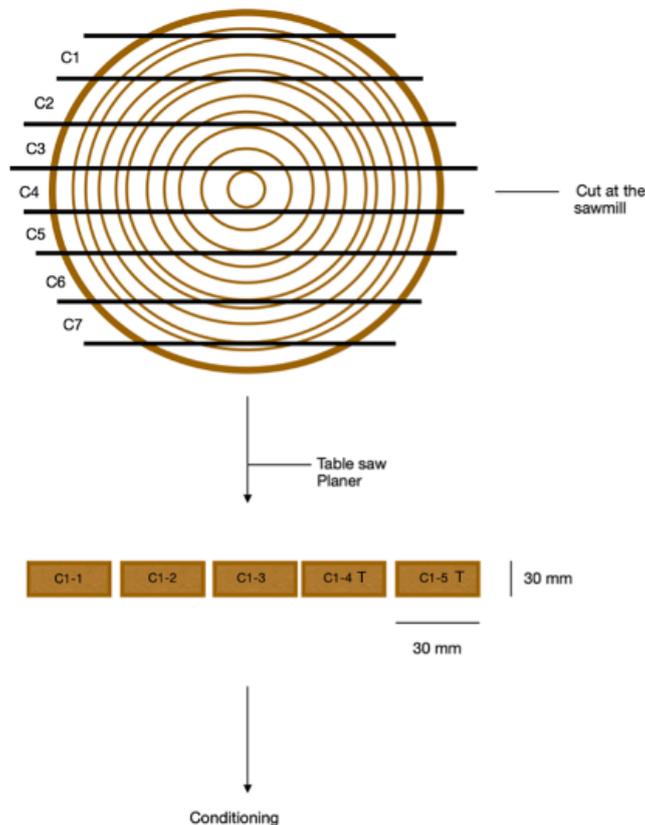


Figure 12: Cutting scheme of temperate species before conditioning

When the samples reach the equilibrium at 20 °C and 65% relative humidity, they will follow the same cutting process as the sorro (previous section) but it will follow their tracing code (for example with hornbeam in figure 13).



Figure 13: Cutting scheme for MOE/MOR and impact bending strength tests and humidity samples (H)

ii. Wood density

The wood density has been measured using the standard ISO 3131:1975 (wood—determination of the wood density for physico-mechanical testing). Wood density (kg/m^3) is the relation between mass (kg) and the volume (m^3). The wood density can be found with the equation 1 in the state-of-the-art section. Wood density has been calculated for each sample of the physico-mechanical test. For the MOE/MOR and the impact bending strength samples, the volume is calculated by multiplying the average section (measured in 3 different points) by the length (mm).

iii. Modulus of elasticity (MOE)

The MOE tests have been done following the standard ISO 3349:1975. The MOE is founded using the arrow of a sample with a progressive load applied in the pure flexion section of it. The progressive charge must be applied to a radial direction of the sample. The tests are run with a machine Instron® 5582. The load is applied 3 times and the MOE is found by doing the average of the three repetitions. The test is a four-point method, two supports and two application points. The dimensions of the samples for the MOE are $20 \times 20 \times 360 \text{ mm}^3$ (R,T,L). The report of the distance between the two application points and the two support points is $\frac{1}{2}$.

The MOE (MPa) is given in the equation 2. The equation is composed of the charge (P) in N, the distance between the two support points (I) in cm, the radial and the tangential sections of the sample (b and h) in mm and the arrow (f) in mm (ISO3349,1975).

Equation 2: Modulus of elasticity (MPa)

$$MOE = \frac{3PI^3}{64bh^3f}$$

iv. Modulus of rupture (MOR)

The MOR tests have been done following the ISO 3133:1975. The MOR is founded with a continuous load applied with a constant speed until the sample breaks. The continuous charge must be applied to a radial direction of the sample. The dimensions of the samples for the MOE are 20*20*360 mm³ (R,T,L). The load is applied at halfway from the support point using a machine Instron® 5582.

The MOR (MPa) is given in the equation 3. This equation is composed of the rupture load (P_{max}) in N, the distance between the support (l) in mm, the average width (b) and the average height of the sample (h) in mm (ISO3133, 1975).

Equation 3: Modulus of rupture (MPa)

$$MOR = \frac{3P_{max}l}{2bh^2}$$

v. Impact bending strength (IBS)

The impact bending strength test has been realized using the standard ISO 3348:1975. This test measures the absorbed energy to induce the breaking point of the sample in one hit. The energy must be applied to a radial direction of the sample. The machine used is a “mouton pendule”, equipped with a 15 kg hammer. The dimension of the samples for the impact bending strength tests are 20*20*300 mm³ (R,T,L).

The resilience (K_h) in kg/cm² for a certain moisture content (h) is given by the equation 4. This equation involved the work done by the pendulums to break the sample (W_h) in kg, the average radial and tangential dimension (b and h) in cm (ISO3348,1975).

Equation 4: impact bending strength (kg/cm²)

$$K_h = \frac{W_h}{bh}$$

C. Durability screening tests

Durability should be tested following the EN 113-2 (2020) against the fungus and EN 117 (2013) against termites. As performing those tests requires a lot of time, screening tests were applied to provide some results. These screening tests require smaller samples than the standardized ones, final dimensions are 25*15*5 mm³ (R, T, L) for both termites and fungi (figure 14). Tests bars were cut in 25*15 mm² (R,T) then 6 samples of 5 mm length were collected on each bar of each species for the 2 treatment parameters (treated and not treated). 48 samples were cut per species and per treatment.



Figure 14: samples for durability tests (fungi and termites)

i. Samples preparation

To realize a screening test, micro samples of $25 \times 15 \times 5 \text{ mm}^3$ (L, R, T) were cut (figure 15). One bar will be collected on each piece of the board selected before (section 2) for each modality, treated and untreated. They were conditioned with the rest of bars in the same conditioned room at $20 \text{ }^\circ\text{C}$ and 65% relative humidity conformed to the ISO 3129:2012 standard for 4 weeks before being cut in the screening tests dimensions.

After four weeks, when the untreated bars are stabilized in the conditioning room at $20 \text{ }^\circ\text{C}$ and 65% relative humidity, they were planned to obtain $25 \times 15 \text{ mm}^2$ (L, R) bars. After being planned to dimension, they were crosscut to 5 mm (T) of length using a miter saw (figure 15). When the samples were ready, they were placed into the envelope and put back into the same conditioning room at $20 \text{ }^\circ\text{C}$ and 65% relative humidity.

Arriving at the CIRAD in Montpellier, all the micro samples were taken out and the edges were lightly sand to cut the sharp edges and labelled. For example, with the sorro SOR4-1D3 was cut into 6 micro samples named SOR4-1D3-1, SOR4-1D3-2, up to 6 (figure 14).

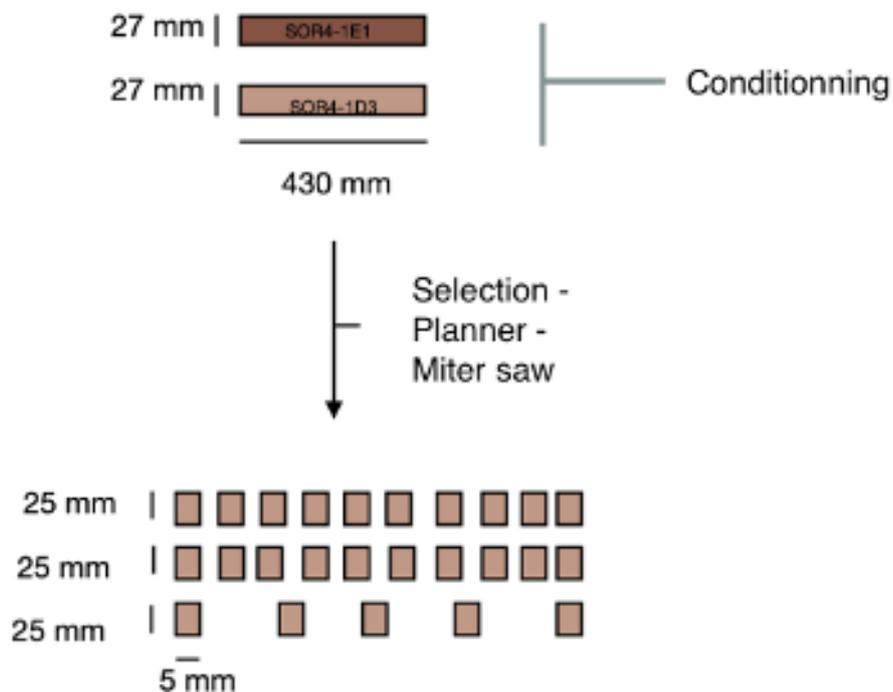


Figure 15: durability samples preparation

The samples needed to be cut 25*15*5 mm³ (L, R, T) but have been cut 25*15*5 mm³ (R, T, L) (figure 16). This change needs to be considered for the results and the manipulation. The biggest impact is the humidity intake during the test and the fragility during the drying of the samples. The samples can crack while drying in the oven. Due to that, the samples need to be dried outside the oven for 24 hours before being placed in at 103 °C in the oven.



Figure 16: Difference between an L, R, T sample (top picture) and an R, T, L sample (bottom picture)

ii. Fungal tests

Two fungi have been tested: *C. puteana* and *T. versicolor*, both Basidiomycetes. *C. puteana* impacts both softwood and hardwood and is an obligatory fungus to determine the natural durability and the durability of heat treated timber (EN 113-2, 2020). On the other hand, *T. versicolor* impacts mainly hardwood, and it is also an obligatory fungus for the durability evaluation. *C. puteana* and *T. versicolor* are cultured and kept in a wood preservation laboratory of BioWooEB.

One hundred flasks were prepared for fungal tests: 50 flasks for *C. puteana* and 50 flasks for *T. versicolor*. The hundred flasks were prepared with a solution of malt and agar (40 g malt powder, 20 g of agar/liter of deionized water). Then, 50 ml of solution was placed in each flask to have 3–4 mm of solution in each flask once placed horizontally. The caps had a hole in the center filled with carded cotton and was placed back on the flask once it was filled up with the solution. The flasks are then put into an autoclave at 121 °C and 1 bar to sterilize for 20 minutes. One flask per batch is marked to have a quality control and prove that the flasks have been sterilized in an autoclave (figure 18). When the autoclave treatment is done, the flasks are placed horizontally on a table to cool down for 4 hours and let the agar-malt solution solidify (figure 17).



Figure 17: fungal test preparation. A: solution preparation. B: flasks placed. Horizontally to cool down. C, D: cotton caps.



