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Efeitos do pré-tratamento alcalino e método de secagem no processo de densificação do bambu

Effects of alkali pretreatment and drying methods on densification process of bamboo

Pirassununga
2024

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Corrected version

Dissertation presented to the Faculty of Zootechnics and Food Engineering of the University of São Paulo as part of the requirements for obtaining the title of Master in Materials Science.

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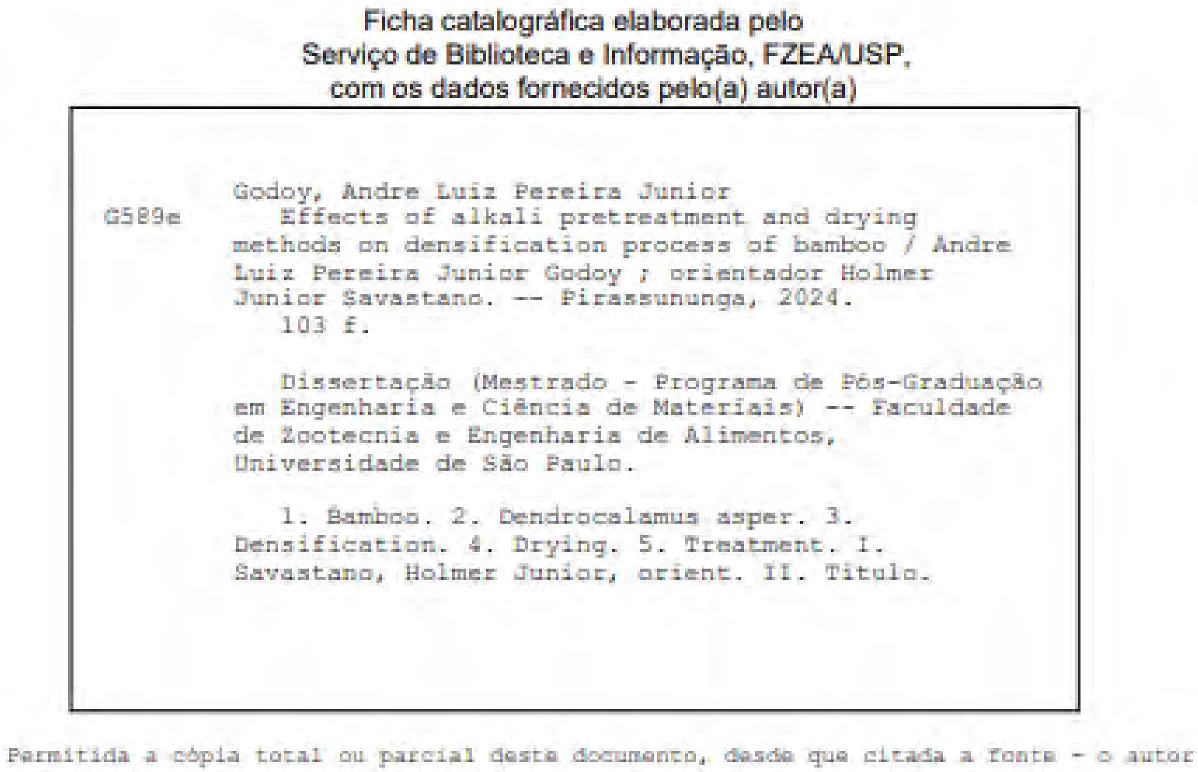

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Dedication

I dedicate this work to my father, mother, my friends and my advisors, for their support, affection and understanding, with admiration and gratitude for their support, affection, patience and presence throughout the period of preparation of this work

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Epigraph

“Ignorance more often generates confidence than knowledge: it is those who know little, and not those who know a lot, who assert so categorically that this or that problem will never be solved by science.”
Charles Darwin (1871)

Abstract

GODOY, A. **Effects of alkali pretreatment and drying methods on densification process of bamboo.** 2024. Master thesis- Faculdade de Zootecnia e Engenharia de Alimentos, Universidade de São Paulo, Pirassununga 2024

Due to its fast growth, short rotation period, and very promising properties including a higher cost-efficient compared to conventional bricks and reinforced concrete, bamboo can be considered as one of the best options for replacing conventional materials, being one of the oldest building materials used in human history. Despite bamboo's promising properties, some problems and unwanted effects like inhomogeneous mechanical properties, dimensional instability, and degradation must be overcome to have a proper role as a building material. As a solution, some kinds of pre-treatments can be used, followed by the densification process. There are numerous reviews of wood treatment in the scientific literature, but few reviews of how these treatments and the drying method affect the bamboo. In this study, a two-step process of delignification and densification was carried out on *Dendrocalamus asper* bamboo specimens. It is aimed to investigate the densification properties of the bamboo samples after partial removal of the lignin. To delignify the samples, two different approaches were applied. The first group was treated in a room-temperature NaOH + $NaSO_3$ solution, while the second group was treated in the same solution at 100°C. Afterward, the samples were dried in either an oven with 100°C or at room temperature of 25°C. Hence, a total of four different groups of delignified and dried samples were produced with an average moisture content of 7 - 10%. The samples were then densified to 50% of their original thickness having the thickness reduction determined a priori using a thermo-mechanical press system at 160°C. The results indicated that all alkali treated samples required a lower load for the densification process compared to the reference. This implies that alkali treatment could enable a greater degree of densification with consistent energy consumption. This outcome has the potential to improve the material's mechanical properties and boost its density. Average compression stress of 17.0 MPa for the reference, 10.3 MPa for the samples treated with alkali solution at room temperature, and 7 MPa for the boiled alkali solution and dried at room temperature samples needed only 40% of the stress applied on non-treated bamboo. This finding implies a significant decrease in the energy and load needed to achieve a 50% densification degree. The drying process also affected the compression resistance of bamboo and oven-dried specimens showed an increase of 30% and 17% for room temperature and boiling temperature treatment respectively. The modulus of rupture, limit of proportionality, and elastic modulus of densified bamboo were all negatively impacted by the pre-treatment, according to the results of the bending test. Overall, untreated and treated bamboo displayed similar behavior for physical attributes in densified samples. After being treated with alkaline solution, bamboo showed reduced levels of lignin as well as hemicellulose and cellulose, according to chemical tests. To summarize, subjecting bamboo to this concentration of alkali solution in an open system with a densification degree of 50% is not enough to have a proper densification compromising the mechanical properties of the material.

Keywords: Bamboo, delignification, drying, densification

Resumo

GODOY, A. **Efeitos do pré-tratamento alcalino e método de secagem no processo de densificação do bambu**. 2024. Dissertação (Mestrado) – Faculdade de Zootecnia e Engenharia de Alimentos, Universidade de São Paulo, Pirassununga 2024.

Devido ao seu rápido crescimento, curto período de rotação e propriedades muito promissoras, incluindo uma eficiência de custos mais alta em comparação com tijolos convencionais e concreto armado, o bambu pode ser considerado uma das melhores opções para substituir materiais convencionais, sendo um dos materiais de construção mais antigos usados na história humana. Apesar das propriedades promissoras do bambu, alguns problemas e efeitos indesejados, como propriedades mecânicas não homogêneas, instabilidade dimensional e degradação, devem ser superados para que desempenhe adequadamente o papel de material de construção. Como solução, alguns tipos de pré-tratamentos podem ser utilizados, seguidos pelo processo de densificação. Existem numerosas revisões sobre tratamento de madeira na literatura científica, mas poucas revisões sobre como esses tratamentos e o método de secagem afetam o bambu. Neste estudo, foi realizado um processo de deslignificação e densificação em duas etapas em espécimes de bambu *Dendrocalamus asper*. O objetivo foi investigar as propriedades de densificação das amostras de bambu após a remoção parcial da lignina. Para deslignificar as amostras, foram aplicadas duas abordagens diferentes. O primeiro grupo foi tratado em uma solução de NaOH + NaSO3 em temperatura ambiente, enquanto o segundo grupo foi tratado na mesma solução a 100°C. Em seguida, as amostras foram secas em um forno a 100°C ou em temperatura ambiente de 25°C. Assim, um total de quatro grupos diferentes de amostras deslignificadas e secas foram produzidos, com um teor médio de umidade de 7 a 10%. As amostras foram então densificadas para 50% de sua espessura original, tendo a redução de espessura determinada previamente usando um sistema de prensa termo-mecânica a 160°C. Os resultados indicaram que todas as amostras tratadas com solução alcalina requerem uma carga menor para o processo de densificação em comparação com a referência. Isso implica que o tratamento alcalino poderia permitir um maior grau de densificação com consumo de energia consistente. Esse resultado tem o potencial de melhorar as propriedades mecânicas do material e aumentar sua densidade. A média da tensão de compressão foi de 17,0 MPa para a referência, 10,3 MPa para as amostras tratadas com solução alcalina à temperatura ambiente e 7 MPa para as amostras tratadas com solução alcalina fervente e secas à temperatura ambiente, necessitando apenas 40% da tensão aplicada no bambu não tratado. Isso implica uma diminuição significativa na energia e carga necessárias para atingir um grau de densificação de 50%. O processo de secagem também afetou a resistência à compressão do bambu, e as amostras secas em forno mostraram um aumento de 30% e 17% para tratamento à temperatura ambiente e fervura, respectivamente. O módulo de ruptura, limite de proporcionalidade e módulo de elasticidade do bambu densificado foram todos impactados negativamente pelo pré-tratamento, de acordo com os resultados do teste de flexão. Em resumo, submeter o bambu a essa concentração de solução alcalina em um sistema aberto com um grau de densificação de 50% não é suficiente para obter uma densificação adequada, comprometendo as propriedades mecânicas do material.

Palavras-chave: Bamboo, deslignificação, secagem, densificação

Summary

Figure List

Figure 26: SEM images of densified bamboo samples. The images were extracted from the outer (a), middle (b), and inner layer (c).

Figure 27: SEM images of undensified bamboo samples. The images were extracted from the outer (a), middle (b) and inner layer (c)

Figure 28: a) reference group without densification b) undensified sample that underwent alkaline treatment at room temperature and dried at room temperature (T25D25UN) c) densified reference group.

Figure 29: FT-IR spectra for densified bamboo samples.

Figure 30: DSC curves of untreated and treated bamboo samples.

Figure 31: DTG curves of untreated and treated bamboo samples.

Figure 32: TG curves of untreated and treated bamboo samples.

Table list

List of abbreviations and acronyms

ASTM - American Society for Testing and Materials

BBP - Bamboo-based panels

EN - European Committee for Standardization.

FTIR - Infrared Spectrometer

ISO - International Standards Organization

LOP - Limit of Proportionality

MC - Moisture Content

MOE - Modulus of elasticity

MOR - Modulus of rupture

SEM - Scanning electron microscopy

THM - Thermo-Hydro Mechanical treatment

TM - Thermo- Mechanical treatment

TG - Thermogravimetric analysis

TVM - Thermo-Vibro-Mechanical

XRD - X-ray diffraction

VTC - Viscoelastic-Thermal-Compression

DD - Densification Degree

TS - Thickness Swelling

WA - Water Absorption

RH - Relative humidity

FSC - Forest Stewardship Council

CKD - Cement Kiln Dust

TR – Thickness Ratio

1. Introduction

With the growth of the world population, the civil construction sector has been growing increasingly, requiring even more natural resources. However, few of these materials are used as primary products, and most are left in landfills. Added to this, construction and demolition sites are huge dumps of solid wastes, presenting huge environmental impacts because they do not degrade, depleting the disposal space more quickly, and depriving the use of this space after the end of activities. In Brazil, construction industries are responsible for consuming 50% of the resources extracted from nature and producing 40% of all waste generated, with approximately 84 million cubic meters of waste according to ABRECON (ABRECON, 2019) making conventional civil construction no longer viable and considered sustainable.

The Brundtland Commission states that in order for environmental sustainability to be achieved, it is important that it does not compromise the natural elements that are vital to maintaining the overall integrity of the ecosystem: the quality of air, soil, water, and health of the living beings. Finding new technologies to reduce pressure on the environment that minimize depletion and provide substitutes for these resources (Giovanetti, 2019).

Environmental sustainability is related to sustainable consumption and production patterns and greater efficiency in using energy to minimize environmental pressures, the depletion of natural resources, and pollution. Governments, together with the private sector and society, must act to reduce the generation of waste and discarded products, through recycling, industrial processes and the introduction of new eco-friendly products (Ulucak, 2020).

Wood-based building materials have been widely used throughout human history showing excellent mechanical and biofunctional Properties (Burgert., 2015). The production and mechanical properties of wood-based fiberboards depend on the microstructure of the material, mainly due to the individual properties of each fiber, the lignin in wood, and the interaction of the fibers in the lignin and hemicellulose matrix.

Among the new research to replace conventional construction materials, bamboo showed to be the most promising one, presenting fast growth, short rotation period, capacity to store CO2, and very promising properties, including its lightweight, flexibility, toughness, high tensile, and higher cost-efficiency when compared to conventional bricks and reinforced cement. Bamboo is a very abundant resource, with a global annual output of 15 billion units (Yadav & Mathur, 2021).

According to international reports on bamboo and rattan, global forest area has declined sharply, but bamboo forest area has increased at a 3% annual rate, specifically, *Dendrocalamus asper*, being the foremost bamboo species in carbon sequestration, exhibiting the highest carbon capture, prioritizing culm over rhizome and leaf components, largely attributable to its extensive biomass. The recorded carbon content of *D. asper* reached 87.52 tons per hectare. Subsequently, *B. balcooa* followed with a carbon content of 56.48 t C ha-1, while *B. vulgaris* demonstrated the lowest carbon content at 33.92 t C ha-1. (Muchiri, 2021). Increasing the carbon storage capacity of bamboo products is critical for increasing forest sinks and mitigating the environmental problems caused by global warming (Yadav & Mathur, 2021). Under the same growth conditions, bamboo's annual output value can reach 78.3 tons, which is 4.47 times that of wood (Van der Lugt, 2003).

Bamboo forests cover approximately 31.5 million hectares worldwide, accounting for 0.8% of the total forest area. There are over 1,500 different types of bamboo products available worldwide, including housing, handicrafts, boards, charcoal, and food (Fang et al., 2018) Bamboo has the potential to replace metal, wood, polyvinyl chloride resin (PVC), and other materials, and it will be used in a variety of applications ranging from water conservation pipelines to high-speed rail cars and capacitors (Fang et al., 2018). The bamboo industry has emerged as one of Asia's pillar industries for rural revitalization. Furthermore, the 26th United Nations Global Climate Change Conference agreed that the world should stop logging virgin forests and tropical rainforests, creating more opportunities for the rapid development of bamboo.

Despite bamboo's promising qualities, few factors hinder its use in large scale, such as: inhomogeneous geometry and physical and mechanical properties along the culm, dimensional instability, natural degradation, and anisotropy. These characteristics must be overcome to promote a wider use for bamboo in the construction industry (Yang et al., 2020).

Through controlled processes that alter the physical and mechanical characteristics of bamboo, it is possible to create alternative products with enhanced standardization and reliability. Exploring options like chemical treatments and densification has become a common practice in both the wood and bamboo industries, aiming to reduce the inherent heterogeneity of these materials and boost their overall performance (Archila-Santos et al., 2014; Kadivar et al., 2020; Kadivar et al., 2019; Gauss et al., 2017).

Bamboo densification can be accomplished through various methods, each with its own advantages and disadvantages that will be discussed in the next sections. The densification process involves compressing samples using an open or closed thermo-hydraulic press system in the radial direction, resulting in a reduction of the internal volume of the material. This reduction occurs either through the plasticization or collapse of cell walls (Dixon, 2016; Pelit et al., 2015; Kadivar et al., 2020). Bamboo densification holds promise for improving the material's properties, rendering it more suitable for construction and diverse applications. Nevertheless, the final product may encounter challenges related to dimensional instability over time. Furthermore, the densification process requires a substantial amount of energy.

To overcome the negative impacts of densification, some pretreatments are necessary to be studied. Bamboo pretreatment is the process of preparing bamboo for further processing or use. Pretreatment can include various methods such as soaking, boiling, and chemical treatment. Soaking bamboo in water is a common pretreatment method that can help to soften the fibers and make the bamboo more pliable. This can make it easier to work with during the manufacturing process. Boiling bamboo is another method used to soften the fibers and make the bamboo more pliable. Boiling bamboo is also known to kill any pests or insects that may be present in the bamboo. Chemical pretreatment is another common method that is used to improve the properties of bamboo. This can include treatment with boron, boric acid, or sodium silicate. These chemicals can help to increase the density of bamboo and make it more resistant to moisture and insects. However, chemical treatment can also be toxic and can affect the environment. (Kaur et al, 2016). These methods serve to dissipate internal stresses, improve dimensional stability, strength, surface hardness, durability, and facilitate the drying and plasticization of the material. However, while processing in an open system, it is impossible to appropriately regulate the process parameters such as the moisture content of the materials. Whether the process is open or closed will affect the densification processing's outcome (Navi P, 2000).

The delignification process entails the extraction of lignin from plant-based resources, leading to the breakdown of the lignocellulosic structure into fibrous components (Schild et al., 2010). In industrial settings, the widely employed method involves chemical processes using sulfite and alkaline pulping (Schild et al., 2010). However, solvent-based pulping, utilizing substances such as sodium hydroxide, anthraquinone, and methanol, offers higher efficiency, and generates fewer emissions and by-products. Despite its advantages, this approach is predominantly utilized on a smaller scale due to a higher cost (Schild et al., 2010).

Studies have explored the use of water as a pretreatment to extract lignin before bamboo densification, aiming to enhance bamboo plasticity and streamline the densification process (Kadivar et al., 2022).

The two-step delignification/densification process involves partially removing lignin and hemicellulose from lignocellulosic materials, followed by thermo-mechanical pressing. This process is commonly applied to wood or bamboo, offering more efficient densification with reduced energy consumption and slight modifications to their structure. The outcome is improved performance, including enhanced biological resistance, superior dimensional stability, lower equilibrium moisture content, reduced contraction and expansion, and improved weather resistance (Song et al., 2018).

In some studies, a two-step process involving delignification with NaOH + Na2SO3 solution, followed by densification, significantly increased the strength of wood. For instance, densified basswood with 45% lignin removal exhibited an ultimate tensile strength of 587 MPa, compared to 51.6 MPa for natural wood and 175.0 MPa for densified wood without delignification (Song et al., 2018). Similarly, *Phillostachys bambusoides* bamboo species demonstrated enhanced tensile (770 MPa) and flexural strength (327 MPa) after undergoing delignification and densification processes, compared to natural bamboo (298 MPa and 148 MPa, for tensile and flexural strength, respectively) (Li et al., 2020).

1.1 General objectives

The general objective of this work is to study chemical modification and delignification through NaOH-based solutions with different temperatures, and two types of drying processes before densification, with the optimal parameters showed by Kadivar 2020 fixing the displacement until achieving 50% of densification degree and evaluate its effects on bamboo physical, mechanical, chemical, and thermal properties of *Dendrocalamus asper* bamboo. It is expected to achieve highly aligned cellulose fibers and material with improved dimensional stability and mechanical performance.