Figure 18: autoclave process. A: control label to prove that the samples have been autoclaved. B: autoclave system

The fungi in the Petri dishes (where they are grown and kept) are being cut into 1*1 cm² and 2 samples are placed in each flask (figure 19). When the fungus has covered the medium, three samples (treated or untreated) and a control sample are placed in one flask (figure 19). Virulence control flasks will contain 4 control samples. *Fagus sylvatica* samples are used to test the virulence of *T. versicolor* and *Pinus sylvestris* to control *C. puteana*. Two kinds of control flasks are done:

- Virulence controls flask with normal micro-samples 25*15*5 mm³ (L, R, T);
- Control flask using 25*15*5 mm³ (R, T, L) to control the impact of the orientation.

All the flasks are placed in a conditioning room at 70% relative humidity and 22 °C for 8 weeks.

Before being placed in the flask, all the samples are measured and weighed a first time (M_h) once conditioned at 20 °C, 65% relative humidity. After that the samples are being placed in an oven at 103 °C for 48 hours then weighted again to obtain the anhydrous mass of the samples (M_0).

After 8 weeks, the samples are taken out of the flask, cleaned and weighed (humid, weight straight after the fungal test) and placed 24 hours at room temperature and then in an oven at 103 °C until it reaches a constant mass. We get the final anhydrous mass (M_f). The difference between the initial anhydrous mass and the final anhydrous mass is used to calculate the mass loss (%) (equation 5).

Equation 5: mass loss for fungal test

$$\text{Mass loss} = \frac{M_0 - M_f}{M_0} \times 100$$

The results of the mass losses should be linked to the table of the standard EN 113-2 (2021) to determine the durability of the sample against the fungi. Given the fact that the samples are not the same as the standard EN 113-2 (2021) (orientation, size, time of exposure), the comparison cannot be established. The standard EN 350-1 (1994) is then used to compare the sample (R, T, L) to the control samples that are the same orientation (R, T, L) using the table of the standard (table 2).

Table 2: relation between the mass loss (%) and the durability class from the EN 350-1 (1994) standard

Durability class	Description	Results of the laboratory test in X*
1	Very durable	$X \leq 0,15$
2	durable	$X > 0,15$ et $X \leq 0,30$
3	moderately durable	$X > 0,30$ et $X \leq 0,60$
4	not very durable	$X > 0,60$ et $X \leq 0,90$
5	Very not durable	$X > 0,90$
*X=Mean of mass loss for the testing samples / Mean of mass loss for the control samples		



Figure 19: Fungal test. Flask preparation with *Coniophora puteana* (CP). The top picture represents a fungal preparation and the bottom picture shows a fungal test after 8 weeks.

iii. Reticulitermes termite tests

Reticulitermes flavipes (ex. *santonensis*) termites are captured in Oleron island (France) then kept in breeding boxes in CIRAD Montpellier in a conditioned room at 27 °C and 80% relative humidity. They are kept in plastic breeding boxes. The boxes are filled with ground, sand and poplar wood to feed the termites (figure 20).



Figure 20: termites' storage at CIRAD, Montpellier

Petri dishes are prepared with wet Fontainebleau sand. The proportions are one quarter of water and three quarters of sand. Thirty grams of wet sand is weighted with a balance Mettler PM100 and placed in each Petri box around the center of the box, leaving the center empty. The center will be used to place the sample. Fifty workers, one soldier and 1 nymph are placed in each box with the sample. The samples are placed on a small 1.5*1 cm² plastic grid. Once the samples are placed, the Petri dishes are closed and put in the conditioning room at 27 °C, 80% relative humidity and left for 4 weeks. On the cap of the Petri dish, the code of the sample(s) will be written and the date when the termites were being placed with the sample (figure 21).



Figure 21: termites tests. A: choice test. B: conditioned room with the Petri dishes. C: nonchoice test with a treated sample. D: Petri dish with a control sample

Three kinds of tests will be run to determine the resistance of the species and the impact of thermal treatment against termites:

- The first test will be a non-choice test. One sample of each species and each treatment will be placed in a Petri dish with termites. Five repetitions will be done per species/treatment.
- The second test will be a choice test. Two samples of the same species (one treated and one untreated) will be placed in a Petri dish to see the preference of the termites. There will be five repetitions per species.
- The third and last test will also be a choice test. One sample of each species will be placed in a Petri dish. All the treated samples will be placed in the same Petri dish and all the samples untreated will be placed in other Petri dishes. There will be five repetitions per treatment. This will be a test to observe the preference of termites for each species, native or thermal treated.

Same as the fungi test, virulence control samples will be tested to see if the termites are virulent. The virulence tests will be tested with *Pinus sylvestris* sapwood because all the species are hardwood species. Same as the fungi virulence control test the two kinds of screening samples will be tested (25*15*5 mm³ (L, R, T) and 25*15*5 mm³ (R, T, L)).

To estimate the durability of the wood against termites, a visual rating according to EN117 (2013) (but adapted to the sample size) will be given to each sample. The mass loss will also be determined. Besides that, dead termites will be counted in each Petri dish and the survival rate will be calculated.

The results of the visual rating can be linked to the table of the standard EN 350 (2016) to determine the durability of the sample against termites (table 3).

Table 3: classes of durability of wood species and wood-based materials to termite attack based on EN 350 (2016) ratings

Durability class	Description	Rating
DC D	Durable	≥ 90% “0 or 1” and max 10% “2” *
DC M	Moderately durable	<50% “3, 4”
DC S	Not durable	≥ 50% “3, 4”
*90% of the test samples rated 0 or 1 and a maximum of 10% of the test samples rated 2 and 0% “3 and 4”		

D. Data treatment and statistics

i. Software

Data treatment has been done with the two software RStudio 2022.07.1+554® using various packages (annex 4), and Microsoft Excel®. Untreated data were centralized into different excel depending on the data (MOE, MOR, termites, ...).

ii. Descriptive statistics

To avoid the influence of extreme data and outliers, they have been taken away of the data. The number of samples, the mean, median, standard deviation has been calculated for all parameters

for the different species and different treatment. The mean was chosen to be included in the boxplots.

iii. Variance analyses

Variance was investigated among 4 variables with different modalities:

1. Treatment: treated, untreated;
2. Species: *S. mannii*, *A. glutinosa*, *C. betulus*, *A. pseudoplatanus*;
3. Sorro heartwood colour: clear, dark and mixed;
4. Sorro's streaks presence: presence, absence.

ANOVA analysis was used when application conditions were met (normality and heteroscedasticity). These have been checked using Shapiro test and Barlett test, respectively. If significant difference was highlighted by the F-test, means were compared using student t-test, using an alpha (α) = 0.05. When conditions were not met, the test of Kruskal-Wallis is used for all the factors studied. This test is based on the median and not the mean. This test will be used in the place to see if the species or the treatment have an influence on the different parameters studied. If a significative difference appears, the Wilcoxon test will be used to structure the median and see where the significant difference is in the population. The same two test will be used for the rest of the factor and the results.

Boxplots are made to present the results and the impact of the treatment and the species. The p-values, the medians will be added in those boxplots to centralize all the information. The number of samples will be added as well in those boxplots.

4. Results and discussion

A. Visual aspect

It is noticeable that the colour changes when a heat treatment is applied to the wood (Figure 22). The four species selected are originally presenting clear coloured. After the treatment they all become darker. Hornbeam and Sorro are the most impacted by the treatment in terms of colour. Alder is the least impacted. Hornbeam and sorro turns into a dark brown. Alder colour turns into a clear brown. Maple is different of the others. It turns into a shiny golden brown. All details from the untreated samples stay after treatment except for the sorro, it is hard to see the details of the wood after treatment.

Sorro wood was burned before planning and particularly dark after. The maple has a nice shiny golden-brown colour that could give a nice visual aspect for indoor use. Hornbeam also has a good-looking visual aspect. It is darker than maple. All the aesthetics from untreated hornbeam are still present and mix well with a darker colour. Those colour can be a good factor in the industry but it is important to remind us that wood turns grey when aging outside.



Figure 22 : comparison of each species before (left) and after treatment (right). The species are organized from left to right ; hornbeam, alder, maple and sorro.

B. Physical properties

i. Equilibrium moisture content

The results of the equilibrium moisture content are given in figure 23 and annex 1. Table 4 summarizes the changes of equilibrium moisture content between treated and untreated wood.

The results reveal an important diminution of the equilibrium moisture for the four species (table 4). Sorro is the species that has the lowest equilibrium moisture after treatment (figure 23). The thermal treatment has a highly significant impact on the equilibrium moisture of the wood ($W = 576$; $p\text{-value} < 0.0001$; $W = 576$; $p\text{-value} < 0.001$; $W = 576$; $p\text{-value} < 0.001$; $W = 576$; $p\text{-value} < 0.0001$) (figure 23).

Cao and al. (2022) explain the diminution of hygroscopicity by the diminution of hemicelluloses that present many hydroxyl groups. This reduction of the hydroxyl group and therefore hygroscopicity in the wood makes it lose a part of its moisture exchange capacity. It induces a diminution of equilibrium moisture in thermal treated wood.

Priadi and al. (2019) also mentioned that the diminution of hygroscopicity is correlated with the increase of treatment temperature.

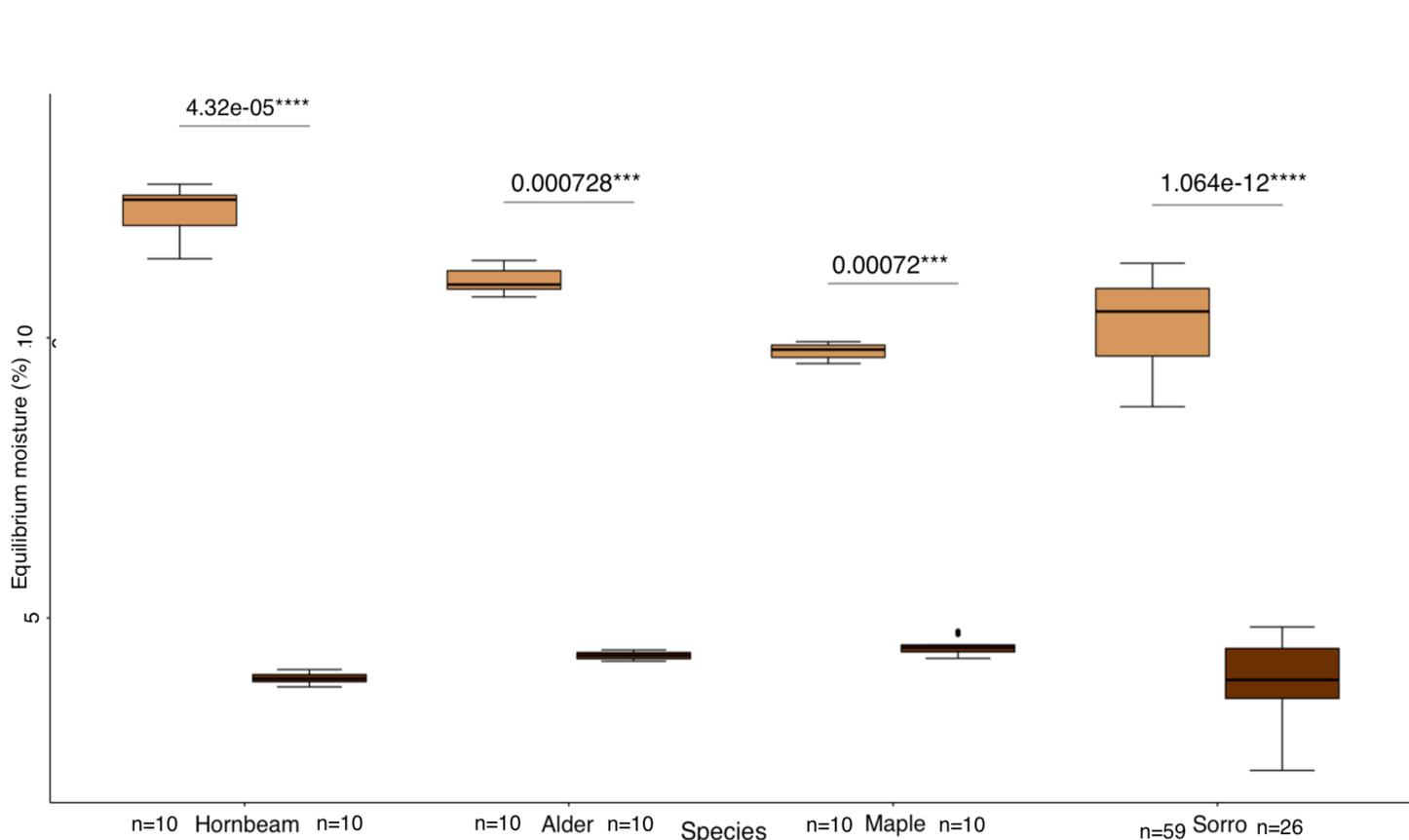


Figure 23 : Boxplots of the impact of species and treatment on equilibrium moisture (%). Wilcoxon test is used to compare the species/treatment. The p-value is added if there is a significant difference between two boxplots with the degree of significance

Table 4 : Relative difference (Δ) of the median values of the equilibrium moisture (%) of treated specimens compared to control specimens (%)

Species	Δ median of equilibrium moisture (%)
Hornbeam	-68,57
Alder	-60,44
Maple	-54,30
Sorro	-62,77

ii. Wood density

The results of the wood density are given in figure 23 and annex 2. Table 5 summarizes the changes of wood density after thermal treatment.

Wilcoxon test show that thermal treatment has a significant impact on alder, maple and sorro (W= 90; p-value <0.001; W=63; p-value <0.05; W=1414; p-value = 0.0001) (figure 24). There is no significant impact of thermal treatment on hornbeam (p> 0.05).

Wood density was taken at equilibrium MC of each specie. The most impacted is alder with a median diminution of 16.77% and the less impacted is hornbeam with a median diminution of 2.58%. Maple and sorro wood density median decrease by 5.47% and 9.52% (table 5).

Boedts (2016) reminds the fact that humidity is important in the wood density calculation. The mass of the samples is correlated with their humidity. Mass decreases more when treatment temperature rises and the equilibrium moisture content decreases also. Besides the mass change, the volume also varies at humidity under the FSP. To remove the humidity factor, infra-density needs to be measured. Repelin (2006) mentions that the FSP is influenced by the temperature of treatment. The effect on the FSP influences the saturated volume. Infra-density can remove the humidity factor but not the saturated volume. The anhydrous wood density is the parameter chosen to remove both humidity and saturated volume factors. Boedts (2016) concludes that the diminution of anhydrous wood density is correlated to the temperature of treatment. As the results of infra density and the anhydrous wood density are not yet available, the conclusion of the four species will be based on the conclusion of Boedts (2016). Furthermore, when thermally treated wood in use normally will never reach the moisture of untreated wood, it's still interesting to compare results at equilibrium moisture.

As expected, wood density decreases when the timber is thermally treated due to mass loss. The percentages of decrease are different for each species. Those results can be linked with the intensity of the treatment (temperature and time).

Gunduz and al. (2009) linked the wood density loss to the intensity of the treatment for hornbeam wood. In their results, the wood density mean diminution is between 0.76% (treated at 170 °C for 4 hours) and 16.12% (treated at 210 °C for 12 hours). In this case, for hornbeam, the median diminution of 2.58% (twice 215 °C for 3 hours) let suggest that this treatment was not adapted.

Bovurka and al. (2015) showed wood density means diminution of 2% (165 °C for 3 hours) and 10% (210 °C for 3 hours) for alder wood. In this case, for alder, the wood density median decreases by 16.77% when heat treated (twice 215 °C for 3 hours). The important wood density median diminution, for alder, let supposed that the treatment was not adapted to this species.

To compare the results of sorro and maple wood density allows to conclude if the treatment applied was adapted or not. Sorro wood density median decreased by 9.52% (twice 215 °C for 3 hours). This result shows that the treatment was not adapted. Maple wood density decreased by 5.47% (twice 215 °C for 3 hours) which shows that the thermal treatment was adapted for this specie.

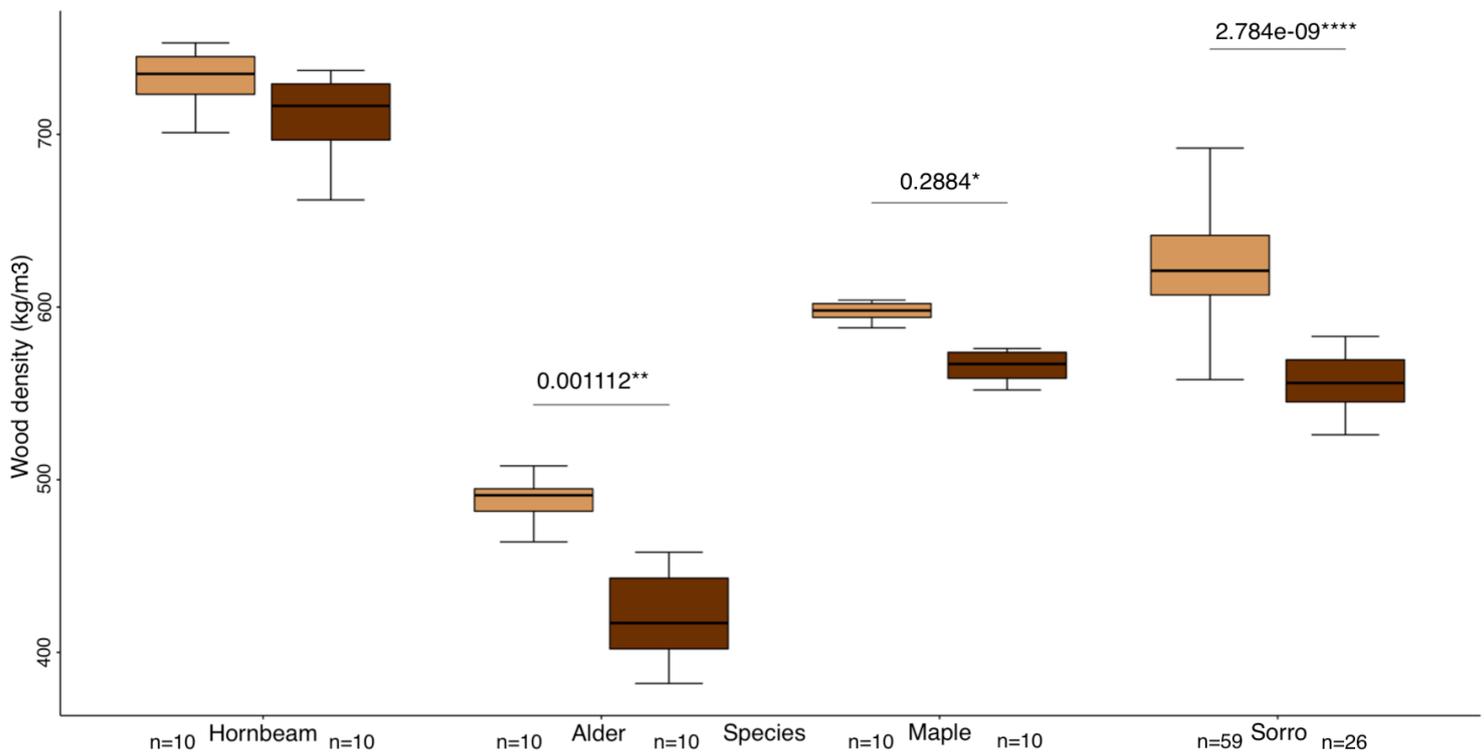


Figure 24 : Boxplots of the impact of species and treatment on wood density (kg/m³). Wilcoxon test is used to compare the species/treatment. The p-value is added if there is a significant difference between two boxplots with the degree of significance

Table 5 : Relative difference (Δ) of the median values of the wood density of treated specimens compared to control specimens (%)

Species	Δ Median of wood density (%)
Hornbeam	-2,58
Alder	-16,77
Maple	-5,47
Sorro	-9,52

C. Mechanical properties

i. Modulus of elasticity

The results of the MOE (MPa) are given in figure 25 and annex 2. Table 6 summarizes the changes of MOE (MPa) due to the thermal treatment.

Wilcoxon test reveals a significative difference ($W = 79$; $p\text{-value} < 0.05$) between the MOE of treated and untreated alder (figure 25). For the other species, there are no significant differences.

The most rigid is maple with a MOE median of 12,831 MPa, closely followed by hornbeam with a median of 11,835.5 MPa. The most flexible species is alder with a median of 8904.5 MPa (untreated) and 7039 MPa (treated) (annex 2). It is interesting to notice that all the MOE decrease when heat treated except for hornbeam. Hornbeam MOE increases. The most impacted by heat treatment is alder with a significant decrease of 20.95% (table 6).

A rating of the MOE mean is done to observe the changes of the parameters considering the diminution of wood density for each species. The rating is obtained by dividing the MOE mean (MPa) by the wood density mean (g/cm^3) (equation 6).

Equation 6: MOE rating

$$MOE\ rating = \frac{MOE\ mean}{\rho\ mean}$$

As previously said, the diminution of wood density serves as a reference of the intensity of the treatment on the species. This will help compare the differences between the species. The rating of the MOE mean, including the diminution of wood density, shows different results. It shows an augmentation for hornbeam and sorro wood. And the tendency of the two most impacted is inversed. The maple has the highest diminution and the alder only decreased of 3.41%. Those results highlight that the treatment was not adapted for sorro and alder. It shows that their MOE median diminution could be the result of their treatment.

Schneid and al. (2014) and Calonego and al. (2012) showed similar studies about thermally treated wood for two other species and it showed that thermal treatment has a light impact on MOE. The results show a light diminution of MOE for Sorro wood and an augmentation for hornbeam wood. The only significant impact is on sorro.

Militz (2008) states that if the thermal treatment intensity is light, the MOE increase but it decreases when the treatment is stronger. If we look at the MOE median and the MOE rating, it is possible to conclude that an adapted thermal treatment can increase the MOE if it is not adapted, the MOE starts to decrease.