1.2 Technical objectives

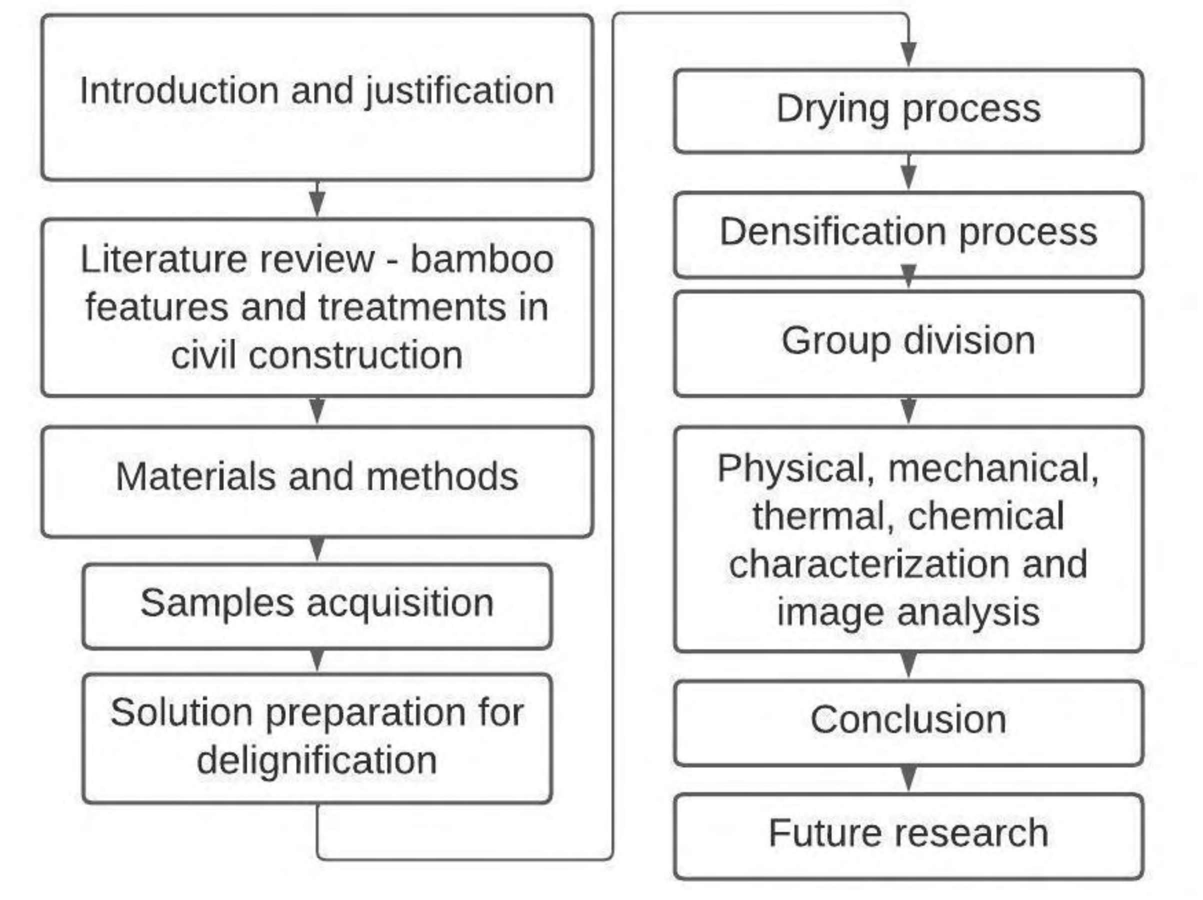
- Evaluate the impact of the NaOH treatment on the physical, mechanical, chemical and thermal properties of *Dendrocalamus asper* bamboo.
- Examine the impact of different temperatures during the alkali treatment on the bamboo, aiming to optimize conditions for enhanced cellulose alignment and properties.

- Analyze the influence of two distinct drying processes following chemical modification, comparing their effects on bamboo properties, including dimensional stability and mechanical strength.
- Utilize microscopy and imaging techniques to analyze the microstructure in the modified bamboo, aiming for a highly aligned structure.
- Explore the consequences of the delignification process on the bamboo's chemical composition, and the influence in the optimal parameters of bamboo densification focusing on lignin removal and its impact on overall material properties.
- Optimize the parameters for the densification process based on the findings from chemical modification and delignification, aiming to achieve improved mechanical properties and dimensional stability.
- Assess the dimensional stability of the modified bamboo under different environmental conditions.
- Investigate the changes in chemical composition of bamboo before and after the modification process, with a focus on cellulose, hemicellulose, and lignin content.
- Establish correlations between the observed structural changes in bamboo and its resulting physical and mechanical properties, analyzing structure-properties relationships.

1.3 Work structure

This section aims to display the division and structure of the research being displayed in Figure 1.

Figure 1- Flowchart on the work structure.



Source: Own authorship

2 Literature review

2.1 Civil construction

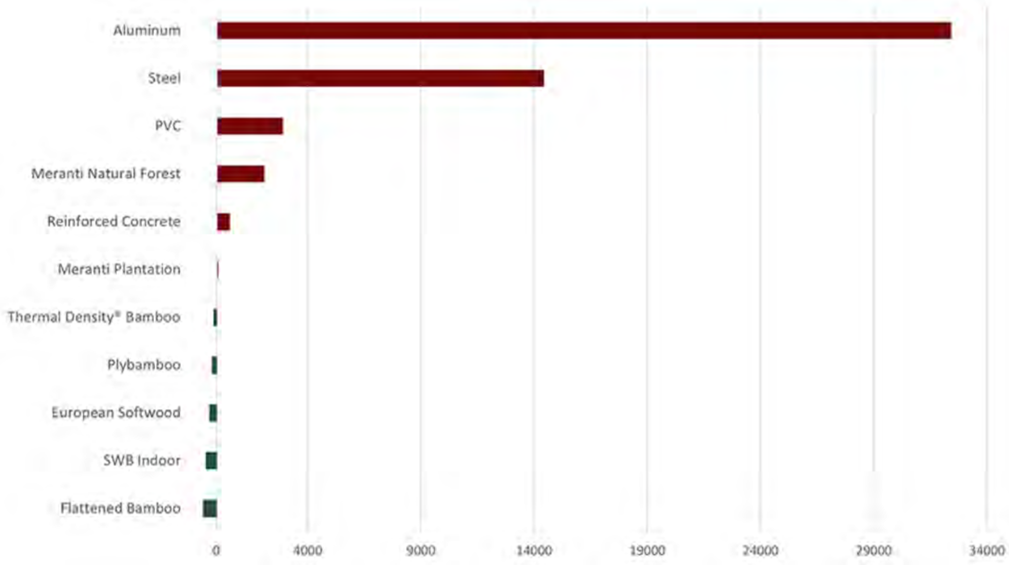
Over the past decade, there has been a rapid influx of groundbreaking technological innovations that have significantly impacted societies and economies globally. While these innovations have ushered in numerous opportunities, they have also brought about unforeseen consequences. To cope with the evolving landscape, civil engineering professionals are compelled to adapt and innovate, particularly in addressing the escalating demands for improved quality and sustainability in today's world (Fiorino, 2016).

Taking the example of United States which can be a good representative of the global trend, where the job market for civil engineering remains robust, with a projected 20%

increase in employment from 2012 to 2022. Civil engineering stands out as the largest employment sector worldwide within the broader field of engineering, offering a variety of courses and experiencing a notable salary growth. This underscores the ongoing significance of civil engineering as an integral component of the global economy (DeZarn, 2023).

Traditionally tasked with designing, planning, and managing infrastructure systems and projects, civil engineers now find themselves facing a paradigm shift. In the modern era, they cannot solely rely on conventional engineering concepts and technical skills to enhance infrastructures. Instead, they must proactively stay ahead of emerging challenges by embracing sustainable approaches and integrating the latest technical innovations (Habash, 2017). Figure 2 shows the carbon footprint of building materials

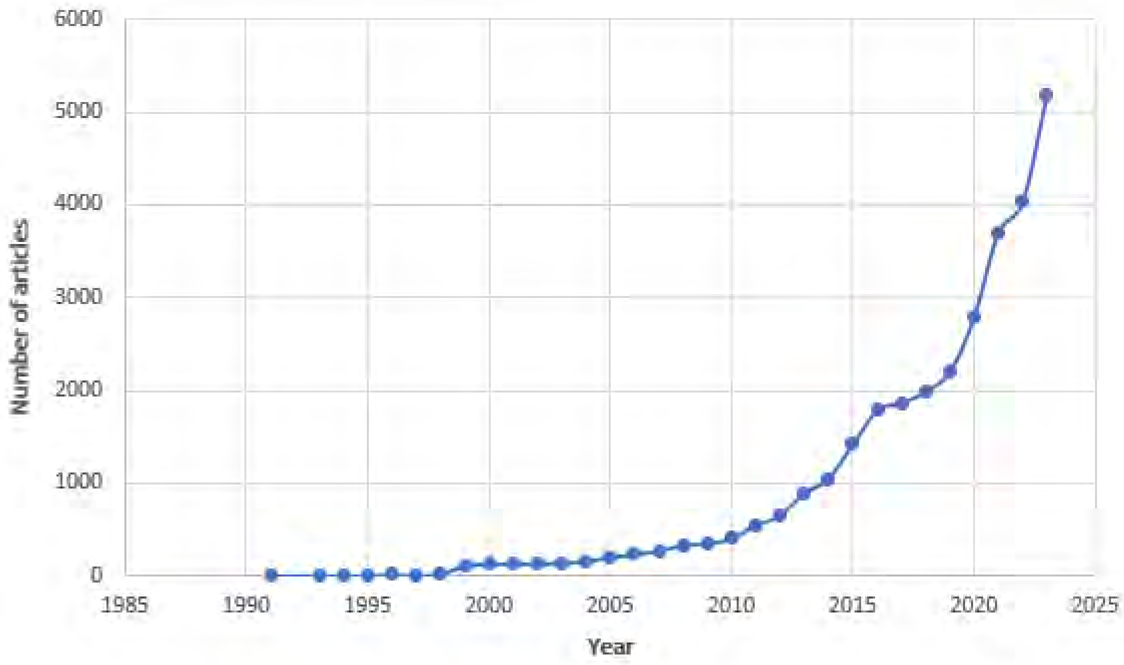
Figure 2: Building material carbon footprint over life cycle in CO2 eq/m3



Source: Voigtländer,2015

A primary contemporary challenge in civil engineering revolves around the imperative to design and construct sustainable housings. This entails not only ensuring social and economic equity but also fostering harmony with the natural environment, encapsulated by the concept of sustainable construction. As the relevance of sustainable development continues to grow, an increasing number of publications are addressing the theme of sustainability in construction (Figure 3).

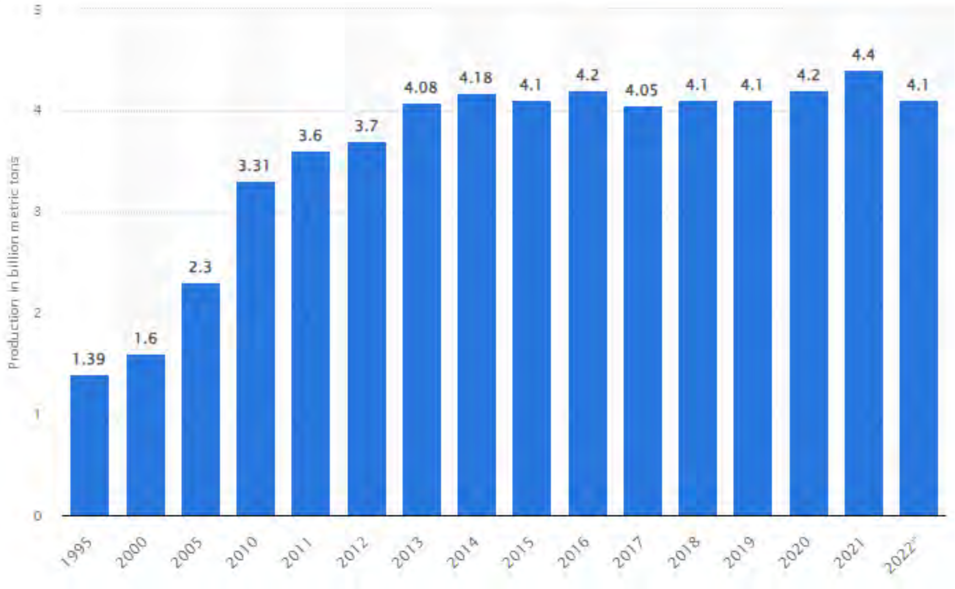
Figure 3: The quantity of publications within the science direct categories related to "sustainability" in civil engineering and construction building technology.



Source: Science direct core collection database, 2023.

Cement-based materials, widely utilized globally, particularly concrete as the second most used substance after water, owe their technological success to versatility, user-friendly molding, and rapid transformation into an 'artificial' rock, crucial for structures from houses to nuclear power plants (Stajanča, 2012). However, the cement industry confronts substantial environmental challenges, emitting pollutants like carbon dioxide, nitrogen oxides, dioxins, and heavy metals, contributing 30-70% of total industrial energy consumption (Saidur, 2010; Subhes, 2005; Strennhof, 2006). Environmental evaluation involves hazardous substances like Cement Kiln Dust (CKD), posing risks to humans, animals, and ecosystems, while the burning of hazardous materials contributes to toxic compounds (Huntzinger and Eatmon, 2009; Yang, 2019). Cement production's energy-intensive nature raises sustainability concerns, as each ton generates an equivalent ton of CO2, necessitating resource-efficient practices (Naik, 2020). The evolution of cement production is illustrated in Figure 4.

Figure 4: Production volume of cement worldwide from 1995 to 2022.



Source: Statista Research Department, 2023

2.1.1 Sustainability of wood in construction

Wood has been a traditional and versatile material in civil construction, offering a range of benefits such as renewability, aesthetic appeal, and a relatively low environmental impact during the manufacturing process. However, the use of wood in construction poses various challenges and sustainability concerns that warrant careful consideration (Asif, 2009).

One of the primary issues associated with wood in civil construction is deforestation. As the demand for timber increases globally, there is a risk of overharvesting and depletion of forests, leading to environmental degradation and loss of biodiversity. Unregulated logging practices can contribute to deforestation, disrupt ecosystems, and have cascading effects on climate regulation and water cycles (Ramage, 2007).

Additionally, the extraction and processing of wood for construction purposes can result in significant carbon emissions. The carbon footprint associated with deforestation, transportation, and manufacturing processes can offset the environmental benefits initially attributed to wood as a renewable resource. Sustainable forestry management practices, certifications (such as FSC - Forest Stewardship Council), and responsible sourcing are essential to mitigate these negative impacts and ensure the long-term viability of wood as a construction material (Woodard, 2016).

Wood is susceptible to decay, insect infestations, and fire, presenting durability challenges. To address these issues, chemical treatments and preservatives are often applied to enhance wood's resistance to decay and pests. However, the use of certain chemicals raises environmental and health concerns, and the treatment processes must be carefully managed to minimize adverse effects (Brischke, 2006).

In terms of sustainability, the concept of embodied energy is crucial when evaluating wood's environmental impact. Embodied energy encompasses the total energy required for the extraction, processing, transportation, and installation of a material. While wood generally has a lower embodied energy compared to many alternative construction materials, it is essential to consider factors such as transportation distances and the energy used in manufacturing (Cabeza, 2013).

To enhance the sustainability of wood in construction, there is a growing emphasis on using engineered wood products. Engineered wood, such as laminated veneer lumber (LVL), glue-laminated timber (glulam), and cross-laminated timber (CLT), allows for efficient use of resources, reduced waste, and improved structural performance. These products often make use of smaller, fast-growing trees and recycled wood, promoting responsible forestry practices (Aarnio, 2020).

2.1.2 Bamboo in construction

Bamboo, classified as a giant grass, holds significant importance as a non-timber forest product with diverse applications. Historically, its utilization in construction dates to ancient times, particularly in regions where bamboo thrived abundantly. The inherent strength and flexibility of bamboo rendered it a preferred choice for constructing dwellings. Over time, the aesthetic appeal of bamboo has also emerged as a crucial factor in its contemporary use.

The resurgence of interest in bamboo as a building material gained momentum during the 1980s, fueled by a global shortage of housing materials, particularly within the timber industry (Rao et al., 1995; Tadesse, 2006; Basumatary et al., 2015; Nurdiah, 2016). The natural growth of bamboo is primarily observed in tropical, sub-tropical, and mild temperate regions across Africa, Asia, America, and Oceania (Clark, 2006; Escamilla and Habert, 2014; Shah, 2014) as shown in Figure 5.

Figure 5: Worldwide distribution of bamboo

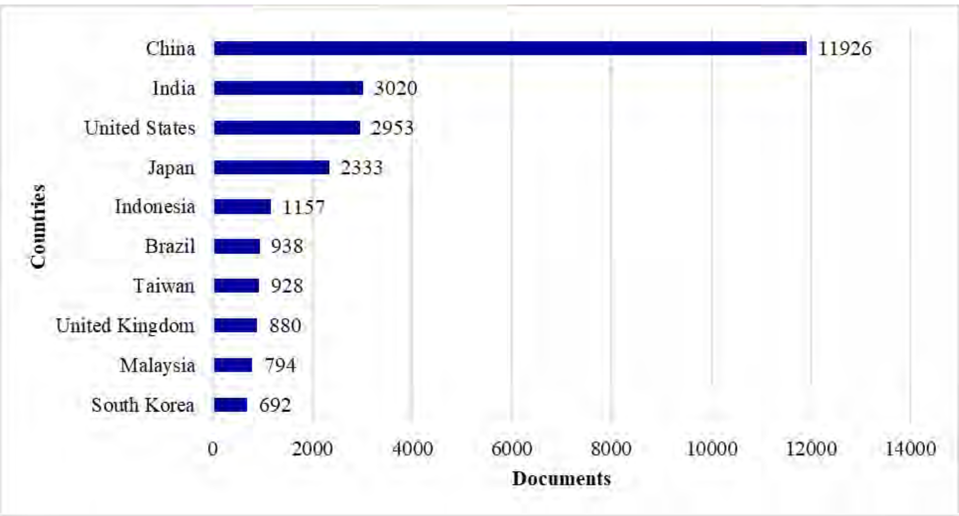
Source: Clark, 2006 – Bamboo Biodiversity

Bamboo exhibits versatility in construction applications, spanning structural elements like walls, columns, woven roofs, doors, and windows. In modern times, bamboo is often combined with cement or adhesives to enhance strength and aesthetics, aligning with contemporary lifestyles. While bamboo is inherently susceptible to natural degradation, proper treatment and industrial processing can extend the lifespan of bamboo components to approximately 30–40 years, contingent on the species and treatment methods (Gichohi, 2014).

Characterized by strength and lightweight properties, bamboo can be used without extensive processing or finishing (Gichohi, 2014). Despite challenges posed by its natural round and hollow form, which complicates construction connections, ongoing research endeavors aim to address these issues through innovative connection details and composite materials. Extensive research by bamboo practitioners underscores that, when appropriately treated, bamboo stands as a sound structural and engineering material, owing to its strength, flexibility, and versatility, thereby making it a viable candidate for sustainable housing (Kibwage, Frith, and Paudel, 2011).