Bhuiyan and al. (2000) explain that thermal treatment increases the crystallization of cellulose and show evidence of the impact on elasticity. Crystalline cellulose form bonds with water molecules. This leads to a higher hygroscopicity then better dimensional stability. MOE's different change between the different species after treatment can be influenced by the quantity of crystallize cellulose.

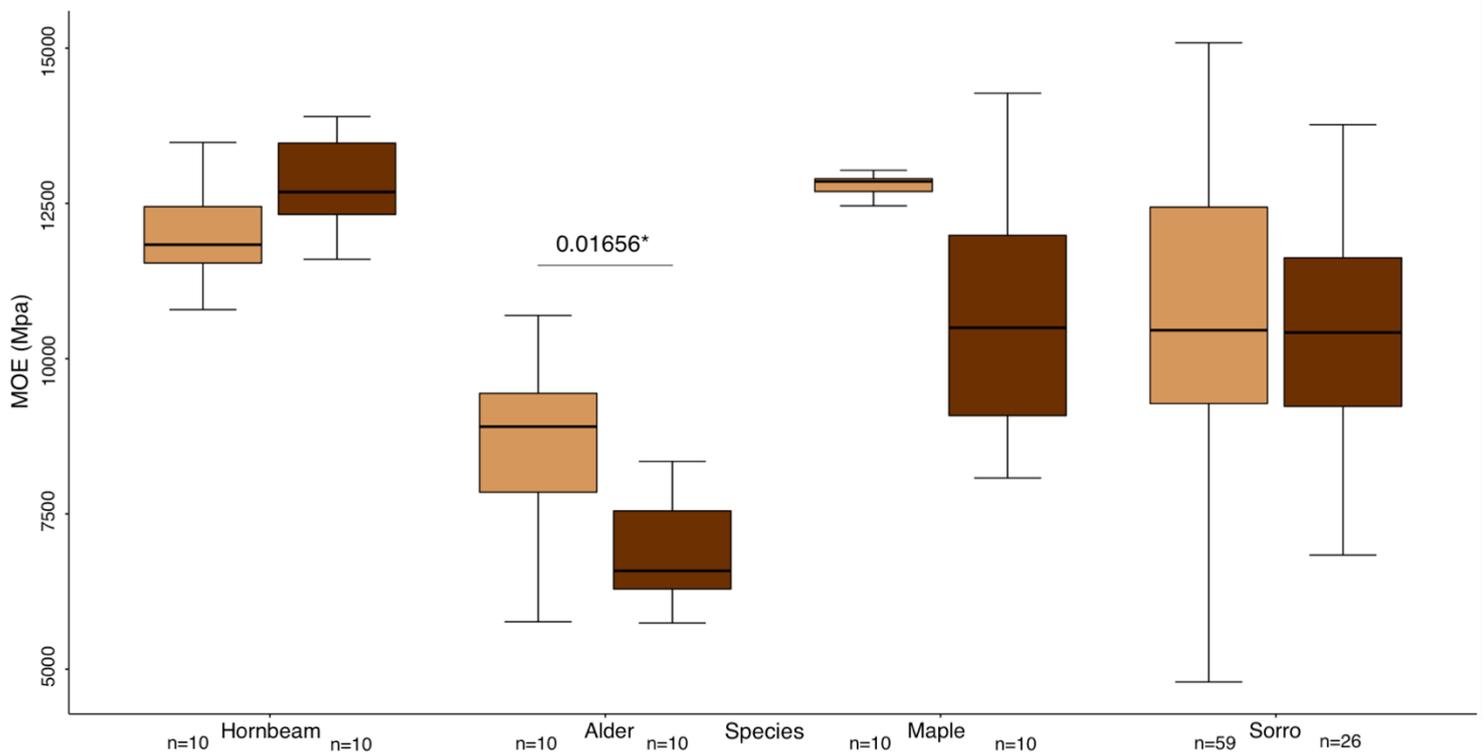


Figure 25 : Boxplots of the impact of species and treatment on modulus of elasticity (MPa). Wilcoxon test is used to compare the species/treatment. The p-value is added if there is a significant difference between two boxplots with the degree of significance

Table 6 : Relative difference (Δ) of the median values of the MOE (MPa) and the mean of MOE rating of treated specimens compared to control specimens (%)

Species	Δ median of MOE (%)	Δ mean of MOE rating (%)
Hornbeam	+ 7,15	+ 9,72
Alder	- 20,95	- 3,41
Maple	- 18,19	- 9,26
Sorro	- 0,35	+ 5,44

ii. Modulus of rupture

The results of the MOR (MPa) are given in figure 26 and annex 2. Table 7 summarizes the changes of MOR (MPa) after thermal treatment.

Wilcoxon test shows a highly significant impact of heat treatment on the MOR of all species (W = 88; p-value <0.001; W= 73; p-value <0.05; W = 90; p-value <0.0001; W = 1351; p-value <0.0001) (figure 26).

A reduction of MOR is observed for the four species when heat treated (figure 26). When no treatment is applied, maple is the most resistant to rupture with a median of 116.9 MPa. Once it is treated, hornbeam becomes the most resistant to rupture with a median of 82.85 MPa (annex 2). It is also the one that is the least impacted by the treatment. Its MOR median is reduced by 25.49%. The most impacted by treatment is maple with a MOR median reduction of 49.61% (table 7). Alder is the least resistant species to rupture before and after thermal treatment. Van Blokland and al. (2018) show the fact that it is the changes of the cell's walls component that the mechanical properties change.

A rating of the MOR mean has been done to observe the changes of the parameters and considering the diminution of wood density for each specie. The rating is obtained by dividing the MOR mean (MPa) of a specie/treatment by the wood density means of the same specie/treatment (g/cm³) (equation 7).

Equation 7: MOR rating

$$MOR\ rating = \frac{MOR\ mean}{\rho\ mean}$$

When looking at the MOR median rating, the results are similar to the MOR median diminution. This show that the least impacted are hornbeam and alder. Maple MOR median is highly impacted by thermal treatment.

The thermal treatment has a significant negative impact on the MOR median for all four species. Those diminutions are confirmed by Schneid and al. (2014) results that show a diminution of the MOR when treated at 200 °C. The least impacted is hornbeam again. The tendance changes for the rest of the species. Maple is the most impacted followed by the sorro and finally alder wood.

Repellin (2006) attests that thermal modified wood is more fragile than untreated wood. Van Blokland and al. (2018) reinforce this statement by pointing to the fact that the changing degree depends on the species but also the temperature of the applied treated process. In this case, the species is a factor but the treatment process (twice 215 °C for 3h) is also an important factor. Each specie needs a particular thermal treatment. This treatment is probably not adapted for all four species.

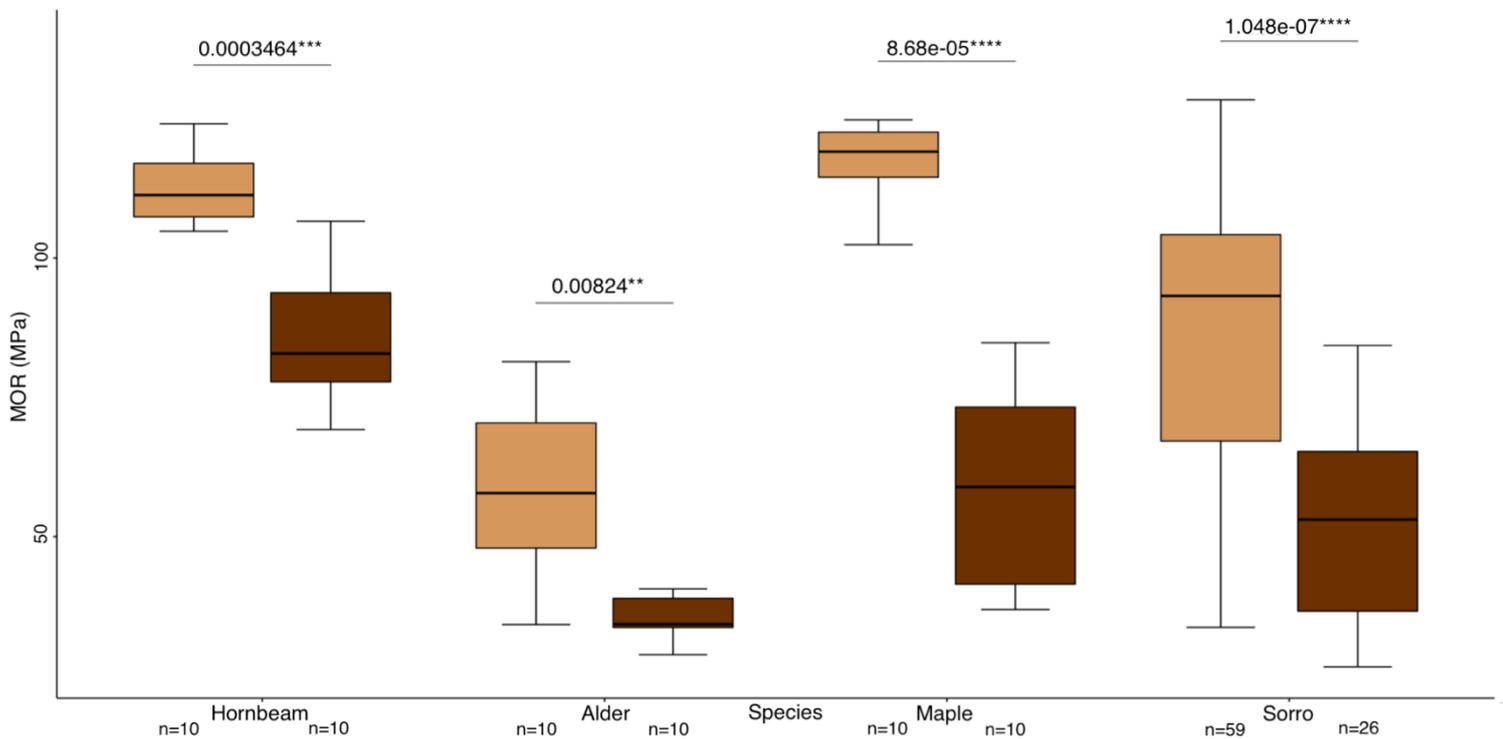


Figure 26 : Boxplots of the impact of species and treatment on the modulus of rupture (MPa). Wilcoxon test is used to compare the species/treatment. The p-value is added if there is a significant difference between two boxplots with the degree of significance.

Table 7 : Relative difference (Δ) of the median values of the MOR (MPa) and the mean of MOR rating of treated specimens compared to control specimens (%)

Species	Δ median of MOR (%)	Δ mean of MOR rating (%)
Hornbeam	- 25,49	- 20,40
Alder	- 40,74	- 26,67
Maple	- 49,61	- 46,50
Sorro	- 43,08	- 33,73

iii. Impact bending strength

The results of the impact bending strength (kg/cm^2) are given in figure 27 and annex 2. Table 8 summarizes the changes of impact bending strength (kg/cm^2) after thermal treatment.

Except alder ($p\text{-value} > 0.05$), Wilcoxon test shows that all shows a highly significant difference before treatment and after treatment for the impact bending strength ($W = 99.5$; $p\text{-value} < 0.001$; $W = 79.5$; $p\text{-value} < 0.05$; $W = 1498$; $p\text{-value} < 0.0001$) (figure 27).

The greatest impact of heat treatment is observed on the impact bending strength. The most resistant species before and after treatment is maple with a median of $0.53 \text{ kg}/\text{cm}^2$ and $0.34 \text{ kg}/\text{cm}^2$ respectively. The least resistant before and after treatment is alder with a median of $0.19 \text{ kg}/\text{cm}^2$ and $0.13 \text{ kg}/\text{cm}^2$ (table 8). There is no significant diminution in alder impact bending strength. The biggest diminution is observed with hornbeam. Its impact bending strength decrease by 58.33% (table 8). The tendency is the opposite than the wood density diminution. The bigger the wood density diminution is the smaller the impact on the impact bending strength is.

A rating of the impact bending strength mean has been done to observe the changes of the parameters and considering the diminution of wood density for each specie. The rating is obtained by dividing the impact bending strength mean (kg/cm^2) of a specie/treatment by the wood density means of the same specie/treatment (g/cm^3) (equation 8).

Equation 8: impact bending strength rating

$$\text{IBS rating} = \frac{\text{IBS mean}}{\rho \text{ mean}}$$

The diminutions are all high but when looking at the rating of the impact strength building alder and sorro have a significant smaller diminution than the other species. It means that those two species impact bending strengths are high because of the intensity of the treatment but they are less infected by thermal treatment than hornbeam and maple. Hornbeam is under treated but still gets the biggest diminution of impact bending strength median.

Boruvka and al. (2015) show similar results for alder and douglas fir. For a thermal treatment at $165 \text{ }^\circ\text{C}$, the diminution of impact bending strength is light but when the temperature of the treatment is $210 \text{ }^\circ\text{C}$, there is a high diminution of the impact bending strength. In this case, the diminution is especially important. The treatment was not adapted to those species. A lower temperature of treatment could be suggested.

The diminution of impact bending strength after treatment are related to the conclusions of Repellin (2016) and Van Blokland and al. (2018) cited in the previous section for the MOR.

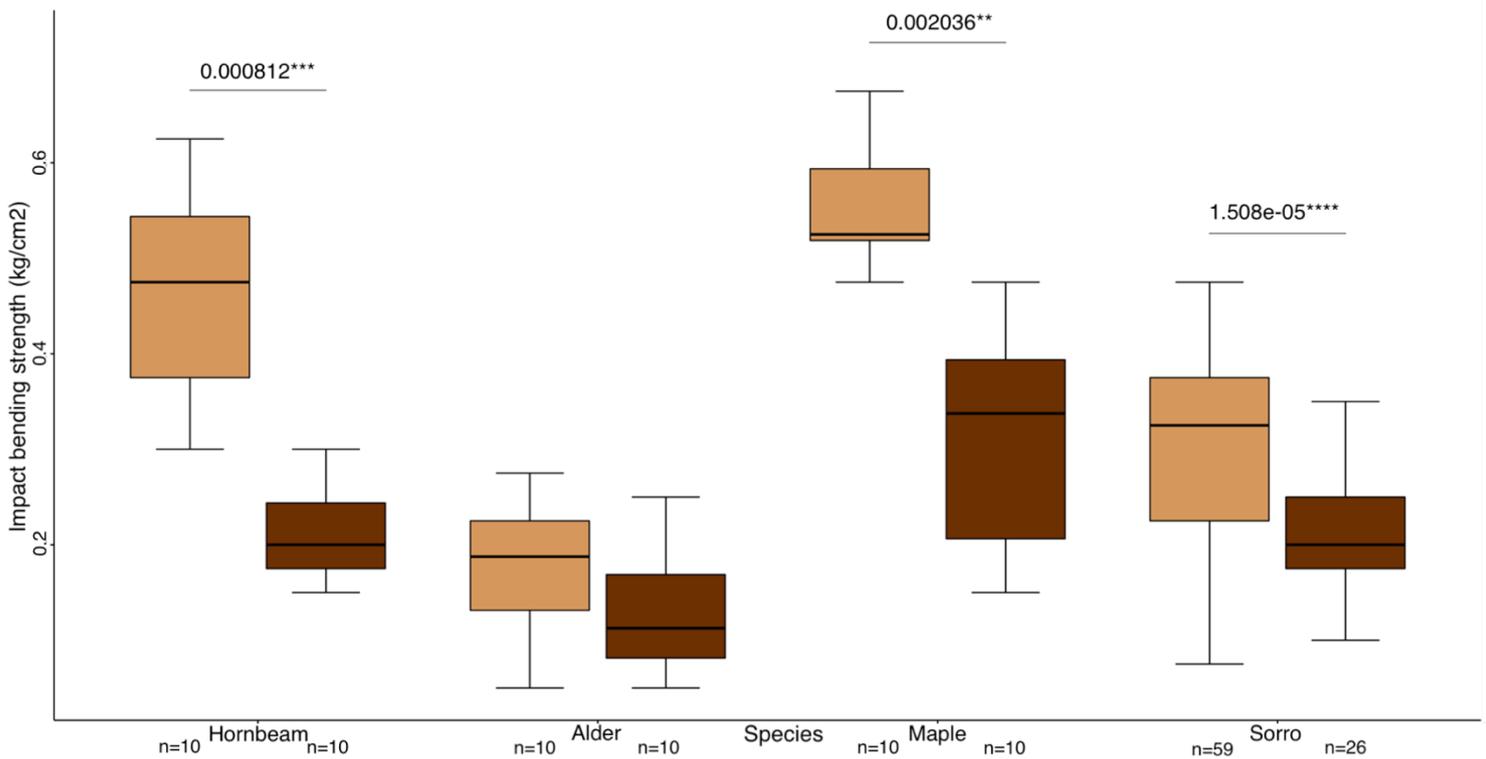


Figure 27: Boxplots of the impact of species and treatment on the impact bending strength (kg/cm²). Wilcoxon test is used to compare the species/treatment. The p-value is added if there is a significant difference between two boxplots with the degree of significance.

Table 8 : Relative difference (Δ) of the median values of the impact bending strength (IBS) (kg/cm²) and the mean of impact bending strength rating of treated specimens compared to control specimens (%)

Species	Δ median IBS strength (%)	Δ mean of IBS rating (%)
Hornbeam	-58,33	- 22,92
Alder	-31,58	- 16,22
Maple	-35,85	- 37,63
Sorro	-39,39	- 19,35

D. Impact of heartwood colour in Sorro

The results of the impact of sorro's heartwood colour are given in figure 28 and the results of heartwood streaks in figure 29.

In the previous sections, it is noticeable that the boxplots of the sorro for each parameter are more spread than the other species. Wilcoxon test has been used to check if the colour of the heartwood and the presence of purple heartwood streaks can be an explication of the data dispersion but also have an influence on different physico-mechanical properties. The results show a significant impact of the colour on the MOE. There is a highly significant difference between the median of clear heartwood and dark heartwood ($W = 95$; p -value <0.001). There is also a highly significant difference between dark heartwood and mixed heartwood median ($W = 63$; p -value <0.05). Furthermore, there is no significant difference between dark and mixed heartwood (figure 27). The impact bending strength is not impacted by the colour of the heartwood or the heartwood streaks, there are no significant differences (p -value >0.05). Another variation of the colour has been studied, the purple heartwood streaks. Those variations of colour have a significant impact on the MOE ($W = 154$; p -value <0.05) and the MOR ($W = 154$; p -value <0.05).

There is a diminution of 32.81% of the MOE median between the clear and the dark heartwood. The dark heartwood has a higher MOE median than the clear one. The mixed heartwood MOE median is between the clear and the dark one (table 28). The presence of heartwood streaks increase the MOE and MOR (figure 28 and 29). There is an augmentation of 18.79% of the MOE median when there is heartwood streaks in the sample. The MOR median increases by 19.85% when there is heartwood streaks in the sample (figure 29).

The results show that the colour of the heartwood only has a significant impact on the MOE median of the sorro. The dark heartwood shows a higher MOE median than the clear heartwood. Mixed heartwood MOE median is between the clear and the dark heartwood which shows the impact of both colours. However, looking at the repartition of the heartwood streaks, they are only present on the samples having a mixed heartwood or dark heartwood. No heartwood streaks have been found in a clear heartwood sample. The results of their influence on the MOE and MOR median are probably due to the heartwood colour and not because of their presence. Further tests need to be done to see what in the dark heartwood impacts those differences.

Moussavou (2021) mention that sorro has a large variety of endophyte fungi. The heartwood streaks could be an endophyte. Their visual appearance is similar. Rodriguez and al. (2009) explain that endophytes increase biomass of the wood and indirectly influence mechanical properties.