Nowadays, bamboo is increasingly recognized as a building material conducive to sustainable development. Its qualifications stem from environmental, social, and economic benefits with increasing research documents in bamboo areas since 1950 until 2023 (Figure 6).

Figure 6: Countries with more research documents in bamboo since 1950



Source: adapted from scopus database

Bamboo, owing to its ready availability, ease of workability, and considerable strength, has been extensively employed as a construction material. Its tensile strength surpasses that of steel, making it suitable for horizontal members up to 3–3.6 meters in length without middle support (Nwoke and Ugwuishiwu, 2011), withstanding pressures of up to 3656 kg/cm2 (358.53 MPa) (Paudel, 2008). The species of bamboo significantly influences its strength, with variations attributed to factors such as age, diameter, wall thickness, load position, radial position, and water content (Leake et al., 2010).

Bamboo's rapid growth rate and adaptability make it suitable for afforestation, although challenges such as loss of species variety and low growth rates in nutrient-deprived areas have been noted (Basumatary et al., 2015). Bamboo's ability to interplant with other crops, especially in reforestation and soil protection efforts, contributes to a sustainable and nutrient-rich vegetation system (Ministry of Forestry and Mines, 2011).

The growth of bamboo facilitates carbon sequestration as it absorbs carbon dioxide, and when used in construction, carbon is stored until the building's end of life. Carbon storage and sequestration rates for bamboo are reported as 30–121 Mg per ha and 6–13 Mg per ha per year, respectively (Nath, Lal, and Das, 2015). Bamboo's potential as a solid wood substitute material reduces pressure on forest resources, lessens energy consumption in construction, and emits lower amounts of carbon dioxide compared to traditional materials like bricks and cement.

Environmental benefits extend to bamboo's renewable nature, aiding in deforestation reduction, encouraging bamboo cultivation on wasteland and river banks, and providing soil conservation. Moreover, the socio-economic impact of bamboo is significant. As a major non-wood forest product, bamboo plays a crucial role in generating income for many individuals and reducing poverty. Local skill development is fostered through bamboo-based housing construction, creating income-generating opportunities and preventing migration. The ease of construction with bamboo, requiring basic carpentry and masonry skills, facilitates widespread adoption and training within communities (Larasati, Ihsan, and Mawardi, 2013). Adding to these benefits, bamboo plantations have remarkable environmental attributes, absorbing 12 tons of CO2 per hectare annually, producing 35% more oxygen than trees, growing up to 3 feet per day, and being harvestable every 3-5 years. *Dendrocalamus asper* Bamboo for example, yields 20 times more timber per hectare than conventional trees, regrows naturally, requires no pesticides, prevents soil erosion, and is biodegradable and compostable after use (INBAR, 2017).

Considering disaster resilience, bamboo contributes positively by providing swift construction of disaster-resistant houses. Its root system prevents landslides, and bamboo has been recognized as an earthquake-resistant material due to its strength and lightweight nature (López, Bommer, and Méndez, 2004). Various studies in South America, Colombia, and El Salvador have highlighted bamboo's efficacy in seismic resistance, with the bahareque construction system being identified as seismically resistant (Saleme and Navarro, 2001).

The resurgence of interest in bamboo over the last four to five decades has transformed its applications in construction, influencing the overall architectural outlook. Traditional construction methods involved utilizing whole culms, split bamboo, pressed flats, or woven mats, often joined with vines or rattan, resulting in relatively weak joints (Mawardi, 2013). Examples include the Bahareque construction system in Indonesia and Colombia, where bamboo or cane elements are integrated with timber vertical elements, mud infill, and plaster finishes (Widyowijatnoko, 2006).

As interest grew, bamboo began to be utilized as a cost-effective substitute for timber in constructing affordable housing in developing countries. This led to the development of various bamboo-based construction materials, including laminated bamboo, bamboo-reinforced concrete, and other biocomposites. These materials, categorized as unconventional, advanced polymer, and inorganic-based biocomposites, exhibit homogeneous quality, improved strength, and resistance to termites (Suhaily et al., 2013). Studies have also explored bamboo's potential as reinforcement in concrete (Asif, 2009).

Interestingly, the processing of bamboo, accompanied by value addition, not only enhances its structural strength but also elevates its aesthetic appeal and social benefits, albeit at a higher cost.

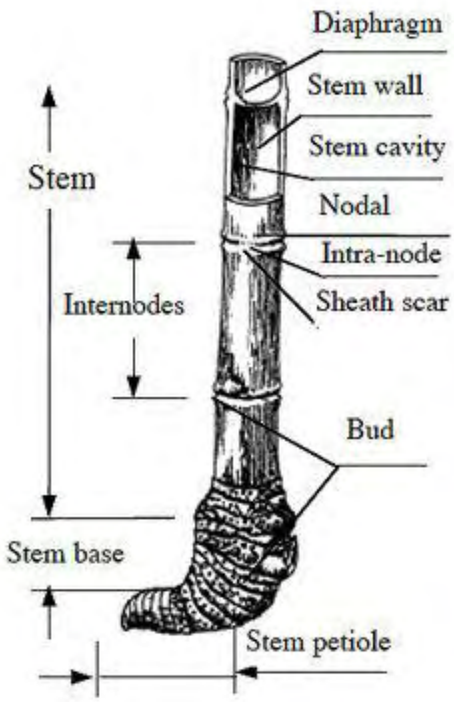
2.2 Bamboo anatomical features

Bamboos, members of the subfamily Bambusoideae, represent a diverse group of plants within the extensive grass family Poaceae. This subfamily, encompassing 1439 species distributed among 116 genera, is further classified into three tribes: Arundinarieae, housing temperate woody bamboos; Bambuseae, consisting of tropical woody bamboos; and Olyreae, which includes herbaceous bamboos (Sendulsky & de Barros, 2007).

The upper section of bamboo, known as the culm, encompasses the majority of its woody composition. Complementing the culm are various components such as a branching system, sheath, foliage leaves, flowering, and seedlings. The culm itself is characterized by its straight, hollow, and cylindrical structure, featuring nodes and internodes that establish connections between them. A crucial element fortifying the culm is the presence of a wooden partition that separates adjacent internodes. Bamboo species exhibit considerable variation in the length of internodes, the number and shape of nodes, culm diameter, and culm-wall thickness. Figure 7 provides an illustrative representation of bamboo anatomy (Roslan, 2018).

Internodes, forming bamboo cavities, are characterized by their hollow interior. Notably, the length of internodes increases from the bottom to the middle and gradually decreases towards the top. The bamboo culm diameter follows a similar pattern, decreasing from the bottom to the top as the culm wall thickness diminishes. This leads to the retention of the outer, more parenchymatous tissue at the expense of the inner, more sclerenchymatous tissue (Chaowana, 2021).

Figure 7: Division and nomenclature of bamboo anatomy.



Source: Adapted from Roslan, 2018.

Bamboo stems exhibit a circular cross-section, enveloped by a specialized tissue on both sides of the wall. The cortex, situated in the outer part, functions as a water-tight seal, safeguarding the living culm from moisture loss. The outer layer, rich in silica, imparts a hard and smooth texture to the exterior. Conversely, parenchyma cells are predominantly situated on the inner side of the bamboo stem (Huang, 2020).

As a naturally occurring composite, bamboo consists of cellulose fibers embedded in a lignin matrix. Renowned for its exceptional strength and properties, bamboo serves as a prominent raw material in Asian factories, contributing to the production of various wood products such as pulp and paper, plywood, Medium Density Fiberboard (MDF), Particleboard (PB), and Oriented Strand Board (OSB) (Kalali, 2019).

Bamboo and wood diverge in several key aspects. The bamboo culm is characterized by its straight, cylindrical, and hollow structure, shielded by a resilient epidermis and an inner wax layer. Notably absent are ray cells, radial pathways, and knots. Macroscopic features like culm diameter, culm-wall thickness, and internodal length are discernible structures in bamboo, while fiber distribution is a microscopic gradation (Chaowana, 2021). Although bamboo shares a primary chemical composition with wood, minor chemical distinctions exist, varying across different parts of the culm (Chaowana, 2021). As a result, wood processing methods, technology, and equipment cannot be applied blindly to bamboo utilization.

Bamboo's carbohydrate content is critical to its durability and service life. The chemical composition of bamboo is strongly related to its resistance to mold, fungal, and borer attacks (Liese, 2015). Chemical content (starch and sugar) will retard the absorption rate of H2O+ ions on the cement mineral surfaces and slow down the setting reaction when producing materials such as cement-bonded particleboard. As a result, information on bamboo properties is required for determining its suitability for composite manufacturing.

Furthermore, the potential of bamboo composite products necessitates the use of adhesive to join bamboo elements. Future development of bamboo-based composites will necessitate a thorough examination of bamboo glueability and strand bonding quality.

The culm tissue is made up of 45% parenchyma, 45% fibers, and ten percent conducting cells. The percentage distribution reveals a distinct pattern both horizontally and vertically within the culm. The inner third of the wall has more parenchyma and conducting cells, while the outer third has a higher percentage of fiber. The amount of fiber increases vertically from bottom to top as the parenchyma content decreases (Chaowana, 2013).

Parenchyma cells, distinguished by their thin walls, feature numerous simple pits on the longitudinal walls connecting them, with fewer pits on the horizontal walls. Within the vascular bundle, the inner and middle layers manifest larger proportions, while the outer layer is smaller and denser (Carlquist, 2018).

The bamboo culm's vascular bundle is made up of two large metaxylem vessels and a phloem (sieve tubes with companion cells). The vessels are larger in the center of the culm and become smaller as they move outward. Sclerenchyma cells surround the vessels and phloem. The shape, size, arrangement, and number of vascular bundles in the transverse section of the internode part can be used to classify the bamboo anatomical structure. Because the vascular bundles contrast with the lighter-colored parenchyma ground tissue. The presence and location of fiber strands on the cross-section can be used to differentiate four types of vascular bundles. The first type only has the central vascular strand supporting tissue as sclerenchyma. The second type consists only of the central vascular strand supporting tissue as sclerenchyma sheaths, but they are noticeably larger at the intercellular space than the other three types. The central vascular strand with sclerenchyma sheaths and one isolated fiber bundle make up the third type. The fourth type consists of a central vascular strand with small sclerenchyma sheaths on both sides and two isolated fiber bundles (Catling, 2012).

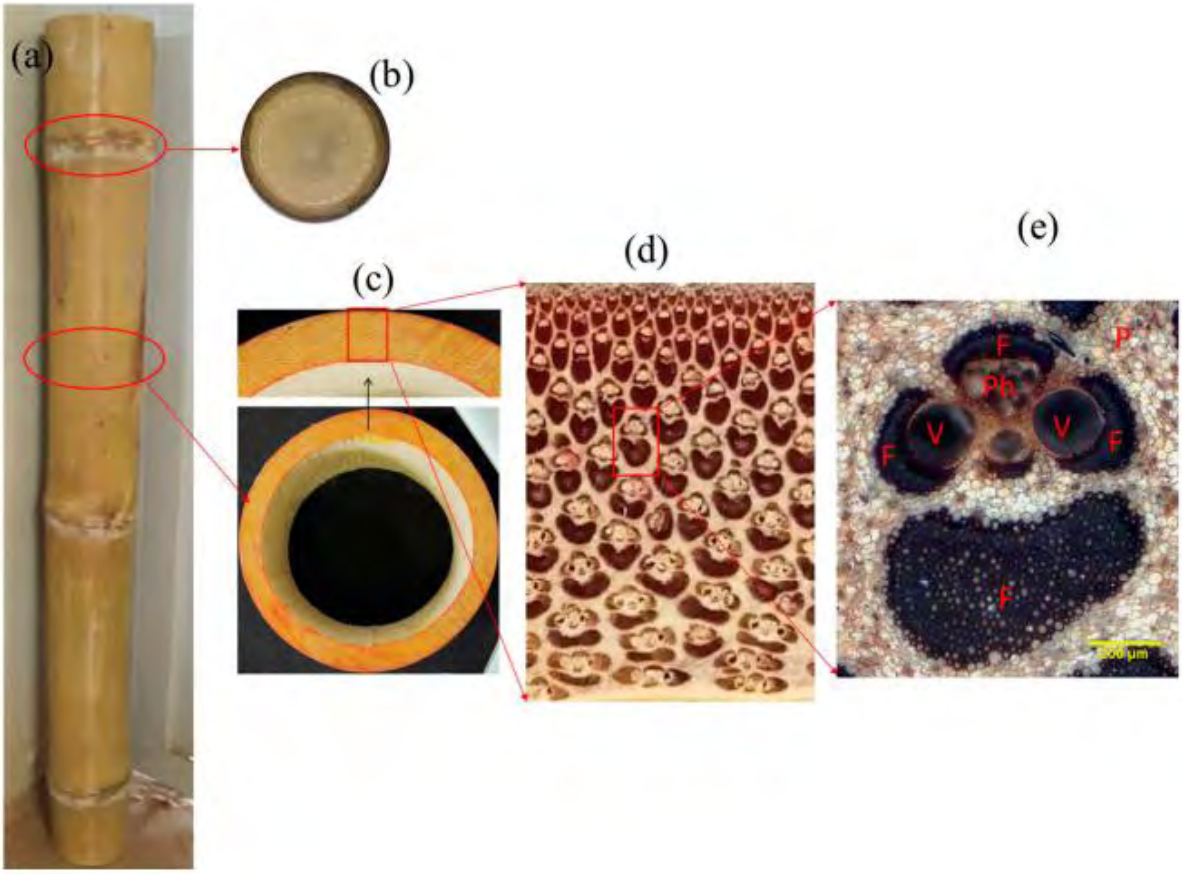
The fibers are made of sclerenchymatous tissue, have a thicker wall, and are long and tapered at the ends. The length-to-width ratio ranges between 150:1 and 250:1. They appear as caps of the vascular bundles and sheaths around the vessels in the internodes. They account for 40- 50% of total culm tissue. The number of fibers, such as sheaths or additional bundles, is proportional to the specific gravity, which rises within the culm from base to top and thus influences the strength properties. The fiber percentage is higher in the outer one-third of the wall and at the top of the culm, which contributes to its superior slenderness (Wahab, 2018).

Many studies on the anatomical features of bamboo that affect its physical and mechanical properties have been published. These characteristics are expected to have an impact on the final application of bamboo. Vascular bundle size (radial/tangential ratio) and fiber length correlated positively with the modulus of elasticity (MOE) and stress at the proportional limit (Wahab, 2018). The maturation process, marked by an increase in size and longer fiber length, has been observed to contribute to enhanced strength properties in bamboo. Notably, bamboo with longer fibers tends to exhibit greater stiffness, particularly when characterized by a larger vascular bundle size. Interestingly, an inverse relationship between fiber length and shear strength has been noted (Huang, 2018).

Furthermore, fiber wall thickness demonstrates a positive correlation with compression strength and modulus of elasticity (MOE), but conversely, a negative correlation with the modulus of rupture (MOR). A notable exception is compression strength, which exhibits a distinct relationship with lumen diameter and various mechanical properties (Mirmehdi, 2016).

The frequency of vascular bundles displays minimal variation concerning culm age or height. The higher density of vascular bundles at the culm's top is attributed to the decrease in culm wall thickness (Benhua, 2016). Figure 8 illustrates a diagram depicting the arrangement of vascular bundles. Importantly, the size of vascular bundles exhibits no significant differences with height or age, indicating an absence of correlation between vascular bundles and age. However, there is a noteworthy decrease in culm height. The presence of mature tissues contributes to a higher ratio of vascular bundle size near the basal location. Interestingly, within the culm's height, the radial size of vascular bundles decreases more rapidly than their longitudinal size, and neither the age nor height of the culm affects fiber wall thickness (Benhua, 2016).

Figure 8 - Diagram of the morphological characteristics of bamboo culm (a) segment of the bamboo culm (b) Cross-sectional view of bamboo, illustrating the node diaphragm (c) Cross-sectional representation of the internode (d) Segmental view through the culm wall (e) detailed view of the vascular bundle, including V (vessel), F (fiber), Ph (phloem), and P (parenchyma).



Source: Adapted from Kadivar 2020

2.3 Bamboo treatments

Environmental pressures have increased in recent years, resulting in significant changes in the field of wood-based materials protection. Thus, modern technologies that perform thermal or chemical modifications have been proposed to comply with the ban on biocide products.

Furthermore, bamboo modification has emerged as a new option for wood-based products preservation, describing the application of chemical, mechanical, physical, or biological methods to change the properties of wood material. Modification is being developed to improve the quality of wood materials associated with moisture sensitivity, low dimensional stability, hardness, and wear resistance, low resistance to biodeterioration against fungi, termites, and marine borers, and low resistance to UV irradiation.

Currently, wood and bamboo modifications are used to improve the material's physical, mechanical, or esthetic properties (Kelkar, 2023).

2.3.1 Chemical treatments

Chemical treatments are the most widely used and effective techniques of protecting wood and bamboo. Chemical treatments are required in applications where long-term durability and safety are critical problems, such as building applications (Gauss, 2021). There is a wide selection of potential chemicals available on the market and recommended by wood/bamboo standards. Its use will be determined by the application, toxicity, and treatment approach. Christian Gauss gathered in his thesis "Preservative treatment and chemical modification of bamboo for structural purposes" (Gauss, 2017) the most used chemical compounds in the market as well as their advantages and disadvantages, being shown in table 1.

Table 1: Most commercialized treatments for wood and bamboo.

Compound	Formula	Average Life	Advantages	Disadvantages
Disodium octaborate tetrahydrate (DOT)	$Na_2B_8O_{13}.4H_2O$	NA (Not Analyzed)	Self-diffusing, effective against fungi, insects, and termites	Highly leachable, not suitable for outdoor application
CCA (copper, chromium, arsenic)	47.5% CrO_3, 18.5% CuO, and 34% As_2O_5	NA	Fixable, broad spectrum biocide	Contains harmful chromium and arsenic
Bis-tri-butyl tin oxide (TBTO)	$[(CH_3CH_2CH_2CH_2)_3Sn]_2O$	NA	Difficult to leach	Toxic
Creosote	By-product of coal manufacture	24.9 years	Good use of an otherwise undesirable	Oil-soluble and expensive

			product	
Zinc chloride / Copper sulphate	ZnCl2/ CuSO4	NA	Hydroscopic, good fungicide, can retard fire as well	Not effective in outdoor applications, ineffective for termites and insects
Sodium pentachlorophenate	C6Cl5ONa	15.9 years	Effective fungicide	Banned in many countries because of presence of PCP
CCB * (copper, chromium, boron)	CuSO4:$K_2Cr_2O_7$:H3BO3 (3:4:1.5)	NA	Fungicide, insecticide	Low fixation, presence of chromium
AmmonicalCopperArsenate(ACA)	CuSO4:As2O3 (3:1) in ammonia	NA	High fixation	Presence of arsenic makes it toxic
Bis-(N-cyclohexyldiazeniumdioxide)-copper(Cu-HDO)	C12H22N4O4Cu	NA	Good fungicide, insecticide	Expensive, toxic to aquatic organisms
3-Iodo-2-propynyl butylcarbamate-(IPBC)	CH3(CH2)3NHCO2CH2C≡Cl	NA	Fungicide	Not an effective insecticide

Source: Adapted from Christian Gauss Thesis 2017.

- Alkaline treatment

Mercerization is the process of alkali treatment of natural fiber. According to ASTM D 1695, this is the process of exposing a vegetable fiber to the action of a concentrated aqueous solution of a strong base to cause significant swelling, resulting in changes in fine structure, dimensions, morphology, and mechanical properties.

The alkali treatment for bamboo begins with the careful selection of mature bamboo, ensuring that the harvested material is optimal for processing. After cleaning and stripping away outer layers to expose the fibers, the bamboo undergoes immersion in an alkali solution, commonly sodium hydroxide (NaOH)(Chen, 2021).

During the immersion phase, a chemical reaction unfolds between the alkali solution and the cellulose fibers within the bamboo. This reaction is essential in modifying the chemical structure of the bamboo, resulting in a variety of enhancements, with a considerable increase in strength, rendering the bamboo more robust and durable. This heightened durability, combined with improved flexibility, makes the treated bamboo an attractive option for industrial usage (Chen, 2021).

Moreover, alkali treatment contributes to the bamboo's resistance against decay, fungi, and insect infestations, ensuring a prolonged lifespan for products derived from treated bamboo. The process also improves dimensional stability, reducing the susceptibility of bamboo to expansion and contraction caused by fluctuations in humidity and temperature. This is particularly crucial for applications in construction materials, where stability is the most important parameter (Kaur, 2016).

Following the desired reaction period, the treated bamboo undergoes a meticulous washing process to eliminate any residual alkali solution. Neutralization may also be employed to prevent the retention of excessive alkalinity. Subsequent drying ensures that the treated bamboo is free from excess moisture, preventing mold growth and achieving the desired physical properties (Kaur, 2016).

Some studies showed that the treatment of the bamboo fibers with sodium hydroxide results in an improvement in the mechanical properties of composites made of bamboo. This enhancement is due to improved adhesion between the bamboo fibers and the resin. An alkali concentration of 6% was discovered to be optimal, resulting in the best mechanical properties for bamboo composites. Bamboo composites with this alkali concentration have bending, tensile, and compressive strength and stiffness higher than untreated composites (Allan C. Manalo, 2015).

2.3.2 Heat treatment

Heat treatment of bamboo alters its physical properties, mechanical properties, chemical composition, and anti-mildew properties significantly. The most important factors influencing bamboo performance after heat treatment are temperature, time, and environment.

- Steam heat treatment

Steam heat treatment is the process of heating bamboo using steam as the medium. Steam can soften bamboo while also isolating oxygen. Steam heat treatment is further classified into saturated steam treatment and superheated steam treatment based on the properties of the steam. When evaporation and condensation are in dynamic equilibrium during water evaporation, saturated steam is formed. It is distinguished by a one-to-one correspondence between temperature and pressure. Because of its high penetration and ability to lower the glass transition temperature (the critical temperature at which polymer material stiffness changes), saturated steam can soften bamboo at a low temperature while minimizing the loss of mechanical properties. Furthermore, saturated steam heat treatment has become one of the most important methods of bamboo modification due to its high efficiency and low pollution (Zhao Zhao, 2022).

The superheated steam treatment equipment consists of a bamboo oven and a superheated steam generator. The temperature of the steam can also be adjusted by heating it in the oven. There is no heating element in the tank (a container for placing bamboo) for saturated steam treatment equipment, and the steam temperature varies with steam pressure (Xixi Piao, 2022).

- **Oil heat treatmen**t

Oil heat treatment is the use of vegetable oil (tung oil, linseed oil, palm oil) or mineral oil (methyl silicone oil) as the heat transfer medium (Yang et al., 2020), which has the benefits of high heat transfer efficiency, uniform heating, and precise temperature control.
The results showed that the chemical composition of bamboo changed after treatment, and other properties such as physical and mechanical properties, decay resistance, and anti-mildew property were greatly improved (Yang et al., 2020).

- **Heat treatment with air or inert gas**

Air or an inert gas (such as nitrogen) can be used as the heat transfer medium in bamboo heat treatment in addition to steam and oil. Bamboo heat treatment has the potential to significantly improve bamboo quality. When bamboo was air-treated at 220 C, however, a reduction in mass and mechanical performance was observed due to the severe degradation of cellulose caused by oxygen. To avoid such losses and the risk of explosion caused by the presence of oxygen at high temperatures, some researchers used inert gas to create an atmosphere with no or low oxygen is needed (Zhang, 2012).

- **Hot water treatment**

Hot water treatment can remove some water-soluble substances in bamboo, such as starch, pectin, and sugar. This can improve the product's dimensional stability, anti-corrosion, and anti-mildew properties. Hot water treatment has also been used to soften bamboo culm or laminated bamboo before cutting it into veneers. Hot water treatment is typically time-consuming because the maximum water temperature at ordinary pressure is 100 C and a large amount of water is required. Furthermore, prolonged treatment with boiling water may cause bamboo properties to deteriorate (Li et al., 2020).

- **Fire heat treatmen**t

Traditional bamboo furniture and handicrafts are typically made by quickly softening the bamboo with fire and then straightening or bending it. Fire treatment is typically used on original bamboo poles and is applied only where straightening or bending is required. The procedure involves heating a specific spot with fire, slowly bending it, and then cooling and fixing the shape with water. This technique is also commonly used on bamboo poles used in construction and building. One disadvantage of this method is that the treated positions become extremely dark, resulting in a non-uniform color on the surface of the bamboo, which has a negative impact on the appearance and final product value. This method frequently results in cracking in bamboo poles (Suhaily, 2013).

2.4 Densification process and techniques

To meet the demand for environmental sustainability, there has been a significant push for bamboo and forest management over the years. As a result, industries are under pressure to develop products that make better use of the forest. The density of bamboo is related to several mechanical properties such as strength, hardness, and surface abrasion resistance. As a result, bamboo species with a higher density are frequently preferred for structural applications where strength is a critical factor. Given the positive correlation between density and various mechanical properties, several attempts have been made to increase the density of bamboo through modification; this process is known as densification. Densification refers to the process of enhancing material density and restructuring its microstructure. For materials featuring a cellular structure, densification represents the final stage in the compression stress-strain curves, occurring after the linear elastic and plateau regions. Following the conclusion of the elastic phase, cell collapse ensues, initiating inelastic behavior. Within this inelastic region, strain experiences a rapid increase with minimal or no change in stress, commonly referred to as the plateau region. With sustained compressive loading, all cells collapse, eliminating cell cavities and transforming the material into a solid body. This transformation significantly elevates stress, marking the onset of the densification region (Kadivar, 2020). Densification is essentially an increase in the quality and density of bamboo by reducing voids in the material using various techniques. These procedures increase the density of wood and, as a result, improve its mechanical properties, such as its Modulus of Rupture (MOR), Modulus of Elasticity (MOE), stiffness, and hardness (Heger, 2004).

Densification of bamboo can be accomplished through mechanical or chemical processes, such as compressing the bamboo and forcefully closing the voids or filling the porosity of the cell wall structure with chemicals to reduce the voids in solid bamboo; mechanical and chemical processes can also be combined for densification. The first densification processes and concepts were proposed in the early 1900s in the United States when patents for compressed bamboo were submitted (Gao, 2018). While early studies primarily focused on various techniques for the densification and compression of bamboo, recent studies are more focused on the stabilization of densified bamboo behaviors. Most of the studies investigated the effects of compression ratio (CR), temperature, pressing time, moisture content (MC), and other parameters on both densification methodologies and densified bamboo quality. Furthermore, optimization using statistical tools was performed to determine the impact of individual parameters as well as combinatorial effects on the responses (Kutnar et al., 2011).

The densification process takes advantage of bamboo's viscoelastic behavior, allowing low-density bamboo species to be engineered and used competitively in place of higher-density and higher-performance bamboo. Bamboo is composed of natural polymers like hemicellulose, lignin, and non-crystalline cellulose, possesses viscoelastic characteristics, positioning its mechanical behavior between linear elastic solids and viscous fluids, similar to polymers and wood. The softening temperature stage, represented by Tg, signifies a notable shift in amorphous polymers' properties, including increased molecular movement and damping properties, coupled with a substantial decrease in strength and elastic modulus. Below Tg, bamboo exhibits a glassy behavior, while at higher temperatures, it takes on a rubbery or viscous nature. The glass transition in bamboo, a viscoelastic material, is influenced by factors like moisture content, chemical composition, and testing method. For instance, in wood, the Tg ranges from 60 °C to 235 °C, with higher moisture content leading to a lower glass transition temperature. The amorphous constituents of bamboo display viscous behavior with an increase in temperature. However, this stage is not reached in wood before thermal degradation starts, resulting in the degradation of amorphous wood components at elevated temperatures. Bamboo undergoes similar stages when exposed to heat, with its mechanical properties dependent on temperature and moisture (Wang, 2018).

In summary, under short durations, low temperatures, and low moisture content, amorphous bamboo constituents exhibit high strength and modulus in a "glassy state." As temperature, moisture content, and duration increase, bamboo transitions to a rubbery state, making it more susceptible to deformation. Therefore, understanding the softening stage is crucial in any bamboo geometry deformation mechanism. Matan et al. (2007) demonstrated the dependence of the glass transition temperature on bamboo's initial moisture content for *Dendrocalamus asper* bamboo, suggesting a constant Tg value above 13% moisture content (between 100 and 120 °C). To characterize the final mechanical and physical properties of the bamboo, the process depends on the extent of the cellular structure collapse. This process is not only limited to softwoods; it can also be used to improve the stiffness and strength of hardwood species (Kadivar, 2020).

Fang et al. (2018) conducted a comprehensive examination of flattening methods and put forth two potential strategies to prevent crack formation. The initial proposal involves minimizing the differentiation in circumference between internal and external layers, achievable through techniques such as skin removal, culm splitting, or superficial scratches on the inner surface. The second method aims to alleviate tangential stress through chemical treatment, heat treatment, and/or an increase in moisture content. The synergistic application of these two solutions—geometric modification and material treatment—can enhance effectiveness in preventing cracks.

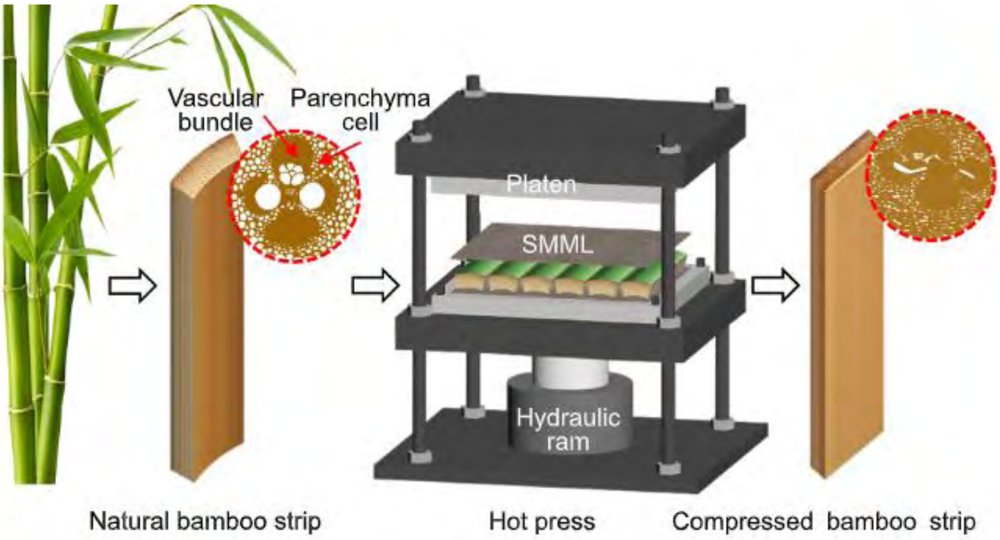
The process of bamboo densification begins with carefully selecting and harvesting bamboo culms, considering factors like size, age, and overall quality. After harvesting, the bamboo undergoes pre-treatment, involving washing and boiling to eliminate impurities and enhance surface properties.

Moisture conditioning is a crucial step, ensuring the bamboo reaches an optimal moisture content of 10% to 20%, influencing its viscoelastic behavior for the densification process. The next phase involves controlled heating, raising the bamboo's temperature above the glass transition temperatures of its components. This softens hemicelluloses at 40 °C, lignin between 50 °C and 100 °C, and cellulose above 100 °C, making the bamboo more pliable for compression.

The core of the densification process is compression. High pressure is applied to bamboo culms, reducing volume and significantly increasing density. This takes advantage of bamboo's viscoelastic nature, allowing it to deform under pressure and retain the new shape after release. Compression often occurs at elevated temperatures to enhance the softening process. Figure 9 illustrates the steps of the bamboo densification process.

After compression, the bamboo enters the cooling and fixation stage, where it cools and sets in its compressed form. Cooling and fixation are crucial to maintaining the new shape and structure. Fixation may involve chemical treatments or natural processes to stabilize the bamboo and prevent it from reverting to its original state (Kong, 2017). This demonstrates the significance of temperature, moisture, and load on the material in allowing the bamboo to be compressed without being crushed. Furthermore, compression is affected by anatomical factors such as density before densification, volume, and load direction.

Figure 9 - Structural transformation of compressed bamboo and its compression setup, with sintered metal mesh laminate plates (Smml)



Source: Adapted from Y Luan, et al, 2023.

2.4.1 Densification by mechanical methods

- **Densification by thermomechanical means (TM)**

Heating is one of the oldest and simplest methods of increasing the value and use of bamboo. Early research laid the groundwork for the concept of densification, which uses heat to improve dimensional stability and compression to increase the density and strength of bamboo (Chen, 2020).

Because there is no control over the moisture content (MC) of the material and relative humidity (RH) of the environment during open hot-press TM densification, pre-treatment is critical. As a result, during the heat-stabilization process, bamboo is preheated and conditioned to an MC of 13%, eliminating the risk of damage during the compression stage. (Navi P, 2000).

Following pre-heating, bamboo is compressed using a hot press at temperatures ranging from 130 to 180 °C, and then set and fixed by cooling the panels to less than the boiling point of water before compression is removed. Due to the thermoplastic nature of lignin, significant spring back will occur if this step is skipped. The finished product has a high density, strength, and dimensional stability (Zakikhani, 2014).

- **THM (thermo-hydro-mechanical) densification**

The THM process is the process of subjecting bamboo to the effects of temperature, moisture, and mechanical action. To overcome the drawbacks of densification and thermal treatment, such as recovery and spring back, a combination of these three effects is used. The injection of steam before or during the compression stage improves the dimensional stability of the bamboo by softening the lignin and hemicellulose matrix rather than degrading the hemicelluloses. Because the softened matrix and microfibrils move, mechanical properties are reduced and surface hardness is increased, while the dimensional stability of the densified bamboo is improved (Archila-Santos, 2014).

Through the promotion of viscous foam of timber substances, this process increases the compressibility of bamboo and reduces the level of stress stored by compression. With increasing temperatures and decreasing times, complete fixation can be achieved through hemicellulose hydrolysis and elution using moisture and its mechano-sorptive effect. This results in stable, permanently fixed bamboo with improved efficiency, dimensional stability, decreased hygroscopicity, and improved density, mechanical properties, and durability over TM Densified wood (Bao M, 2017).

The possible mechanism underlying shape fixation via steaming is explained by changes in the crystallinity and crystal width of the cellulose. Furthermore, changes in mechanical properties caused by steaming were explained using a slip-cure model, in which the bamboo cell wall acquires microfractures during compression and steaming causes a rearrangement of the crystalline cellulose, thereby determining the compressed shape of the bamboo (Heger F, 2004).

The technique of creating densified bamboo is similar to heat stabilization, with the exception that steam circulation is used to fixate the deformation of the bamboo rather than dry heat during compression. This necessitates the creation of holes or grooves on the surface of the compression plates. This process also employs an enclosed system, which allows for precise control of variables such as temperature, moisture content, pressure, and relative humidity within the closed system. Furthermore, the use of moisture during the treatment stages contributes to the hydrolysis of the celluloses and allows stress relaxation to occur in the bamboo, increasing dimensional stability and improving the physical and mechanical properties (Skyba, 2009).

- **Viscoelastic thermal compression (VTC)**

VTC densification takes advantage of the viscous and elastic nature of the polymers found in bamboo (cellulose, hemicellulose, and lignin).

The VTC densification process includes a continuous apparatus that allows for simultaneous heating and conditioning; bamboo softening via rapid vapor decompression; compression; annealing to promote thermal degradation and relaxation; and panel cooling and conditioning (Kamke F, 2004).

Bamboo viscoelastic polymers can behave as viscous fluids or as linear elastic solids depending on temperature, exposure time, and diluent concentration. High temperatures, high MCs, and longer exposure times produce bamboo with compliance and a rubbery state, whereas lower temperatures, low MCs, and shorter exposure times produce bamboo with glassy behavior. The properties of these viscoelastic polymers change when the bamboo exceeds its Tg, taking advantage of the mechanosorptive effect of bamboo.

Temperatures above the Tg, combined with rapid vapor decompression and water removal in the cell wall, soften the bamboo molecules and promote polymer mobility, preventing brittle fractures even with large cross-sectional movement (Kamke F, 2004).

The resulting material is a densified bamboo with increased strength, stiffness, and decreased hygroscopicity after treatment during this transition phase.

- **Surface densification**

Because surface densification affects only the first few millimeters of bamboo, the densification process is faster than usual, resulting in a change in the density profile of the densified bamboo board. Surface densification, which uses the same principles as heat stabilization, uses heat to press the first few millimeters of the bamboo. Early studies investigated this by using a belt and heated shoes that moved in a straight line. This process sandwiched the panels and cooled them down at the end with water-cooled shoes. This exposes the bamboo to short bursts of heat and pressure, followed by a cooling step (Kadivar, 2020).

The process of surface densification, however, is not limited to this. In recent years, various processes have emerged, such as the use of linear vibration friction technology (LVFT), VTC, THM, or regular heat-stabilization with altered compression times. All of these have successfully changed the density profiles of the bamboo and increased the densities of the bamboo's surface. Due to the variation of density peaks and valleys, changes in density profiles have the potential for different applications, and because wear and abrasion resistance are mostly density-dependent, this type of bamboo can be used in applications such as flooring, where surface hardness and softcore are required (Wang & Cooper, 2005).

2.4.2 Pretreatments for densification

The pursuit of dimensional stability and reduced SR on densified bamboo has led researchers to investigate the use of chemicals to impregnate bamboo, thereby improving its mechanical properties. Chemical modification is a process in which the polymeric constituents of bamboo, such as lignin, hemicellulose, or cellulose, undergo a chemical reaction with an agent, resulting in the formation of a stable covalent bond between the reagent and the constituents of bamboo because the primary purpose of chemicals in bamboo is to improve dimensional stability, overall durability, and lifespan, the combination of impregnation and densification complements each other. Acetic anhydride, phenol-formaldehyde, and melamine-formaldehyde are some of the chemicals used (Azeez, 2018).