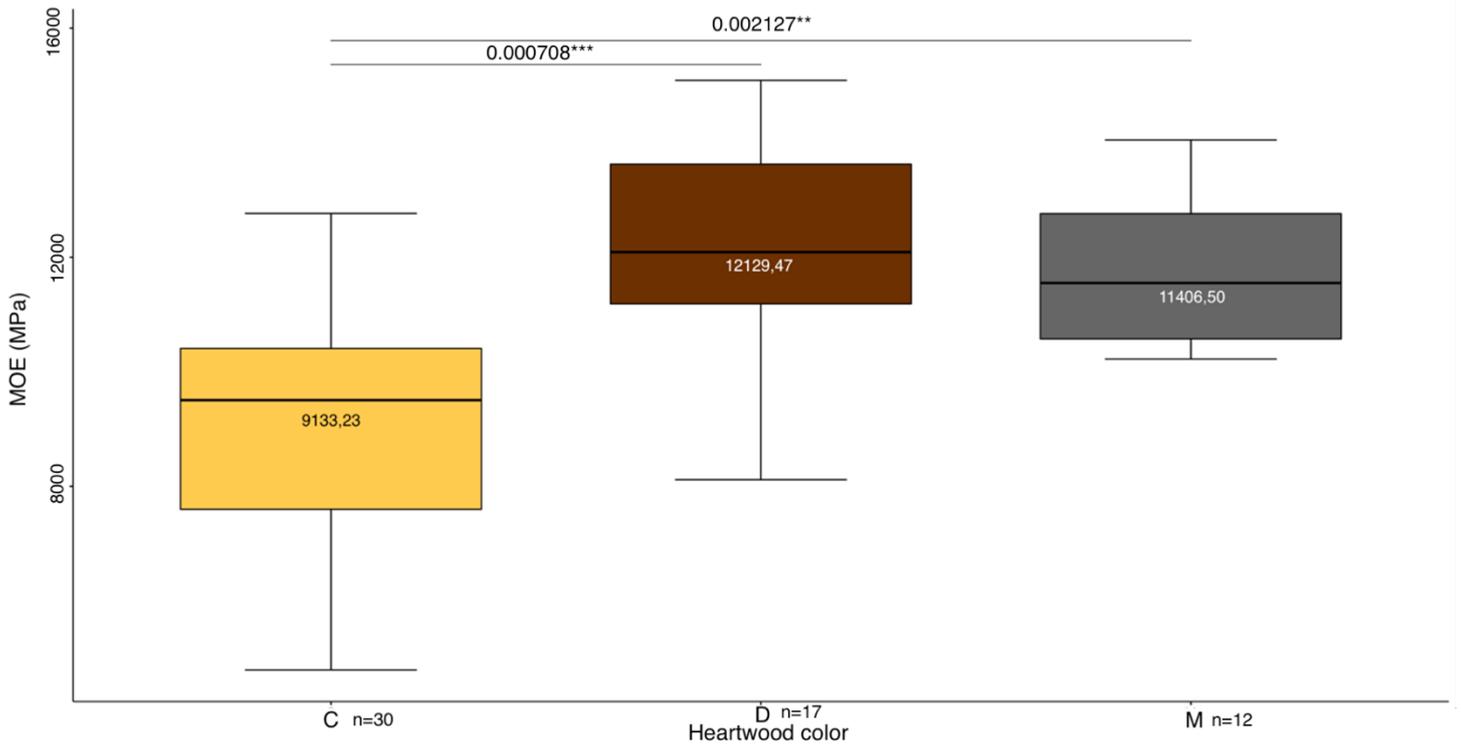


Figure 28 : Impact of heartwood colour in sorro on the modulus of elasticity (MPa) ; C = Clear, D = Dark, M = mixt.). Wilcoxon test is used to compare the color of heartwood. The p-value is added if there is a significant difference between two boxplots with the degree of significance. The median has been added in the boxplot

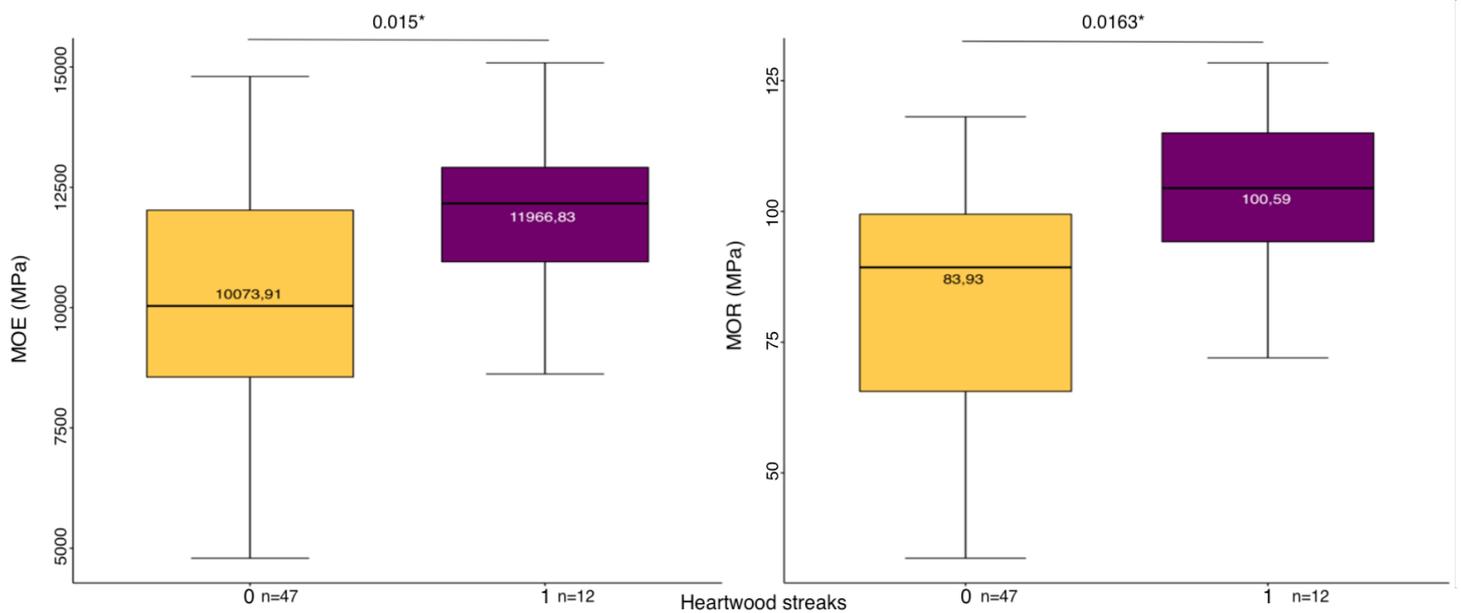


Figure 29 : Impact of the presence of heartwood streaks in sorro on the modulus of elasticity (MPa) and the modulus of rupture (MPa); 0 = no present, 1 = present.). Wilcoxon test is used to compare the presence or not of heartwood streaks. The p-value is added if there is a significant difference between two boxplots with the degree of significance. The median has been added in the boxplot

E. Fungi

The results of the fungal durability tests are given in the figures 30 and 31 for *T. versicolor* and *C. puteana* respectively. The results are summarized in table 9 for both fungi.

T. versicolor test shows that thermal treated samples of all species are less attacked by the fungus. There is a significant increase of fungi resistance when the wood is thermal treated for hornbeam, alder and sorro ($W = 25$; $p\text{-value} < 0.05$; $W = 0$; $p\text{-value} = 0.0001$; $W = 0$; $p\text{-value} < 0.001$). The difference for maple is not significant ($p\text{-value} > 0.05$) (figure 30).

C. puteana test shows that thermal treatment has a highly significant positive impact on the resistance of the species against the fungus ($W = 0$; $p\text{-value} < 0.001$; $W = 0$; $p\text{-value} < 0.0001$; $W = 0$; $p\text{-value} < 0.001$; $W = 0$; $p\text{-value} < 0.0001$) (figure 31).

The change of the orientation of the samples can impact the results (table 9). The samples $25 \times 15 \times 5 \text{ mm}^3$ (L, R, T) are less impacted by the fungi attacks than the $25 \times 15 \times 5 \text{ m}^3$ (R, T, L). To balance the difference, the standard EN 350-1 (1994) has been chosen to analyze the results. The control samples placed with the testing samples are the same orientation so they can be used in table 2.

For *T. versicolor*, before treatment, it is the hornbeam the highest median of mass loss (%) and sorro the smallest one. After treatment, it is the maple that has the highest median of mass loss (%). Treated sorro wood seems to have the smallest median of mass loss (%) (table 12).

For *C. puteana*, the thermal treated samples are almost not attacked by the fungus. The specie that has the highest median of mass loss (%) before treatment is alder and the least impacted is sorro (figure 31).

The thermal treatment increases the resistance to both fungi *T. versicolor* and *C. puteana*. The median of mass loss (%) for treated samples is inferior to untreated samples. Chaouch (2018) presents a similar trend on *Populus* when thermal treated.

Treatment is effective against *T. versicolor* but its protection is not as effective against this fungus than for *C. puteana*. Even if the species has been thermal treated, there is an impact of the fungus. Treated maple has a median of mass loss of 14.83% (*T. Versicolor*) (table 9). The treatment does not have a significant impact on its resistance to *T. versicolor* (figure 30).

Alder and hornbeam are the two species that benefits the most of the treatment. Before treatment, those two species are not durable (DC 5 for alder and DC 4 for hornbeam) against *C. puteana* and not very durable against *T. Versicolor* (DC 4 for alder and DC 5 for hornbeam). After treatment, they are rating as very durable and durable (DC 1 and DC 2) against both fungi. Maple is not very durable against both fungi (DC 4 and DC 5). After Treatment, it is durable (DC 1) against *C. puteana* but still not very durable (DC 4) against *T. Versicolor*. Sorro is durable against *C. puteana* (DC 2) and moderately durable against *T. Versicolor* (DC 3) when untreated and its durability class increase after treatment (DC 1) (table 9).

Hakkou and al. (2006) explains that those results are the consequence of the degradation of the hemicelluloses when the wood is thermally treated. It reduces the available resources for the fungi. It is also mentioned that the diminution of moisture content due to thermal treatment of

the wood and reduce the humidity for the fungus to develop. Peters and al. (2009) mention that toxic molecules can block the propagation of the fungi. It could explain the resistance of sorro wood against both fungi before and after treatment.

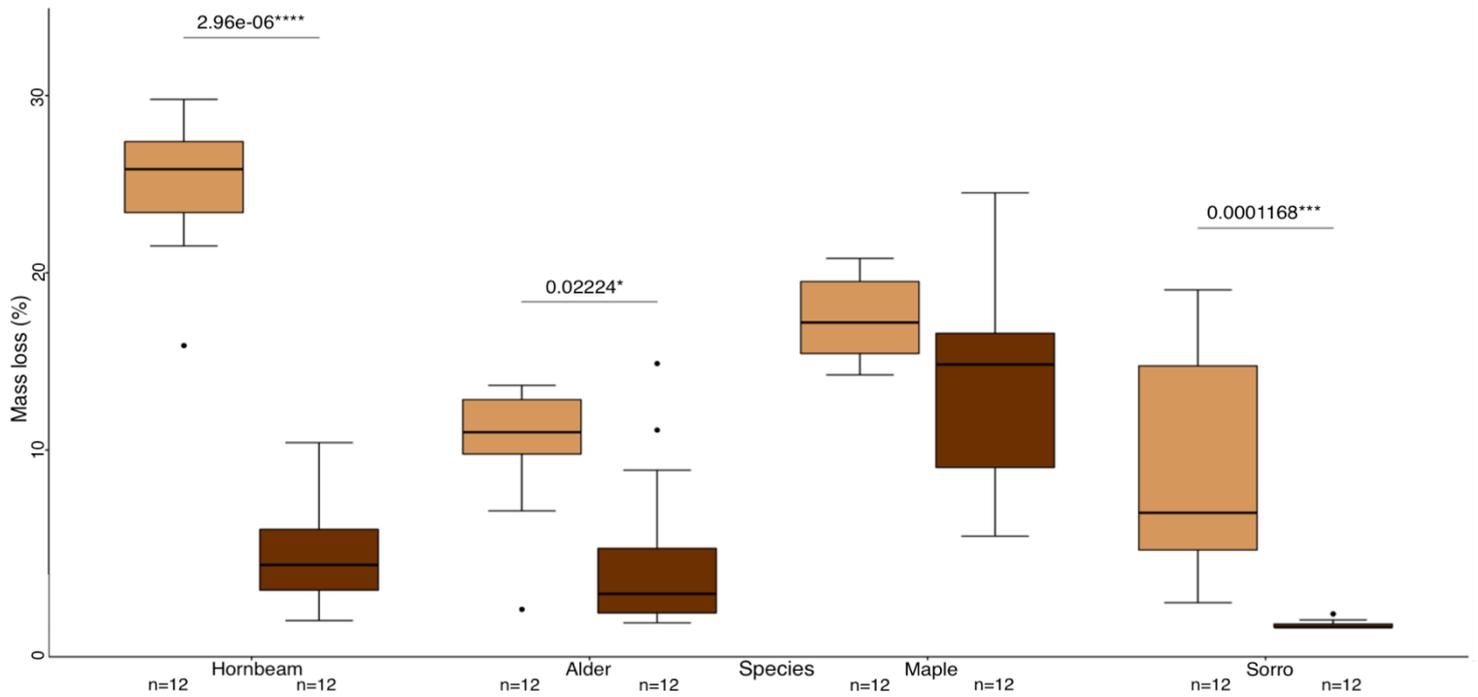


Figure 30 : Impact of thermal treatment on the resistance to *Trametes versicolor* expressed in mass loss (%) for each species. Wilcoxon test is used to compare the species/treatment. The p-value is added if there is a significant difference between two boxplots with the degree of significance