The removal of undesirable components optimizes bamboo for densification, ensuring adaptability and consistent outcomes. The environmental and economic advantages of well-prepared bamboo contribute to its viability as a renewable resource in construction and manufacturing. In table 2 is presented some of the commonly used pretreatments for densification.

Table 2: Summary of advantages and disadvantages of pretreatments for bamboo densification

Pretreatment	Process	Purpose	Advantage	Disadvantage
Heat Treatment (li,2022)	Bamboo is subjected to high temperatures in the range of 160 to 240 degrees Celsius in an oxygen-limited environment.	Heat treatment helps in reducing bamboo's hygroscopicity (ability to absorb moisture), enhances dimensional stability, and improves resistance to decay and insects.	Increased dimensional stability, improved resistance to decay.	High energy consumption.
Steam Treatment (Zhang, 2019)	Bamboo culms are exposed to steam at elevated temperatures.	Steam treatment makes bamboo more flexible, facilitating the subsequent densification process. It also helps in reducing brittleness.	Softens bamboo for easier shaping, enhances durability.	Energy-intensive, may cause discoloration.
Acetylation (Guo, 2022)	Bamboo is treated with acetic anhydride, leading to the substitution of hydroxyl groups in bamboo with acetyl groups.	Acetylation increases bamboo's dimensional stability, reduces its hygroscopicity, and enhances resistance to decay and insect damage.	Improved dimensional stability, resistance to decay.	Costly, may affect natural bamboo appearance.
Alkali Treatment (Chan,2023)	Bamboo is treated with alkali solutions, such as sodium hydroxide.	Alkali treatment removes hemicelluloses and lignin from bamboo fibers, increasing its porosity and making it more receptive to subsequent densification processes.	Enhanced strength, flexibility, and decay resistance.	Potential environmental impact, requires careful handling of alkali.

Enzymatic Treatment (Liu,2012)	Enzymes are applied to bamboo to break down Bamboo components.	Enzymatic treatment modifies bamboo's cell wall structure, making it more amenable to densification. It can improve the overall mechanical properties.	Environmentally friendly, precise control over properties.	Longer processing times may be costly.
Fungal Pretreatment (Fatriasari, 2014)	Bamboo is exposed to certain fungi, leading to partial degradation of hemicellulose and lignin.	Fungal pretreatment modifies the bamboo's structure, making it more receptive to densification. It can improve the dimensional stability and mechanical properties.	Biodegradable, potential for enhanced mechanical properties.	Variable results, potential for uncontrollable decay.
Water-Soluble Extractives Removal (Zhao,2019)	Bamboo is treated to remove water-soluble components, including sugars, starches, and other extractives.	Extractive removal minimizes bamboo's susceptibility to biological attacks and improves its bonding capabilities during densification processes.	Improved durability and dimensional stability.	Labor-intensive, may affect natural bamboo color.
Ozone Treatment (Wu, 2012)	Bamboo is exposed to ozone gas.	Ozone treatment modifies the chemical composition of bamboo, enhancing its resistance to decay and insects. It also influences the bamboo's color and can improve its mechanical properties.	Environmentally friendly, effective against pathogens.	Equipment costs, potential health risks.
Silicon Impregnation (Cheng, 2018)	Bamboo is impregnated with silicon-based compounds.	Silicon impregnation increases bamboo's hardness, abrasion resistance, and dimensional stability. It can also contribute to enhanced resistance against fungal decay.	Increased hardness, resistance to insects.	May affect visual appearance, potential environmental concerns.
Sodium Silicate Treatment (Wang, 2010)	Bamboo is treated with sodium silicate solutions.	Sodium silicate treatment reinforces bamboo fibers, contributing to increased strength and stiffness. It also enhances fire resistance and dimensional stability.	Improved fire resistance, hardness	May affect bamboo's natural appearance, potential environmental impact.
Combined Steam and Lactic Acid Treatment (Suzuki,2023)	Bamboo undergoes a dual treatment involving steam and lactic acid.	This combined treatment can improve the flexibility and plasticity of bamboo, making it more amenable to densification processes. It also enhances durability.	Improved dimensional stability, reduced environmental impact.	Complexity in treatment process.

Polyethylene Glycol (PEG) Impregnation (Rao, 2019)	Bamboo is impregnated with polyethylene glycol.	PEG impregnation increases bamboo's moisture resistance and improves its dimensional stability. It can also enhance the densification process by reducing the risk of cracks.	Enhanced dimensional stability.	Energy-intensive, potential environmental concerns.
Peroxide Bleaching (Okan, 2013)	Bamboo is subjected to peroxide bleaching.	Peroxide bleaching modifies the color of bamboo and can influence its mechanical properties. It also removes impurities and improves the overall aesthetics.	Color lightening, improved appearance.	Chemical usage, potential impact on mechanical properties.
Malic Acid Treatment (Chen, 2016)	Bamboo is treated with malic acid.	Malic acid treatment contributes to the removal of undesirable components, enhancing bamboo's compatibility with densification processes. It can improve the material's durability.	Eco-friendly, potential improvement in properties.	Limited studies, variable results.
Ammonium Persulfate Treatment (Shen, 2021)	Bamboo is treated with ammonium persulfate.	Ammonium persulfate treatment modifies bamboo's surface chemistry, enhancing its reactivity during densification processes. It can lead to improved bonding and densification characteristics.	Improved hardness and decay resistance.	Chemical handling, potential environmental impact.
Polyvinyl Alcohol (PVA) Impregnation (Adamu, 2019)	Bamboo is impregnated with polyvinyl alcohol solutions.	PVA impregnation can enhance the bonding properties of bamboo, contributing to improved strength and durability during the densification process.	Improved dimensional stability, moisture resistance.	Costly, potential environmental concerns.
Formic Acid Treatment (Sun, 2008)	Bamboo undergoes treatment with formic acid.	Formic acid treatment modifies the bamboo's cell wall structure, making it more amenable to compression during densification. It can influence mechanical properties and dimensional stability.	Enhanced dimensional stability.	Handling safety concerns, potential environmental impact

Microbial Pretreatment (Fuke, 2021)	Specific microbes are employed to pre-digest bamboo.	Microbial pretreatment breaks down certain components, facilitating subsequent densification. It can influence bamboo's structure and enhance its overall mechanical properties.	Environmentally friendly, potential for controlled degradation.	Variable results, longer processing times.
Polymer Impregnation (Widiastuti, 2018)	Bamboo is impregnated with polymer solutions.	Polymer impregnation can improve bamboo's toughness, impact resistance, and overall mechanical performance. It enhances the material's suitability for applications requiring high strength.	Enhanced hardness and dimensional stability.	May affect visual appearance, potential cost.
Ethanol-Water Extraction (Ma, 2013)	Bamboo is treated with ethanol-water solutions to extract certain components.	Ethanol-water extraction can improve the compatibility of bamboo with densification processes. It may influence the material's porosity and mechanical properties.	Environmentally friendly, potential improvement in properties.	Variable results, potential cost.
Alkaline Peroxide Treatment (Yamashita, 2010)	Bamboo is treated with a combination of alkaline and peroxide solutions.	Alkaline peroxide treatment can alter the bamboo's chemical composition, making it more receptive to subsequent densification. It may improve dimensional stability.	Improved dimensional stability, potential for color improvement.	Chemical handling, potential environmental impact.
Acoustic Wave Treatment (Carrasco, 2023)	Bamboo is exposed to acoustic waves.	Acoustic wave treatment can induce changes in bamboo's microstructure, influencing its mechanical properties. It is an innovative approach to enhancing bamboo for various applications.	Non-destructive, potential for improved properties.	Limited studies, equipment costs.
Silane Coupling Agent Treatment (Ismail, 2002)	Bamboo is treated with silane coupling agents.	Silane treatment enhances the bonding between bamboo fibers and matrix materials during densification. It improves the overall compatibility of bamboo composites.	Improved adhesion, resistance to moisture.	Potential impact on appearance, cost.

Ionic Liquid Treatment (Muhammad, 2012)	Bamboo is treated with ionic liquid solutions.	Ionic liquid treatment influences the structure of bamboo fibers, making them more receptive to densification. It can improve the overall strength and durability of the densified material.	Environmentally friendly, potential for controlled properties.	Limited availability, potential cost.
Urea-Formaldehyde (UF) Resin Impregnation (Liang, 2021)	Bamboo is impregnated with urea-formaldehyde resin.	UF resin impregnation enhances the bonding between bamboo fibers, contributing to improved strength and stiffness during densification. It also enhances resistance to moisture.	Enhanced hardness, durability.	Potential formaldehyde emissions, cost.
Citric Acid Treatment (Gauss, 2021)	Bamboo undergoes treatment with citric acid.	Citric acid treatment modifies the surface chemistry of bamboo, influencing its reactivity during densification. It can improve bonding characteristics and overall mechanical performance.	Eco-friendly, potential improvement in properties.	variable results, potential cost.
Hydrogen Peroxide Bleaching (Lu, 2006)	Bamboo is subjected to hydrogen peroxide bleaching.	Hydrogen peroxide bleaching alters the color of bamboo and may influence its mechanical properties. It is often used as a preparatory step before densification.	Color lightening, potential improvement in appearance.	Chemical usage, potential impact on properties.
Phosphoric Acid Treatment (Hong, 2012)	Bamboo is treated with phosphoric acid.	Phosphoric acid treatment modifies bamboo's chemical composition, enhancing its suitability for densification processes. It can influence both structural and mechanical properties.	Improved hardness, potential resistance to decay.	Potential impact on appearance, cost.
Nitric Acid Treatment (Zhang, 2014)	Bamboo undergoes treatment with nitric acid.	Nitric acid treatment introduces chemical modifications to bamboo, affecting its reactivity during densification. It may enhance the material's resistance to decay.	Enhanced hardness and decay resistance.	Chemical handling, potential environmental impact.

Microwave Treatment (Nordin, 2010)	Bamboo is exposed to microwave radiation.	Microwave treatment induces changes in bamboo's microstructure, potentially improving its mechanical properties. It is an energy-efficient method for preparing bamboo for densification.	Energy-efficient, potential for controlled properties.	Uneven treatment, equipment costs.
Oxygen Plasma Treatment (Liu, 2015)	Bamboo is exposed to oxygen plasma.	Oxygen plasma treatment modifies the surface properties of bamboo, enhancing its wettability and adhesion properties. This can positively impact the bonding during densification processes.	Improved adhesion, environmentally friendly.	Limited studies, equipment costs.
Alkaline Peroxide-Urea Treatment (Ni, 2018)	Bamboo is treated with a combination of alkaline, peroxide, and urea solutions.	This multifaceted treatment alters both the surface chemistry and internal structure of bamboo, making it more receptive to densification. It can improve strength and dimensional stability.	Improved dimensional stability, potential for color improvement	Chemical handling, potential environmental impact.
Alkaline Sulfite Treatment (Qin, 2015)	Bamboo undergoes treatment with alkaline sulfite solutions.	Alkaline sulfite treatment can selectively remove lignin from bamboo, improving its fiber flexibility and making it more conducive to densification. It may enhance the overall mechanical properties.	Improved dimensional stability, potential for color improvement.	Chemical handling, potential environmental impact.
Polyvinyl Acetate (PVA) Impregnation (Li, 2014)	Bamboo is impregnated with polyvinyl acetate solutions.	PVA impregnation improves the bonding between bamboo fibers, contributing to increased strength and stiffness during densification. It enhances the material's resistance to moisture.	Enhanced dimensional stability, moisture resistance	Costly, potential environmental concerns
Chitosan Treatment (Sun, 2012)	Bamboo is treated with chitosan, a derivative of chitin.	Chitosan treatment imparts antimicrobial properties to bamboo, reducing susceptibility to decay. It can also modify bamboo's surface characteristics, influencing densification processes.	Eco-friendly, potential for improved decay resistance.	Variable results, potential cost.

Lignin-Depolymerization Treatment (Chio, 2019)	Bamboo lignin is selectively depolymerized.	Lignin depolymerization alters the composition of bamboo, potentially improving its reactivity during densification. It can lead to enhanced bonding and mechanical performance.	Potential improvement in mechanical properties.	Limited studies, variable results.
Citric Acid-Phosphoric Acid Treatment (Holilah, 2022)	Bamboo undergoes treatment with a combination of citric acid and phosphoric acid.	This combined treatment can modify both the surface and internal properties of bamboo, making it more adaptable to densification processes. It may influence both strength and durability.	Eco-friendly, potential improvement in properties.	Variable results, potential cost.
Potassium Hydroxide (KOH) Treatment (Ra, 2020)	Bamboo is treated with potassium hydroxide solutions.	KOH treatment can selectively remove lignin from bamboo fibers, improving their flexibility. It enhances the material's receptivity to densification and can influence mechanical properties.	Improved hardness and decay resistance.	Chemical handling, potential impact on appearance.
Ethylene Diamine Treatment (Ling, 2022)	Bamboo is treated with ethylene diamine.	Ethylene diamine treatment modifies bamboo's surface chemistry, potentially improving its adhesion properties. It can contribute to better bonding during densification processes.	Enhanced hardness and decay resistance	Chemical handling, potential environmental impact.
Acetylation (Cai, 2013)	Bamboo undergoes acetylation, involving the introduction of acetyl groups.	Acetylation alters bamboo's cell wall structure, improving its resistance to decay and dimensional stability. It can positively influence the densification process.	Enhanced Dimensional stability and improved appearance	Costly and complex process

Source: adapted from various sources

2.5 Delignification

The most abundant renewable resources are cellulose, lignin, and plant oils. The main constituents of bamboo are three macromolecular species: cellulose, hemicellulose, and lignin. Pectin, fats, wax, moisture, and water solubles are minor components. The most well-known and widely available of these three is cellulose. Photosynthesis is estimated to produce 830 million tons of cellulose per year (Gilbert, 2000).

Hemicellulose is a low-molecular-weight polysaccharide with a degree of polymerization ranging from 70 to 200, with softwood having lower degrees of polymerization. When hemicellulose is isolated, it is found to be amorphous and either water soluble or has a strong swelling behavior in water. Hemicelluloses are heteroglycans composed of a limited number of sugar residues (Ebringerová, 2005).

Lignin is found in all vascular plants as a cell wall component, as well as in the woody stems of arborescent angiosperms (hardwoods) and gymnosperms. The lignin content of woody stems ranges from 15% to 40%. Lignin serves as a water sealant in the stems and is crucial in controlling water transport through the cell wall. It also protects plants from biological attack by interfering with enzyme penetration. Finally, lignin is a permanent glue that bonds cells together in woody stems, giving the stems their well-known rigidity and impact resistance (Esther, 2012).

2.5.1 Lignin

Lignin is broadly defined as "polymeric natural products derived from the dehydrogenative polymerization of three primary precursors: trans-coniferyl, trans-sinapyl, and trans-p-coumaryl" (Collinson, 2010).

Enzyme-initiated polymerization produces extremely stable bonds: Biphenyl carbon-carbon linkages between aromatic carbons, alkyl-aryl carbon-carbon linkages between an aliphatic and aromatic carbon, and hydrolysis-resistant ether linkages are all examples of carbon-carbon linkages (Macfarlene, 2014).

The relative amounts of the respective precursors, and thus the final structure of lignin, is determined by the plant type. However, due to the similar base structure of the lignin precursors, each lignin molecule is made up of phenyl-propane unit sequences. To account for molecular weight effects, functional groups on the lignin molecule are generally reported per phenyl-propane unit (PPU), (Sun, 2020).

2.5.2 Delignification mechanism

Delignification, or the extraction of lignin from plant sources, can be accomplished through a variety of methods. Its goal is to break down the lignocellulosic structure into its fibrous components. Delignification processes are classified into two types: chemical and solvent processes.

The two most common and widely used conventional and industrial pulping processes are sulfite and alkaline pulping, both of which are classified as chemical pulping processes

(Mboowa, 2021). They are the pH scale's two extremes, where the alkaline or kraft pulping process produces alkali- or thio-lignin, whereas the acidic sulfite process produces lignin sulphonic acid. Lignin sulphonic acid is also known as lignosulfonate. The details of these pulping processes, as well as the effects on lignin under harsh alkaline or acidic conditions, are well documented. It is important to note that kraft pulping has become the dominant industrial pulping process (Hubbe, 2019).

Solvent-based or Organosolv pulping processes are either in development or are used on a small scale commercially. ASAM (alkali-sulfite-anthraquinone-methanol), Organocell (sodium hydroxide, methanol, and anthraquinone), Alcell (water and ethanol), Formacell (acetic acid, formic acid), and Milox are some of these processes (multistage peroxy-acid treatment). Except for the ASAM process, where inorganics are primarily responsible for delignification, these processes delignify lignocellulosic using organic solvents. They usually promise increased efficiency, fewer byproducts, lower capital costs, and/or lower emissions. Because of the chemical structure of their respective lignins, organosolv delignification is more selective for hardwoods than softwoods (Thielemans, 2004).

Clean-fractionation is a relatively new delignification method that uses steam explosion of biomass before solvent treatments and/or distillation (Grzegorz, 2012).

While steam explosion has been around for centuries, it has only been considered as an economical alternative for producing fuel and chemicals from biomass in the last two decades. Clean fractionation is an Organosolv method with the advantages of high organic solvent recovery (>99%), higher energy efficiency than current industrial processes, and, most importantly, the ability to recover all constitutive bamboo constituents without destructive degradation of any of these components (Iroegbu, 2021).

The high solvent recovery reduces downstream effluent treatment by eliminating downstream solvent evaporation and thus odorous emissions. It also reduces the large capital outlay required for other delignification processes.

However, because this technology is still relatively new, it has yet to be used on a large industrial scale for lignin production and is currently limited to laboratory-scale experiments.

3 Materials and methods

3.1 Material and sample acquisition

Mature bamboo poles of the *Dendrocalamus asper* species, aged three years, controlled and cataloged since their planting, were harvested from an experimental field situated in the state of São Paulo, Brazil (21°59'S 47°26'W). *Dendrocalamus asper,* also known as Rough Bamboo or Giant Bamboo, is a massive dense clumping tropical and subtropical species native to Southeast Asia. This bamboo is used for heavy construction, and the shoots are eaten as a vegetable (Banik, 2016).

D. asper is a sympodial bamboo with densely tufted leaves. The standing culm is a dark green color. That can grow to a height of about 20-30 m; lower nodes are covered with a circle of rootlets; internode length is 20-45 cm with a diameter of 8-20 cm and relatively thick walls (11-20 mm), sometimes almost solid at the base, and when young it is covered with fine, golden-brown hairs, giving the overall appearance of velvet.

D. asper has typical bamboo anatomical characteristics, such as the presence of vascular bundles and parenchyma. It is classified as anatomical group D because it has type four vascular bundles. The dimensions of *D. asper* fiber are quite similar to those of softwood tracheids when compared to some wood species (Wang, 2014). It has fibers that are approximately 2 to 3 times longer than those of hardwood species. As a result, it can be used as a substitute raw material in the pulp and paper industries.

Bamboo's physical and mechanical properties are affected by species, location/soil and climatic conditions, silvicultural treatment, harvesting technique, age, density, moisture content, position in the culm, nodes or internodes, and bio-degradation (Ebanyenle, 2007).

The moisture content of bamboo varies from bottom to top and from outer layer to inner layer. D. asper has a high specific gravity when compared to softwood and hardwood species used in panel manufacturing.

Bamboo, like wood, undergoes anisotropic shrinkage, where different directions experience varying rates of shrinkage. In the case of *D. asper*, longitudinal shrinkage is notably low due to the organized orientation of fibers along its length. Interestingly, the most significant shrinkage occurs in the tangential direction, approximately 1.3 times greater than radial shrinkage. This tangential shrinkage is approximately twice as significant as the radial shrinkage observed in both softwood and hardwood. These variations in shrinkage rates can be attributed to the unique fiber orientation, vascular bundle structure, and cell wall composition in bamboo, coupled with growth ring formation (Anokye, 2014).

The MOR and MOE have ranges of 92.5 to 135 MPa and 13,115 to 63,000 MPa, respectively. D. asper is stronger than wood in bending, tension parallel to the grain, and compression perpendicular to the grain, and similar in shear parallel to grain (Hamburg, 2009).

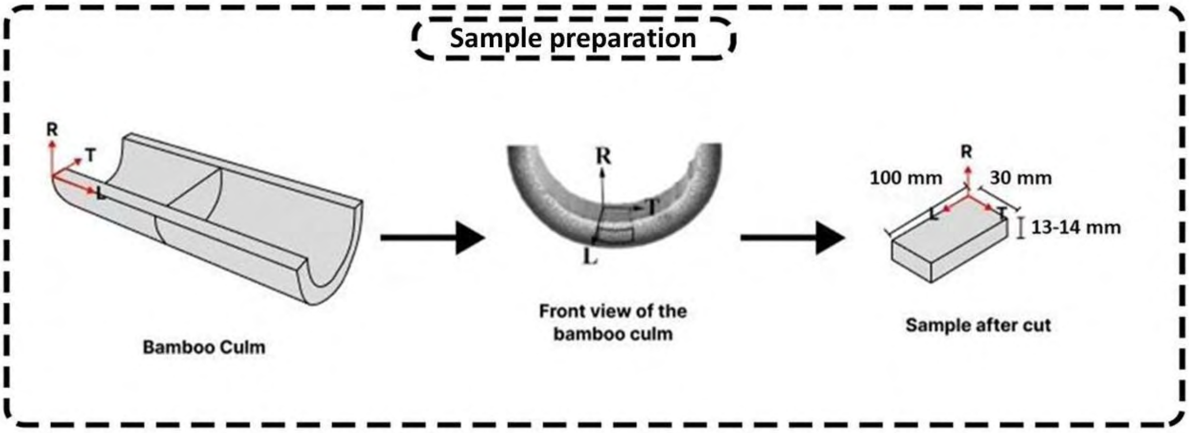
The chemical composition of bamboo should be considered for the use of wood-composite products. The chemical composition of each bamboo varies according to age, height, and layer. It also has an impact on its physical and mechanical properties. *D. asper* culms' main chemical constituents are cellulose, hemicellulose, and lignin, which account for more than 90% of the total mass. Bamboo's minor constituents account for 10% of its total weight and are made up of resins, tannins, waxes, and inorganic salts (Chaowana, 2013).

Except for the higher ash content, the chemical compositions of D. asper are similar to those of hardwoods. Bamboo ash is made up of inorganic minerals, primarily silica, calcium, and potassium. Other common minerals include manganese and magnesium (Fatriasari, 2014) Higher ash content in some bamboo species can harm processing equipment. In addition to cellulose and lignin, bamboo contains other organic components. It's made up of starch, deoxidized saccharide, fat, and protein.

The holocellulose, α-cellulose lignin, hot and cold-water solubility, and 1% NaOH solubility all vary slightly with the location. The ash content is highest at the top and lowest at the bottom. The solubility of alcohol in benzene increases significantly with location. Furthermore, the internode of bamboo has significantly more lignin, ash, and 1% NaOH than the node, while the internode has greater hot- and cold-water solubility than the node (Kamthai, 2003).

The harvested culms, with an average diameter of 18 cm, underwent a preservation process based on the technique explained by Kadivar et al. (2022). This preservation method involves immersing the bamboo poles in a solution of Disodium octaborate tetrahydrate (DOT) for a duration of 5 days, followed by a subsequent 3-day drying period at room temperature, ensuring the long-term durability of the bamboo. The processed bamboo poles underwent storage in a controlled environment at room temperature for a duration of four months, facilitating the attainment of an equilibrium moisture content within the range of 9 to 11%. Samples, each with average dimensions of 100 mm in length and 30 mm in width, were sourced from various internodes situated in the central portion of the culms (refer to Figure 10). The wall thickness of all samples measured between 13 and 14 mm.

Figure 10: Sample preparation from the bamboo culm.

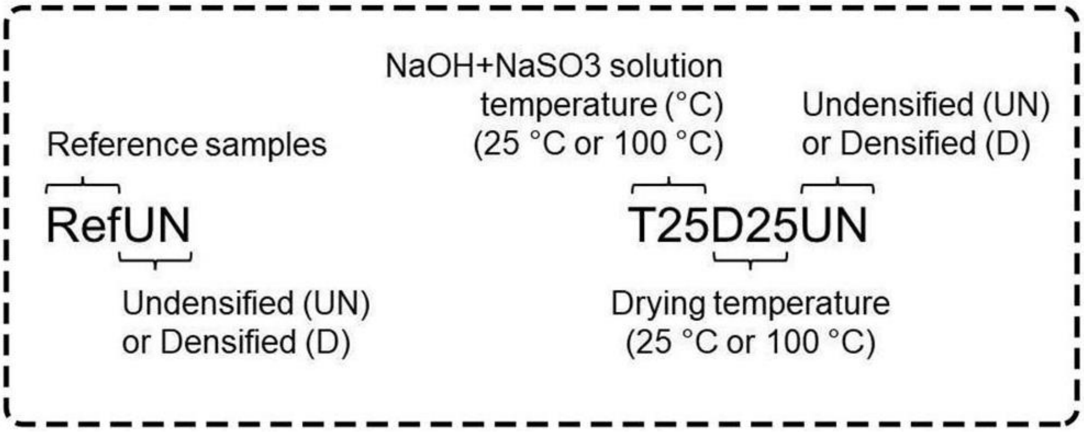


Source: Own authorship

3.2 Methods

The specimens underwent a sequential series of delignification, followed by drying and densification. Subsequently, different samples were prepared to undergo physical, mechanical, chemical, and thermal tests. Before the commencement of testing, all samples underwent meticulous conditioning in a controlled environment set at 25 ± 2 ºC and 65 ± 3% relative humidity. For ease of identification and subsequent analysis, each group was assigned a unique code (refer to Figure 11). The sequence of administered treatments and the resulting groups are visually presented in Figure 12. A summary of the number of samples for each treatment group and their dimensions for each evaluation is provided in Table 3.

Figure 11: Sample group identification



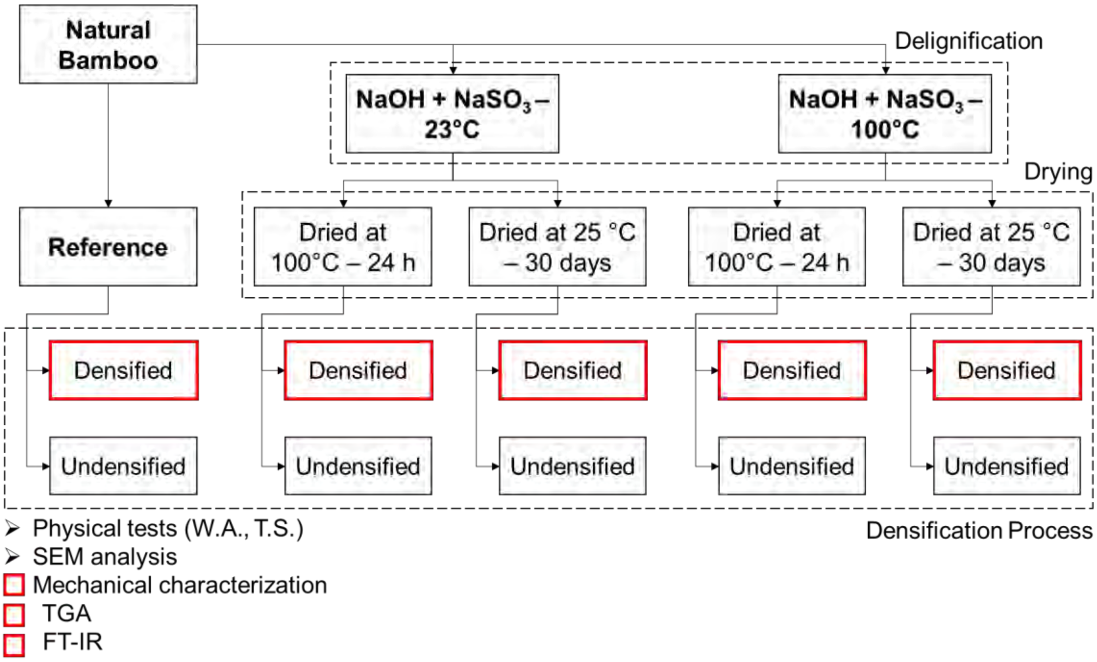
Source: Own authorship

Table 3: Distribution of samples for evaluation.

Type of test	Dimensions (L x W x T) (mm)	Number of samples per group	Total number of samples	Densification
Physical properties	21 x 21 x t	5	25	Densified
	21 x 21 x t	3	15	Undensified

Image analysis	21 x 21 x t	1	5	Densified
	21 x 21 x t	1	5	Undensified
Three point bending test	100 x 8 x 7	9	45	Densified
TGA and FT-IR	Bamboo powder	-	-	Densified

Figure 12: Fluxogram of treatment and evaluation.

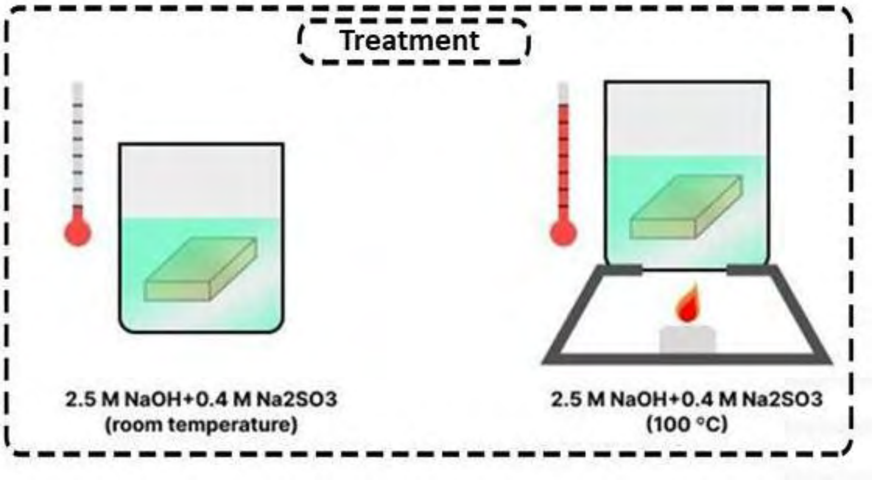


Source: Own authorship

3.3 Delignification

The delignification process employed a solution comprising sodium hydroxide (NaOH) at a concentration of 2.5 mol.L^{-1}, in combination with sodium sulfite (Na_2SO_3) at a concentration of 0.4 mol.L^{-1} where the solution influences the acetyl group in hemicellulose and the linkages of lignin–carbohydrate ester. Importantly, this treatment does not cause significant disruption to the aromatic structure of lignin. Bamboo samples were categorized into two distinct groups and immersed in the solution. One group underwent immersion in a boiling aqueous solution of (100 ± 2) ºC for approximately one hour, while the other group was subjected to the same composition of the aqueous solution at room temperature (25 ± 2) ºC for a duration of one hour (refer to Figure 13). After the treatment, the removal of the solution involved both groups being immersed in boiling deionized water repeatedly until a neutral pH was achieved.

Figure 13: Scheme of delignification process.

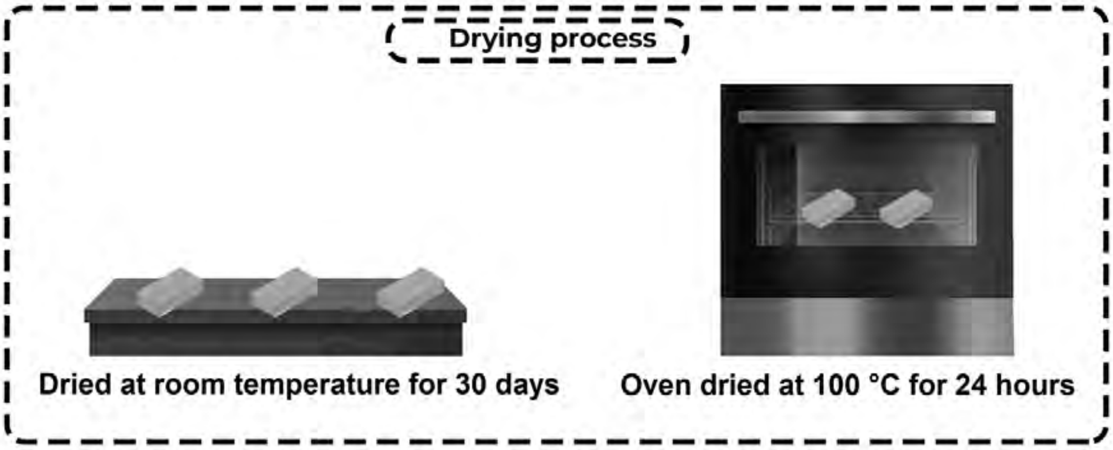


Source: Own authorship.

3.4 Drying process

Drying bamboo is a crucial step in enhancing its overall quality and usability. By removing excess moisture, the process helps prevent decay and rot, safeguarding the bamboo against fungal growth. Additionally, drying contributes to increased strength and hardness, making bamboo more resilient in construction and furniture applications. This method also minimizes the risk of insect infestations, as pests are less attracted to dry conditions. Furthermore, drying reduces the natural shrinkage and expansion tendencies of bamboo, mitigating the potential for cracking or warping. Improved aesthetics, with reduced discoloration and mold growth, are additional benefits of the drying process, especially in decorative applications. The increased durability of properly dried bamboo makes it suitable for outdoor use. Moreover, the material becomes easier to work with, allowing for more effective cutting, shaping, and manipulation. Bamboo samples obtained from each delignification process underwent two distinct drying procedures. The first method involved a 30-day conditioning period in a controlled environment at room temperature (25 ± 5) ºC with (65 ± 3)% relative humidity. The second method entailed placing the samples in an oven at 100ºC for 24 h, as illustrated in Figure 14.

Figure 14: Scheme of drying process.



Source: Own authorship

3.5 Densification process

In the densification process, the samples experience radial compression within an open thermo-hydraulic press system. This compression leads to a reduction in the internal volume of the material, caused by either plasticization or the collapse of cell walls (Dixon, 2016). Following the methodology described by C. Gauss and Kadivar (2021), a universal testing machine, specifically the EMIC DL-30000 equipped with a 300 kN load cell, was utilized. The machine incorporated a small press plates system designed to heat the samples and apply the desired pressure, as shown in Figure 15. In the experimental procedure, prior to pressing, precise measurements of the specimens were conducted, with a weight accuracy of 0.01

grams and a measurement accuracy of 0.01 millimeters. The densification degree (DD) was determined by measuring the thickness of the test specimens twice along the pressing direction: first before the pressing process and then immediately after removal from the pressing equipment. The densification degree (DD) was calculated using the formula shown in Equation (1):

$$DD = (T0 - T1)/T0 \times 100\% \quad (1)$$

Here, T0 represents the initial thickness of the samples before densification, and T1 represents the thickness after the densification process. The formula allows for the quantification of the densification degree as a percentage, making sure of the thickness reduction during the pressing procedure.

Figure 15: Adapted open thermo-hydraulic press system

Source: own authorship

The densification process was carried out with control over displacement, following the optimal conditions delineated by Kadivar, 2020. In this context, the compression of strips further then 50% of their initial thickness induced a partial lateral spread of the tissue, thereby resulting in lateral expansion. The pressure was applied at a compression rate of 6.7 mm/min until the samples achieved a densification degree of 50% avoiding lateral expansion. At this point, average compression stresses of 17 MPa for the reference, 10.31 MPa for the samples treated with alkali solution at room temperature, and 6.95 MPa for the boiled alkali solution treated samples were employed. The temperature of the plates was set at 160°C (C. Gauss, Kadivar, Harries, et al., 2021). A stress-strain plot was generated until the densification degree reached 50%. Once the desired densification degree was attained, the equipment was configured to cease applying load and maintain the displacement.

Upon the removal of the applied load and the establishment of a constant displacement, the internal stress began to decrease until it reached a stable value. This period, referred to as the relaxation time, signifies the duration for the internal stress to stabilize. Data related to the applied load was collected over a 10-min period to analyze the relaxation behavior and generate the corresponding relaxation curves.

3.6 Physical properties

Samples sized (21 × 21 × t) mm were prepared and subjected to a 48-hour drying period in an oven at (100 ± 5)°C until reaching a constant weight to acquire the dry mass. For water absorption, and thickness swelling assessments, the dimensions and weight of bamboo samples were measured after oven drying and following immersion in water for 1 h and 24 h. A digital caliper (± 0.01 mm precision) and a digital scale (± 0.5 g precision) were employed for accurate measurements.