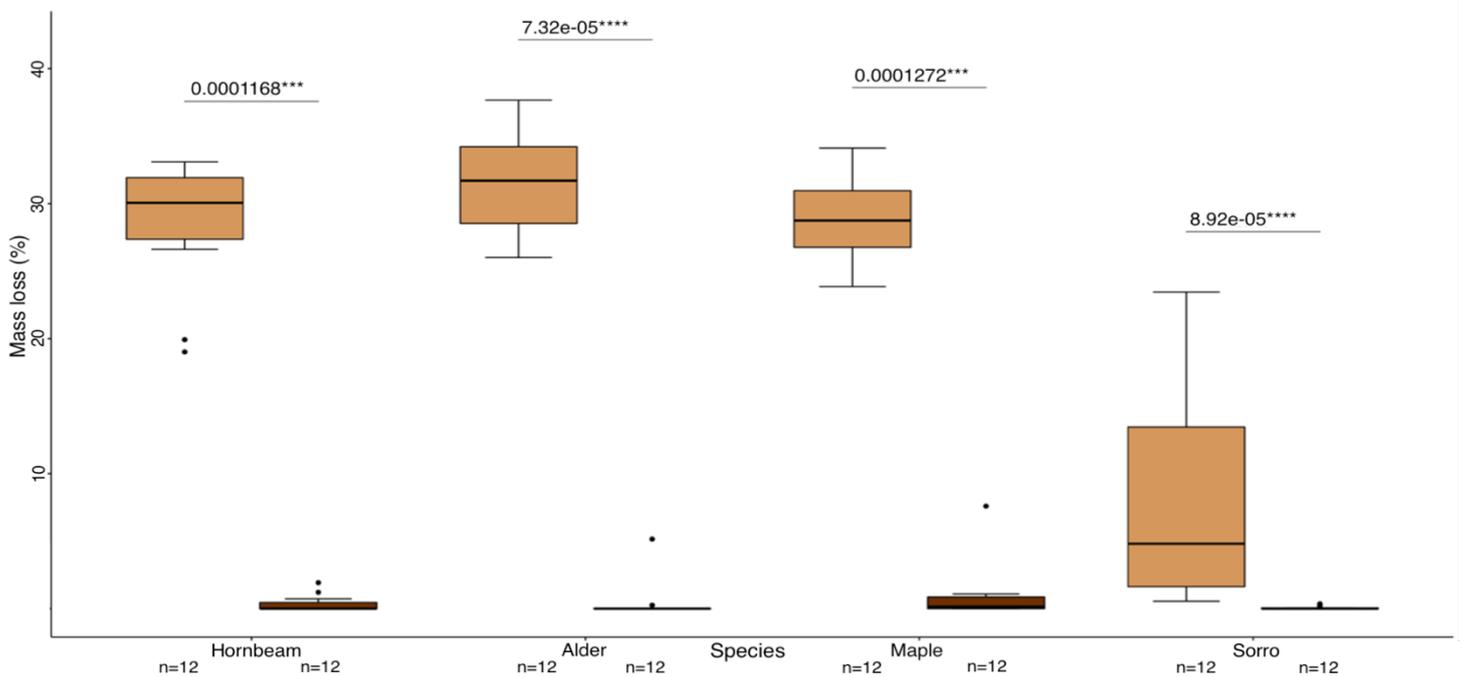


Figure 31 : Impact of thermal treatment on the resistance to *Coniophora puteana* expressed in mass loss (%) for each species. Wilcoxon test is used to compare the species/treatment. The p-value is added if there is a significant difference between two boxplots with the degree of significance

Table 9 : summary of fungal testing for the four species treated and untreated with their median of mass loss (%) and their durability class based on the rapport between samples mass loss (s) and the control sample mass loss (c) accord to the EN 350-1 (1994) standard

Species	Fungus	Treatment	Median of mass loss (%)	Mean of mass loss (%)	Mean of mass loss for control samples (%)	s/c (%)	Durability class
Alder	<i>C. puteana</i>	Untreated	31,70	31,64	33,84	0,93	5
		Treated	0,00	0,45	34,69	0,01	1
	<i>T. versicolor</i>	Untreated	10,87	10,33	16,53	0,62	4
		Treated	1,89	31,64	19,78	0,20	2
Hornbeam	<i>C. puteana</i>	Untreated	30,06	28,63	39,55	0,72	4
		Treated	0,00	0,36	36,20	0,01	1
	<i>T. versicolor</i>	Untreated	25,85	24,94	18,20	1,37	5
		Treated	3,52	3,94	18,75	0,21	2
Maple	<i>C. puteana</i>	Untreated	28,75	28,88	35,97	0,80	4
		Treated	0,14	0,94	39,39	0,02	1
	<i>T. versicolor</i>	Untreated	17,20	17,45	18,98	0,92	5
		Treated	14,83	14,21	19,14	0,74	4
Sorro	<i>C. puteana</i>	Untreated	4,81	8,12	36,17	0,22	2
		Treated	0,00	0,34	38,45	0,01	1
	<i>T. versicolor</i>	Untreated	3,92	6,85	16,69	0,41	3
		Treated	0,31	1,92	20,67	0,09	1
Control samples (L,R,T)	<i>C. puteana</i>	Untreated	/	16,64	/	/	/
Control samples (R,L,T)	<i>C. puteana</i>	Untreated	/	29,13	/	/	/

F. Termites' tests

First, non-choice tests have been realized. One sample combining two factors (species and treatment) was placed in a Petri dish. The results presented a survival rate of the termites (%), the mass loss (%) and the visual rating distribution (%) are in table 10.

The results of the virulence tests on pine sapwood show a visual rating of 4, a termite survival rate higher than 50% (table 10). It allows to validate the different termite tests carried out. The orientation of the samples (L, R, T and R, T, L) show really similar results and the virulence control samples all show a visual rating of 4 (table 10). The orientation does not have an impact on termites' tests.

All the timber species are nondurable to termite attack whether they are native or thermal treated. Except for untreated sorro, they all have a visual rating 4 (DC S) of 100%. Sorro is the only one presenting different results but is still rated at nondurable (DC S) (table 10).

For alder, the mean of mass loss is lower and the mean of survival rate is higher when the timber has been thermal treated. Hornbeam results show a higher mean of mass loss but a higher mean

of survival rate when thermal treated. Maple and sorro wood results show the same tendency as hornbeam (table 10).

Doi and al. (1999) shows the same results and explain that it is caused by the polysaccharide degradation into light molecular weight sugars. Thermal treated wood produce attractive substances for termites. Thermal treated wood is easier to eat and more attractive for termites which makes them easier to feed on the samples and survive in the Petri dishes.

The relation between wood density and the mass loss after termite tests can partly explain the preference for thermal treated wood. Wood's density is not the only factor that influences termites' preferences but it has a significant influence (Olaniran and al., 2013). As previously said, the chemical composition of the wood and the production of attractive substances are an important factor. The four species resistance to termites is impacted negatively by the treatment. Alder is the most sensitive species to termites' attacks. Treated and untreated alder samples have a visual rating of 4 after being put together for termite test which means that they are both sensible (DC S). To have a better look at the impact it is important to notice that even if they are both rated as a 4, the mass loss is higher for the treated samples (table 10). Hornbeam is denser than alder (annex 1). Hornbeam's mass loss is less important than alder. This could be explained by the wood density but not entirely.

Untreated Sorro is less impacted by termites (table 10). It could be due to extractives or secondary metabolites since it is a tropical species. In its article, Salman (2019) explains that the presence of chemicals compounds in the wood of some tropical species can affect the termites negatively.

The results of the first-choice tests, where one untreated sample of each species is placed in a Petri dish is given in table 11. The results of the second-choice test where one treated sample of each species is placed in a Petri dish is given in table 12. Those two choice tests were done to compare the different untreated species (table 11) and thermally treated species (table 12). The choice test when the species are untreated shows that termites have a preference for alder and the least impacted is hornbeam and sorro (table 11). The choice test when species are thermally treated (table 12) shows the same trend as table 11. Alder is the most impacted and sorro the least impacted. Hornbeam is exposed to termites' attacks after treatment (table 12).

The results of the third-choice tests, where one untreated and one thermal treated sample are placed in the same Petri dish, are given in table 13. The third-choice test has been made to compare the impact of the treatment on each specie (table 13). For each species, the thermal treated samples are more impacted. The mean of mass loss is higher for all species after treatment. Except alder, all the species are moderately durable against termites before treatment and become nondurable after treatment (table 13).