Apparent density values were calculated to the recommendations of ASTM Standard D2395-17 (ASTM D2395-17, 2017) following equation 2, after drying at (100 ± 5) ºC in the oven and after 1 h and 24 h exposed to the environment with 25 ± 2 ºC and 65 ± 3% relative humidity.

$$\rho = \frac{m}{v} \tag{2}$$

Thickness swelling (TS) and water absorption (Wa) values, post 1-hour and 24-hour water immersion, were determined relative to the weight and dimensions of the samples after oven drying at (100 ± 5)°C following equations 3 and 4.

$$Wa = (m1-m0)/m0 \ \text{x} \ 100\% \tag{3}$$

$$TS= (t1-t0)/t0 \ x\ 100\% \qquad (4)$$

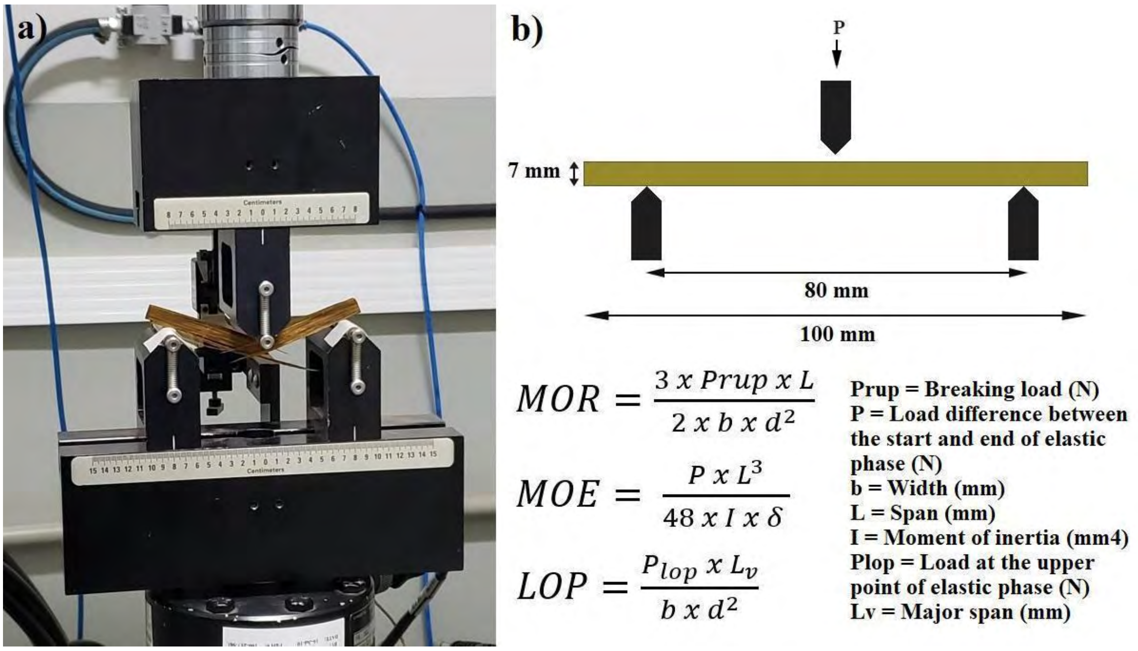
Here, "m0" and "m1" represent the masses before and after the immersion of the samples in water. Additionally, "t0" and "t1" denote the thicknesses before and after the immersion, respectively.

For statistical comparisons between groups, one-way ANOVA and post-hoc Tukey tests were conducted for each physical property. All comparisons were conducted within the same condition, whether oven-dried, after 1 h, or 24 h of water immersion.

3.7 Mechanical characterization

Samples, with an average size of 7 mm thickness, 8 mm width, and 100 mm length post-densification, were prepared for three-point bending tests with a span of 80 mm, following the ASTM D790 test method recommendations (ASTM D790-17, 2017). The testing apparatus employed was a mechanical servo-hydraulic testing machine (MTS Model 370.02, Eden Prairie, MN with a 1kn load cell), as illustrated in Figure 16a.

Figure 16: Bending test (a) equipment (b) scheme and formulations.



$$MOR = \frac{3\ x\ Prup\ x\ L}{2\ x\ b\ x\ d^2}$$

$$MOE = \frac{P\ x\ L^3}{48\ x\ I\ x\ \delta}$$

$$LOP = \frac{P_{lop}\ x\ L_v}{b\ x\ d^2}$$

Prup = Breaking load (N)
P = Load difference between the start and end of elastic phase (N)
b = Width (mm)
L = Span (mm)
I = Moment of inertia (mm4)
Plop = Load at the upper point of elastic phase (N)
Lv = Major span (mm)

Source: Own authorship

The Modulus of Rupture (MOR), Modulus of Elasticity (MOE), and Limit of Proportionality (LOP) were calculated using the equations provided in Figure 16b. Additionally, the Specific Energy (EE) was determined as the area under the stress-strain curve divided by the cross-section of the samples.

To assess statistical differences between groups for each mechanical property, one-way ANOVA and post-hoc Tukey tests were applied. These statistical analyses provide a comprehensive understanding of the variations and significance among different groups in terms of MOR, MOE, LOP, and Specific Energy.

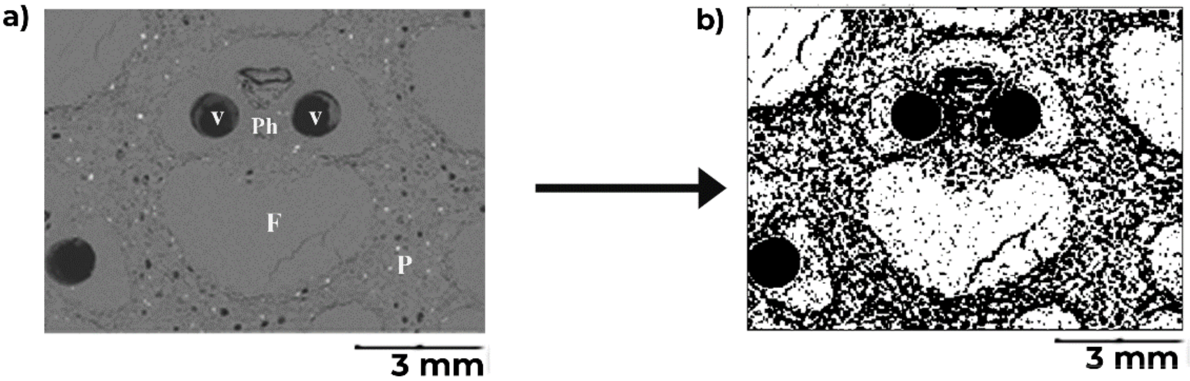
3.8 Image analysis

A Hitachi TM-3000 Scanning Electron Microscope (SEM) with an accelerating voltage of 15 kV and a backscattered electron detector was employed to capture images of the cross-sectional area of the samples.

For sample preparation, the Struers Tegra Pol-11 machine facilitated sanding and polishing. To achieve a highly reflective surface, samples underwent polishing involving different grit sizes of paper (ranging from P320 to P2000) with water. The polishing process was completed using polycrystalline diamond suspension solutions.

ImageJ software was utilized to calculate the fiber volume fraction of bamboo. Initially, images of the samples were collected, focusing on the usable area; Figure 17a provides a representation of the components in the structure. Subsequently, the image was transformed into an 8-bit (grayscale) image to isolate the fiber portion from the holes and parenchyma, utilizing the software command "limit"; Figure 17b represents the same structure on ImageJ. The software automatically identifies the darkest areas, corresponding to parenchyma and empty spaces.

Figure 17: Representation of ImageJ analysis (a) reference photo (b) grayscale image with fiber bundles isolated.



V=Vessels; Ph=Phloem; F=Fiber bundles; P=Parenchyma.

Source: Own authorship

3.9 Chemical and thermal analysis

Fourier Transform Infrared Spectroscopy (FT-IR) analysis was conducted on densified groups after the delignification and drying processes. Bamboo particles were obtained using the analytical mill Quimis - Q298A. Subsequently, a PerkinElmer Spectrum One spectrometer with an ATR (Attenuated Total Reflectance) accessory was employed to perform scans across the range of 4000 to 600 cm^{-1}, with a resolution of 4 cm^{-1}. The FT-IR analysis aimed to provide information about the chemical composition and structural changes of the bamboo material following the delignification and densification procedures.

For the Thermogravimetric Analysis (TGA), both natural and treated densified bamboo samples were utilized for simultaneous TG-DSC (Thermogravimetric Differential Scanning Calorimetry) measurements. The analysis was carried out using a NETZSCH STA 449F3 apparatus, operating within a temperature range from 25 to 600 °C under a nitrogen environment, purged at a rate of 40 mL min^{-1}. The TGA measured the weight loss of the samples over the specified temperature range, providing information about their thermal stability and decomposition characteristics. A constant heating rate of 10 °C min^{-1} was maintained throughout the analysis.

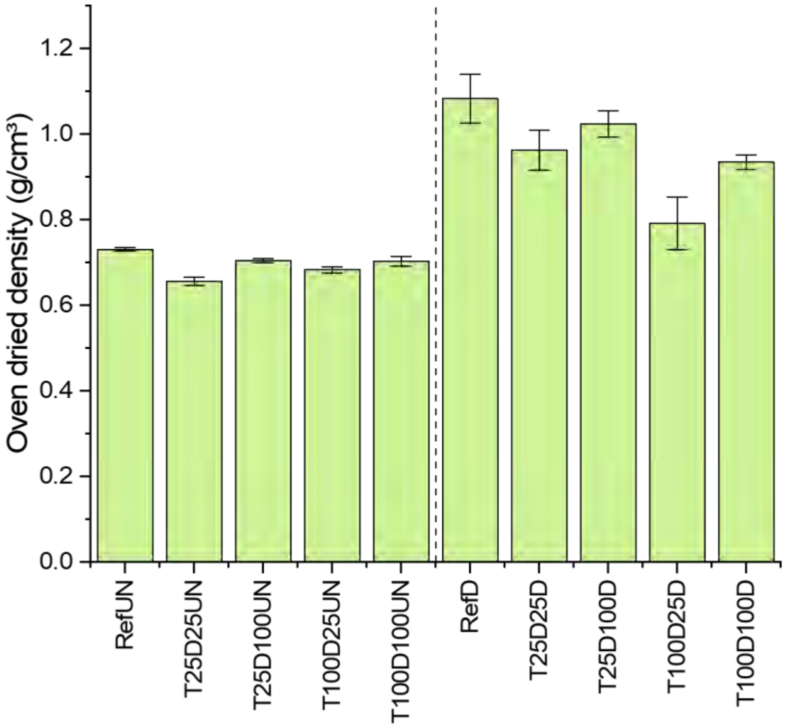
4 Results and discussions

This section is dedicated to presenting the results of the treated samples, analyzing their physical, mechanical, chemical, and thermal properties. The aim is to offer an analysis of how the treatment process has influenced and modified the characteristics of the samples. Through an examination of these properties, it is possible to analyze the overall impact of the treatment on the material's performance and structural attributes.

4.1 Physical properties

The statistical analysis, along with apparent density values for oven-dried, 1-hour, and 24-hour in the environment, is provided in Table 5. The apparent density values for all groups under oven drying conditions are illustrated in Figure 18, which corresponds to the first column of table 5. Notwithstanding the similarity in the densification process during delignification with the optimal parameters from Kadivar 2020, variations in density are attributed to the removal of lignin from the bamboo structure through the action of the alkaline solution and the drying process, a topic that will be further discussed in the chemical analysis section. All samples, under controlled conditions, achieved a densification degree of 50%. It is noteworthy that the drying process at room temperature leads to a prolonged duration for effective accommodation of the material. Conversely, samples subjected to rapid drying lack the necessary time for the material to achieve its equilibrium condition, as the accelerated drying presents insufficient time for the material to organize itself (further discussions in section 4.3). All treated samples exhibited lower density after densification compared to the reference samples due to the extraction of lignin and drying process, with no statistical difference observed for the non-densified condition. Therefore, it can be stated that the samples could undergo a higher degree of densification to achieve the density of the reference samples. Analyzing Figure 18, for densified samples, it is evident that samples treated with boiling alkaline solution exhibit lower densities than those treated at room temperature (T100D25 and T100D100). Additionally, samples dried at room temperature also show lower values compared to those dried at 100ºC (T25D25 and T100D25). Thus, the T100D25 group presents the lowest density values for a densification degree of 50%, indicating that this group could be densified beyond all other groups to reach the density of the reference group. In section 4.5 is also discussed a higher lignin removal when treating the samples with boiling solution, leading to smaller density values. It is also possible to analyze a higher standard deviation after the densification process. This observation could be linked to an open thermo-hydraulic system that doesn't impose movement restrictions in the horizontal direction during the pressing process. Even for undensified samples, the groups dried in room temperature present lower density values. In table 4 it is possible to see the difference in density after 24 hours due to moisture absorption, with the reference densified group having a constant density mean value presenting a low moisture absorption during the first 24 hours.

Figure 18: Bamboo oven dried density values before and after treatment.



Source: Own authorship

Table 4: Density of dry samples and after 1 and 24 hours.

Density (g/cm³) - Mean (SD)			
Undensified groups			
Treatment	Oven dried	1 hour	24 hours
RefUN	0.731 a (0.004)	0.773 a (0.021)	0.907 a (0.005)
T25D25UN	0.656 a (0.010)	0.714 a (0.008)	0.897 a (0.012)
T25D100UN	0.704 a (0.005)	0.747 a (0.020)	0.945 a (0.014)
T100D25UN	0.683 a (0.007)	0.792 a (0.0007)	0.946 a (0.024)
T100D100UN	0.703 a (0.011)	0.788 a (0.022)	0.969 a (0.015)
Densified groups			

Treatment	Oven dried	1 hour	24 hours
RefD	1.083 a (0.057)	1.078 a (0.022)	1.076 a (0.042)
T25D25D	0.963 b, c (0.047)	1.009 a (0.039)	1.067 a (0.022)
T25D100D	1.024 a, c (0.031)	1.039 a (0.049)	1.289 a (0.421)
T100D25D	0.791 d (0.062)	0.740 b, c (0.333)	1.017 a (0.055)
T100D100D	0.934 b (0.017)	1.001 a, c (0.013)	1.083 a (0.026)

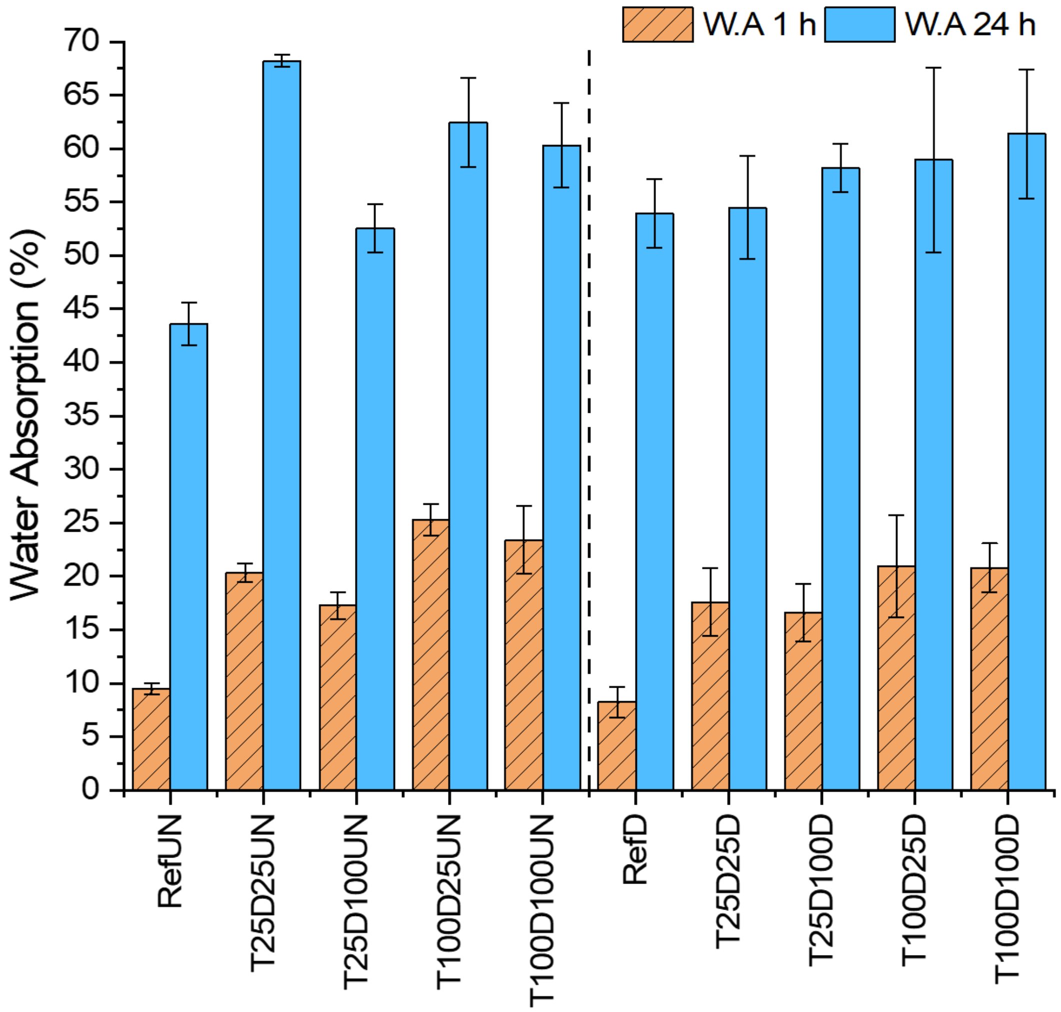
a, b, c letters indicate significant statistical differences between values in the same column (Tukey statistical test method).

In terms of water absorption (Figure 19 and Table 5), undensified reference samples exhibited lower absorption, both after 1 hour and 24 hours, compared to all treated groups. This phenomenon may be attributed to the damage induced by alkaline treatment and the incomplete densification to a densification degree of 50%, resulting in increased gaps in the bamboo structure. The higher water absorption of the sclerenchyma compared to the parenchyma suggests that the removal of lignin does not significantly affect this parameter. In contrast to the natural bamboo results, the water absorption exhibited no distinct trend after a 24-hour immersion period. The densification process generally increased bamboo water absorption, although this trend was not confirmed for a few treated groups, namely T25D25 and T100D25 in which the densified showed less water absorption for both average values of 1 hour and 24 hours.

Densified reference samples displayed an expected higher water absorption compared to non-densified samples. However, the values for 1-hour immersion of densified samples were lower than those observed for both treated and untreated for all non-densified samples (Figure 19). Although without statistical difference for the densified samples after 24 hours of immersion, reference samples presented lower mean values compared to treated ones for both densified and undensified. This effect might be associated with the delignification process caused by the alkaline treatment, resulting in more gaps in the bamboo structure.

Additionally, since sclerenchyma has higher water absorption than parenchyma, the removal of lignin may not significantly influence this parameter, being the higher temperature for both treatment or drying and the pressure applied during densification responsible for creating more gaps in the microstructure resulting in a higher water absorption for densified samples (Yuan et al., 2021).

Figure 19: Bamboo water absorption values before and after treatment.



Source: Own authorship

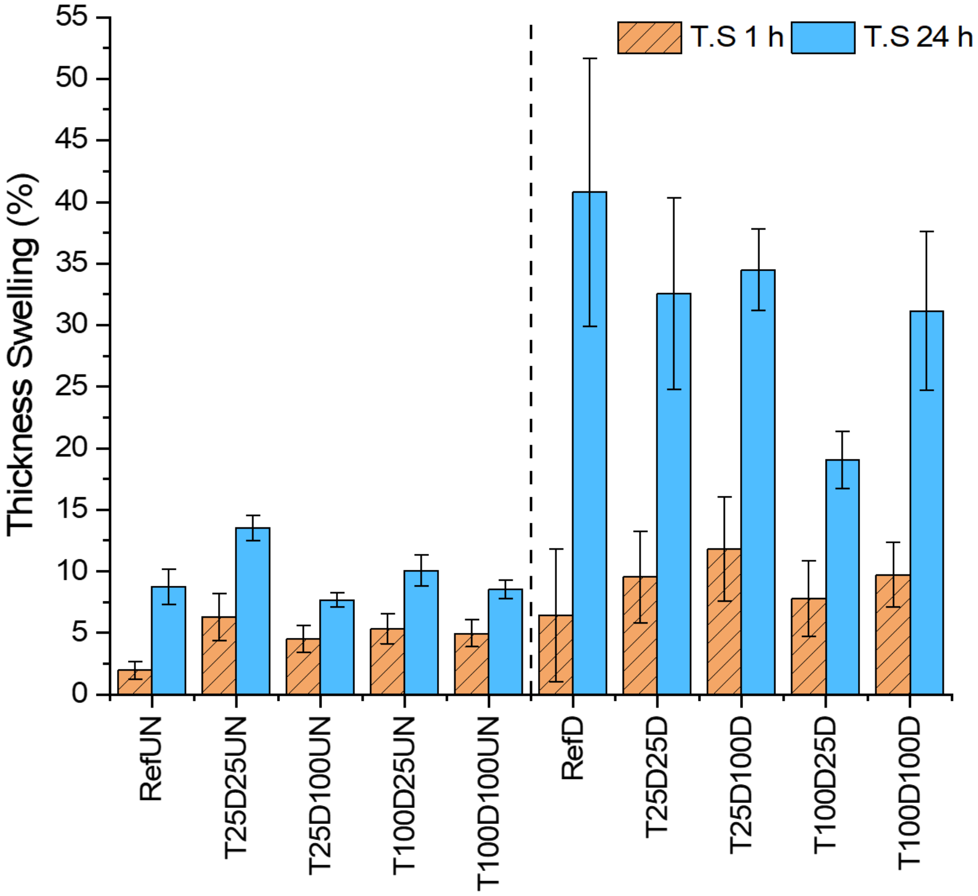
Table 5: Water absorption of bamboo samples according to treatment and time in water.