Table 10 : Results of the termite non-choice test for each species/treatment

Species	Treatment	Mean of Survival rate (%)	Mean of mass loss (%)	Visual rating* distribution (%)					Durability class towards termites
				0	1	2	3	4	
Alder	Untreated (n = 5)	79,6	27,6					100	S
	Treated (n = 5)	87,2	21,0					100	S
Hornbeam	Untreated (n = 5)	58,8	5,3					100	S
	Treated (n = 5)	62,6	9,6					100	S
Maple	Untreated (n = 5)	71,6	10,7					100	S
	Treated (n = 5)	85,6	15,5					100	S
Sorbo	Untreated (n = 5)	38,8	5,5			20	20	60	S
	Treated (n = 5)	51,6	12,6					100	S
Virulence controls L,R,T	N=5	72	10.49					100	S
Virulence controls R,T,L	N=5	62,8	10,34					100	S

*visual rating: no attack (0), attempted attack (1), slight attack (2), average attack (3), strong attack (4)

Table 11 : Results of the first choice test for each untreated species

Box number	Species	Workers survival rate (%)	Mass loss (%)	Visual rating	Durability class towards termites
1	Alder	54	6,59	4	S
	Hornbeam		2,88	1	D
	Maple		2,31	1	D
	Sorro		1,36	3	S
2	Alder	68	8,58	4	S
	Hornbeam		1,50	1	D
	Maple		3,59	3	S
	Sorro		1,21	1	D
3	Alder	70	2,03	4	S
	Hornbeam		1,52	1	D
	Maple		7,03	3	S
	Sorro		0,96	2	M
4	Alder	52	2,28	2	D
	Hornbeam		1,04	1	D
	Maple		6,70	3	S
	Sorro		1,73	1	D
5	Alder	66	8,96	4	S
	Hornbeam		2,73	1	D
	Maple		2,96	3	S
	Sorro		12,81	3	S

Table 12 : Results of the second choice test for each treated species

Box number	Species	Workers survival rate (%)	Mass loss (%)	Visual rating	Durability class towards termites
1	Alder	62	8,49	4	S
	Hornbeam		2,58	3	S
	Maple		1,98	2	D
	Sorro		0,00	2	D
2	Alder	90	8,25	4	S
	Hornbeam		3,91	3	S
	Maple		1,60	2	D
	Sorro		1,48	1	D
3	Alder	90	9,60	4	S
	Hornbeam		0,91	1	D
	Maple		5,40	4	S
	Sorro		5,75	4	S
4	Alder	66	10,93	4	S
	Hornbeam		3,41	4	S
	Maple		0,87	2	D
	Sorro		1,03	2	D
5	Alder	70	0,52	2	D
	Hornbeam		7,38	4	S
	Maple		2,89	2	D
	Sorro		0,59	2	D

Table 13 : Results of the third choice test for each species (untreated and treated in the same Petri dish)

Species	Treatment	Mean of survival rate (%)	Mean of mass loss (%)	Visual rating distribution (%)					Durability class toward termites
				0	1	2	3	4	
Alder	(NT)	72,8	0,93					100	S
	(T)		10,63					100	S
Hornbeam	(NT)	74,4	2,19	20	40	20	20		M
	(T)		9,19					100	S
Maple	(NT)	70,8	3,41	20	40	40			M
	(T)		9,43					100	S
Sorro	(NT)	43,6	1,94	60		20	20		M
	(T)		8,54					100	S

G. All in one

The results show a diminution of the physico-mechanical properties but also an augmentation of resistance against fungal attacks. Gunduz and al. (2009) and Boruvka and al. (2015) do not recommend thermal treated wood in construction because increase of durability does not compensate the diminution of physico-mechanical properties. Furthermore, it is noticeable that the treatment pattern adapted to hornbeam decreases its physico-mechanical properties less than the other species but it still gets the same durability increase as the others. This is a promising conclusion for future studies. They could focus on the intensity (time and temperature) of treatment to see if a good compromise can be made between durability increase and physico-mechanical properties diminution.

5. Conclusion and perspectives

Thermal treatment is still new on the market and more studies need to be done about it. This paper has studied the impact of thermal treatment on four LUS. Some conclusions can be drawn for this treatment (two treatments at 215 °C for 3 hours) for the four species tested:

1. Thermal treatment decreases the physico-mechanical properties of the four species. The modulus of elasticity is the least impacted and even increased for hornbeam. The modulus of rupture and the impact bending strength is highly impacted by the treatment. It is noticeable that if the treatment is adapted for the species, the physico-mechanical properties are less impacted.
2. Heartwood colour in Sorro have an impact on its mechanical properties. Dark duramen present better mechanical properties than clear heartwood. Better studies need to be made about heartwood streak to see if it is their influence or the influence of dark heartwood that impacts the mechanical properties.
3. Resistance to fungi is increased and highly efficient when the wood is thermally treated. This improvement was already known for thermal treated species like *Fagus sylvatica* or *Pinus sylvestris* but not for the four species studied in this paper. The improvement of durability against fungi has been confirmed for the four species. Even when not treated at an adapted intensity (temperature and time), species still get fungal durability benefits from the treatment.
4. Thermal treatment of the four species decreases the resistance to termites' attack. Thermal treatment of certain species can improve the resistance against termites by releasing toxic chemical compounds for termites. In this case, none of the species present this characteristic. They seem to be more palatable for the termites after treatment.
5. Finally, it is interesting to notice the colour change in the different species and to see their new aesthetic aspect after treatment. Those thermal treated species could be employed for indoor use and give a "touch" of thermal treated wood colour in a house.

For future studies related to this master thesis, there are a couple of recommendations that need to be made to improve the results and to help improve the knowledge of thermal treatment of LUS.

First, it would be interesting to test different treatment parameters and to control them. Different temperature levels could be tested on a species to see the influence of the temperature on the physico-mechanical properties and the durability. Another parameter that could be tested would be time. It would be interesting to see at a certain temperature the effect of the time of treatment on the properties of a species. Chemical analysis would be a good way to understand thermal treatment. An analysis of what leaves the wood when treated but also a chemical analysis of the wood before and after treatment.

Some physico-mechanical properties have not been tested due to time limitations but need to be considering for future studies such as hardness, dimensional and volume stability.

Now, studies have only been made about thermally treated mass timber (boards). In the future when the material is better known, it would be interesting to consider it into more complex construction products such as glulam, fibreboard or other wood products. If the physico-mechanical limit can be overcome, the alternative of thermal treated wood to biocide could be

a great advantage in construction. Besides that, new ways need to be found to counter the loss of durability against termites.

Other products that do not require high physico-mechanical resistance and that use of imported tropical species could be replaced by thermal treated wood for outdoor use.

6. Annexes

Annex 1: descriptive statistics of equilibrium moisture of each species per treatment

specie	treatment	Equilibrium moisture median (%)	Equilibrium moisture mean (%)	Equilibrium moisture standard deviation (%)
Alder	untreated	10,95	11,01	0,22
	treated	4,33	4,33	0,07
Hornbeam	untreated	12,46	12,27	0,44
	treated	3,92	3,92	0,10
Maple	untreated	9,79	9,76	0,14
	treated	4,47	4,49	0,15
Sorro	untreated	10,47	10,29	0,68
	treated	3,90	3,91	0,62

Annex 2: descriptive statistics of wood density, MOE, MOR, IBS for each species per treatment

Species	Treatment	Wood density median (kg/m ³)	Wood density mean (kg/m ³)	MOE mean (MPa)	MOE median (MPa)	MOE standard deviation (MPa)	MOE rating
Sorro	Untreated (n = 59)	621	625	10459	10457	2516,01	16734,4
	Treated (n = 26)	567	571	9934	10420,5	1828,33	17644,76
Hornbeam	Untreated (n = 10)	735	731	11987	11835,5	748,12	16398,08
	Treated (n = 10)	716	711	12792	12682	780,27	17991,56
Maple	Untreated (n = 10)	598	599	12400	12831	1091,95	20701,17
	Treated (n = 10)	567	571	10707	10496,5	2017,90	18784,21
Alder	Untreated (n = 10)	491	489	8692	8904,5	1438,35	17775,05
	Treated (n = 10)	420	428	7211	7039	1307,06	17169,05

Species	Treatment	Wood density median (kg/m ³)	Wood density mean (kg/m ³)	MOR mean (MPa)	MOR median (MPa)	MOR standard deviation (MPa)	MOR rating
Sorbo	Untreated (n = 59)	621	625	87,32	93,2	22,30	139,71
	Treated (n = 26)	567	571	52,13	53,05	17,67	92,59
Hornbeam	Untreated (n = 10)	735	731	110,80	111,2	9,11	151,57
	Treated (n = 10)	716	711	85,78	82,85	11,91	120,65
Maple	Untreated (n = 10)	598	599	115,80	116,9	8,65	193,32
	Treated (n = 10)	567	571	58,95	58,9	18,40	103,42
Alder	Untreated (n = 10)	491	489	58,49	57,8	17,27	119,61
	Treated (n = 10)	420	428	36,84	34,25	8,92	87,71

Species	Treatment	Wood density median (kg/m ³)	Wood density mean (kg/m ³)	Impact bending strength mean (Kg/cm ²)	Impact bending strength median (Kg/cm ²)	Impact bending strength standard deviation (Kg/cm ²)	Impact bending strength rating
Sorbo	Untreated (n = 59)	621	625	0,30	0,33	0,09	0,48
	Treated (n = 26)	567	571	0,21	0,20	0,06	0,37
Hornbeam	Untreated (n = 10)	735	731	0,47	0,48	0,11	0,64
	Treated (n = 10)	716	711	0,21	0,20	0,05	0,3
Maple	Untreated (n = 10)	598	599	0,56	0,53	0,07	0,93
	Treated (n = 10)	567	571	0,33	0,34	0,11	0,58
Alder	Untreated (n = 10)	491	489	0,18	0,19	0,07	0,37
	Treated (n = 10)	420	428	0,13	0,13	0,06	0,31

Annex 3: Sorro package information

Exploitation number	ID of the tree	Clear heartwood	Mixt	Colored heartwood
2798	S01	4	2	4
2803	S02	1	3	3
2796	S03	1	2	3
2799	S04	2	2	4

Annex 4: packages used in Rstudio

```
#load library
library(ggplot2)
library(dplyr)
library(ggpubr)
library(plyr)
library(png)
library(lme4)
library(lmerTest)
library(ade4)
library(vegan)
library(FactoMineR)
library(factoextra)
library(wesanderson)
library(multcomp)
library(corrplot)
library(Rmisc)
library(car)
library(dunn.test)
library(gridExtra)
library(ggsignif)
library(ecodist)
library(BiodiversityR)
library(ellipse)
library(party)
library(randomForest)
library(caret)
library(stringr)
library(rstatix)
```

7. Bibliography

Standards:

EN 113-1:2020 : « Durability of wood and wood-based products - Test method against wood destroying basidiomycetes - Part 1: Assessment of biocidal efficacy of wood preservatives »

EN 117:2012 : “Wood preservatives - Determination of toxic values against *Reticulitermes* species (European termites) (Laboratory method)”

ISO 3129:2012 : « Wood -- Sampling methods and general requirements for physical and mechanical testing of small clear wood specimens »

ISO 3131:1975 : « Bois -- Détermination de la masse volumique en vue des essais physiques et mécaniques »

ISO 3133:1975 : « Bois - Détermination de la résistance à la flexion statique »

ISO 3348:1975 : « Bois - Détermination de la résilience en flexion »

ISO 3349:1975 : « Bois - Détermination du module d'élasticité en flexion statique »

NBN EN 13183-1:2002 : « Teneur en humidité d'une pièce de bois scié - Partie 1: Détermination par la méthode par dessiccation »

NBN EN 350-1:1994 : Durabilité du bois et des matériaux dérivés du bois - Durabilité naturelle du bois massif - Partie 1: Guide des principes d'essai et de classification de la durabilité naturelle du bois

NBN EN 350:2016 : « Durabilité du bois et des matériaux dérivés du bois - Méthodes d'essai et de classification de la durabilité vis-à-vis des agents biologiques du bois et des matériaux dérivés du bois »

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