Water Abs (%) - Mean (SD)		
Undensified groups		
Treatment	1 hour	24 hours
RefUN	9.51 a (0.50)	43.63 a (2.00)
T25D25UN	20.36 b, c (0.88)	68.21 b (0.59)
T25D100UN	17.29 b (1.25)	52.56 a, c (2.22)
T100D25UN	25.27 c (1.48)	62.46 b, c (4.16)
T100D100UN	23.40 b, c (3.18)	60.32 b, c (3.94)
Densified groups		
Treatment	1 hour	24 hours
RefD	8.23 a (1.41)	53.92 a (3.20)
T25D25D	17.59 b (3.16)	54.51 a (4.84)
T25D100D	16.59 b (2.69)	58.21 a (2.25)
T100D25D	20.93 b (4.79)	58.95 a (8.65)
T100D100D	20.80 b (2.29)	61.40 a (6.03)

a, b, c letters indicate significant statistical differences between values in the same column (Tukey).

The results of thickness swelling indicate that densified reference samples demonstrate lower dimensional stability compared to the treated ones after 24 hours although having the lowest mean values for 1 hour immersion. It is possible to note a relationship between density and thickness swelling in densified samples. The higher the density, and consequently the effectiveness of the densification process with fixed displacement, the greater the thickness swelling (Figure 20 and Table 6). Notably, a statistically significant difference is observed only within Samples treated in a boiling alkaline solution and dried at room temperature (T100D25D) group that have the lowest density values, also exhibiting significantly higher stability compared to other treatment conditions and the reference. On the other hand, for non-densified ones, bamboo samples with pretreatment demonstrate similar stability between each other but is possible to note that lower density values correspond to higher values of thickness swelling due to extraction of lignin and hemicellulose (T25D25UN and T100D25UN). The difference between the increase in thickness between densified and undensified samples is very visible, being one of the biggest problems of the densification process

Figure 20: Bamboo thickness swelling values before and after treatment.



Source: Own authorship

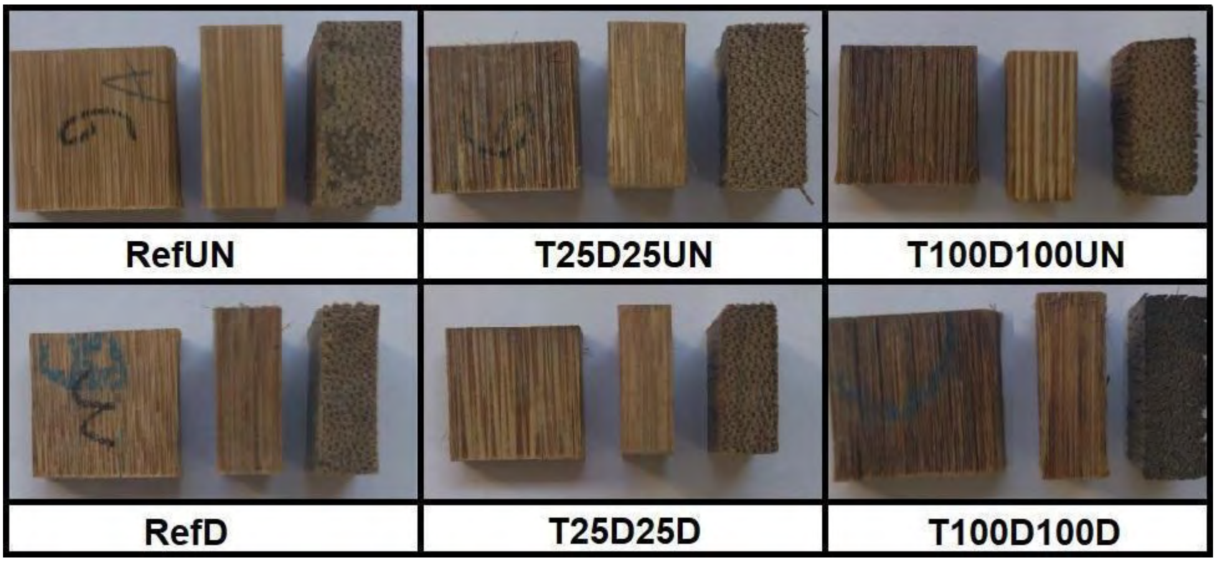
Table 6: Thickness swelling of bamboo samples according to treatment and time in water.

Thickness Swelling (%) - Mean (SD)		
Undensified groups		
Treatment	1 hour	24 hours
RefUN	1.98 a (0.71)	8.75 a (1.43)
T25D25UN	6.29 a (1.90)	13.53 a (1.02)
T25D100UN	4.52 a (1.09)	7.68 a (0.58)
T100D25UN	5.34 a (1.21)	10.08 a (1.27)
T100D100UN	4.97 a (1.10)	8.55 a (0.76)
Densified groups		
Treatment	1 hour	24 hours
RefD	6.46 a (5.39)	40.79 a (10.85)
T25D25D	9.56 a (3.72)	32.55 a (7.79)
T25D100D	11.81 a (4.24)	34.50 a (3.33)
T100D25D	7.81 a (3.08)	19.05 b, c (2.33)
T100D100D	9.74 a (2.63)	31.15 a, c (6.44)

a, b, c letters indicate significant statistical differences between values in the same column (Tukey).

Figure 21 displays the test bodies of six sample groups after physical tests and a 24-hour water immersion. Notably, both undensified and densified groups showed that the chemical treatment led to a slight defibrillation, which involves the separation or disintegration of cellulose fibers into finer fibrils within the bamboo structure after immersion in water.

Figure 21: Samples after physical tests.



Source: Own authorship

4.2 Mechanical characterization

Table 7 displays the modulus of rupture (MOR), limit of proportionality (LOP), modulus of elasticity (MOE), and specific energy (EE) values obtained from the three-point bending test conducted on both densified reference and treated samples.

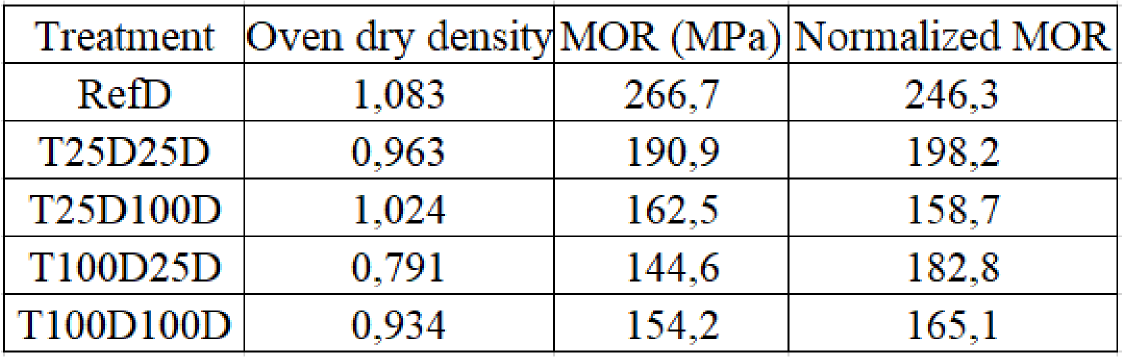
Table 7: Mechanical properties of densified bamboo with different pretreatments.

Treatment	MOR (MPa)	LOP (MPa)	MOE (GPa)	EE (kJ/m²)
RefD	266.7 a (27.8)	174.2 a (23.5)	19.2 a (2.6)	19.5 a (10.5)
T25D25D	190.9 b (13.9)	118.3 b (9.71)	14.7 b (1.3)	20.4 a (3.3)
T25D100D	162.5 c (19.2)	118.7 b (17.8)	15.8 b (2.6)	20.2 a (5.9)
T100D25D	144.6 c (18.3)	90.9 c (10.9)	12.9 b (3.2)	14.1 a (6.9)
T100D100D	154.2 c (20.0)	102.1 b, c (23.2)	14.6 b (2.3)	13.0 a (0.8)

a, b, c letters indicate significant statistical differences between values in the same column (Tukey).

The results suggest that densified reference samples exhibit superior mechanical properties compared to all treated groups because they achieved a higher level of densification. This observation can be attributed to the extraction of lignin resulting from the alkali effect during the pretreatment in which a higher densification degree is needed to achieve the density of the reference group and consequently achieving higher mechanical properties. While lignin constitutes a relatively weaker component in the bamboo structure when juxtaposed with cellulose, its removal contributes to the deterioration of the interface between the matrix and bamboo fibers, consequently diminishing the overall strength of the composite without further densification. Analysis of average fiber fraction images (refer to section 4.4) reveals a decrease in the fiber content of treated and densified samples, indicating the removal of cellulose post-treatment and a higher amount of cracks. This emphasizes the rationale behind the detrimental effects of the delignification process on the mechanical properties of the material. Conversely, alkali-treated samples exhibited lower density (ranging from 0.79 to 1.02 g/cm³) compared to the reference group (1.08 g/cm³). For a fair comparison, if the modulus of rupture (MOR) of the RefD and T100D25 groups (with higher and lower densities) were normalized based on their density values, their flexural resistances would be approximately 246 and 182 MPa, respectively (table 8). While the mechanical resistance for treated samples would still be lower, the difference between treated and untreated ones would decrease.

Table 8: Normalized values for modulus of rupture for 1 g/cm³.

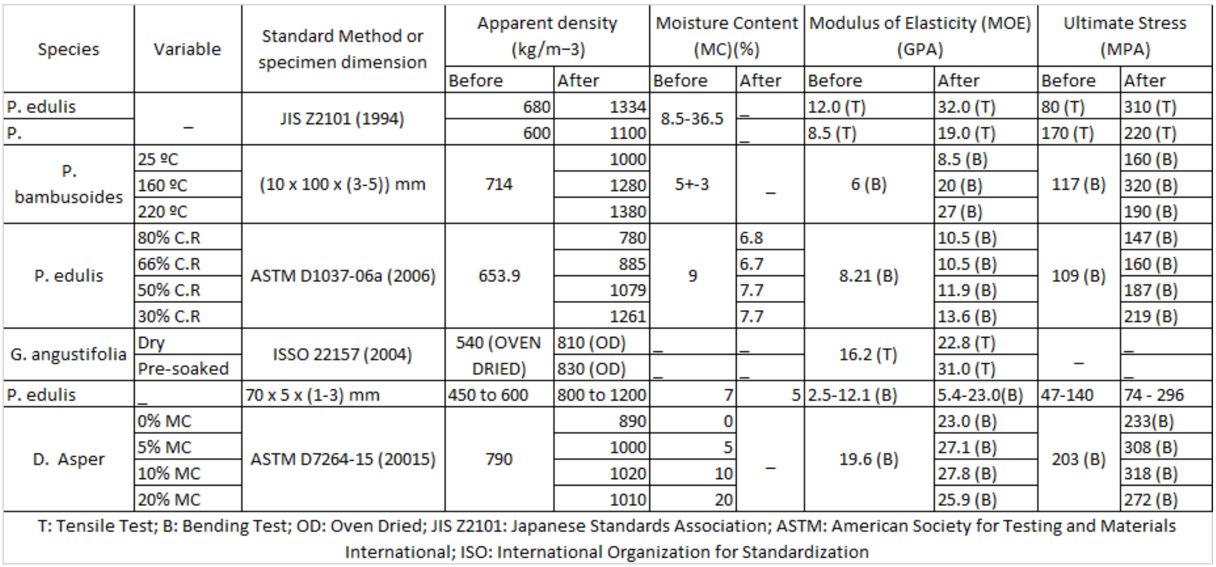


Treatment	Oven dry density	MOR (MPa)	Normalized MOR
RefD	1,083	266,7	246,3
T25D25D	0,963	190,9	198,2
T25D100D	1,024	162,5	158,7
T100D25D	0,791	144,6	182,8
T100D100D	0,934	154,2	165,1

Source: Own authorship.

The modulus of rupture (MOR) and modulus of elasticity (MOE) values for untreated densified and undensified *Dendrocalamus asper* bamboo species, where Marzieh Kadivar gathered in her work entitled “Densification of Bamboo: State of the art” (Kadivar, 2020) a summary of the mechanical and physical properties of bamboo being shown in table 9, ranges from 233 - 318 MPa and 23 - 28 GPA for densified and 203 MPa and 19.6 GPa depending on the initial moisture content, respectively for *Dendrocalamus asper*. In contrast, the results obtained in this study indicate an enhancement in MOR following densification, while chemically treated groups exhibit a decrease in both modulus of rupture and elastic modulus until 50% of densification degree.

Table 9: summary of the mechanical and physical properties of bamboo before and after densification process.



Species	Variable	Standard Method or specimen dimension	Apparent density (kg/m−3)		Moisture Content (MC)(%)		Modulus of Elasticity (MOE) (GPA)		Ultimate Stress (MPA)	
			Before	After	Before	After	Before	After	Before	After
P. edulis	–	JIS Z2101 (1994)	680	1334	8.5-36.5	_	12.0 (T)	32.0 (T)	80 (T)	310 (T)
P.			600	1100		_	8.5 (T)	19.0 (T)	170 (T)	220 (T)
P. bambusoides	25 ºC	(10 x 100 x (3-5)) mm	714	1000	5+-3	–	6 (B)	8.5 (B)	117 (B)	160 (B)
	160 ºC			1280				20 (B)		320 (B)
	220 ºC			1380				27 (B)		190 (B)
P. edulis	80% C.R	ASTM D1037-06a (2006)	653.9	780	9	6.8	8.21 (B)	10.5 (B)	109 (B)	147 (B)
	66% C.R			885		6.7		10.5 (B)		160 (B)
	50% C.R			1079		7.7		11.9 (B)		187 (B)
	30% C.R			1261		7.7		13.6 (B)		219 (B)
G. angustifolia	Dry	ISSO 22157 (2004)	540 (OVEN DRIED)	810 (OD)	_	_	16.2 (T)	22.8 (T)	–	_
	Pre-soaked			830 (OD)	_	_		31.0 (T)		_
P. edulis	_	70 x 5 x (1-3) mm	450 to 600	800 to 1200	7	5	2.5-12.1 (B)	5.4-23.0(B)	47-140	74 - 296
D. Asper	0% MC	ASTM D7264-15 (20015)	790	890	0	–	19.6 (B)	23.0 (B)	203 (B)	233(B)
	5% MC			1000	5			27.1 (B)		308 (B)
	10% MC			1020	10			27.8 (B)		318 (B)
	20% MC			1010	20			25.9 (B)		272 (B)

T: Tensile Test; B: Bending Test; OD: Oven Dried; JIS Z2101: Japanese Standards Association; ASTM: American Society for Testing and Materials International; ISO: International Organization for Standardization

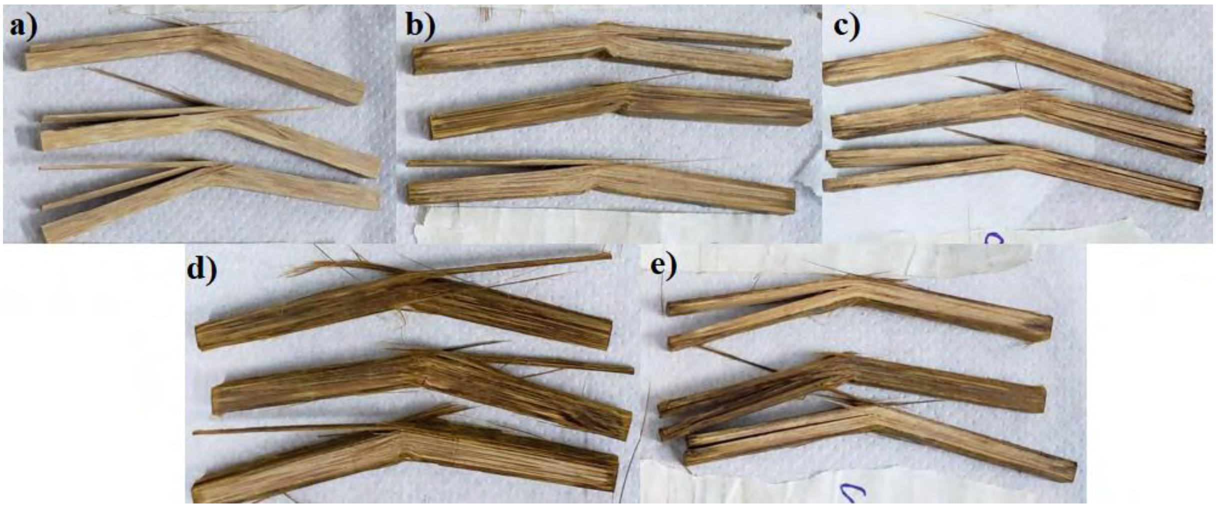
Source: Adapted from Kadivar, 2020.

These findings diverge from the outcomes reported for *Phyllostachys bambusoides* by Li et al. (2020), where pretreated and densified samples demonstrated an improvement in flexural strength. It is essential to note that the disparities in immersion time in alkaline solution, densification time, and densification degree between this study and the referenced work contribute to these opposing trends.

The failure patterns for all experimental groups are illustrated in Figure 22. A limited number of individual fibers were observed, attributable to the tension fibers functioning as bundles during crack propagation. The rupture pattern is attributed to the splintering of fibers in tension, and this effect is more prominent in the treated groups. The increased splintering is a result of lower adhesion between fiber bundles and parenchyma tissue. Some samples even exhibit shear failure, particularly the middle and bottom samples from Figure 22. In the compression area, the bending action led to crack propagation in the compacted fibers, resulting in fiber breakage.

In terms of crack propagation, treated samples demonstrated a slower rate compared to the reference group. Notably, among the treated groups, those exposed to a boiling solution exhibited more plastic deformation. This suggests that the treatment either softened the bamboo tissue or the lower density of the alkali-treated samples influenced the deformation.

Figure 22: Failure patterns a) RefD b) T25D25D c) T25D100D d) T100D25D e) T100D100D.

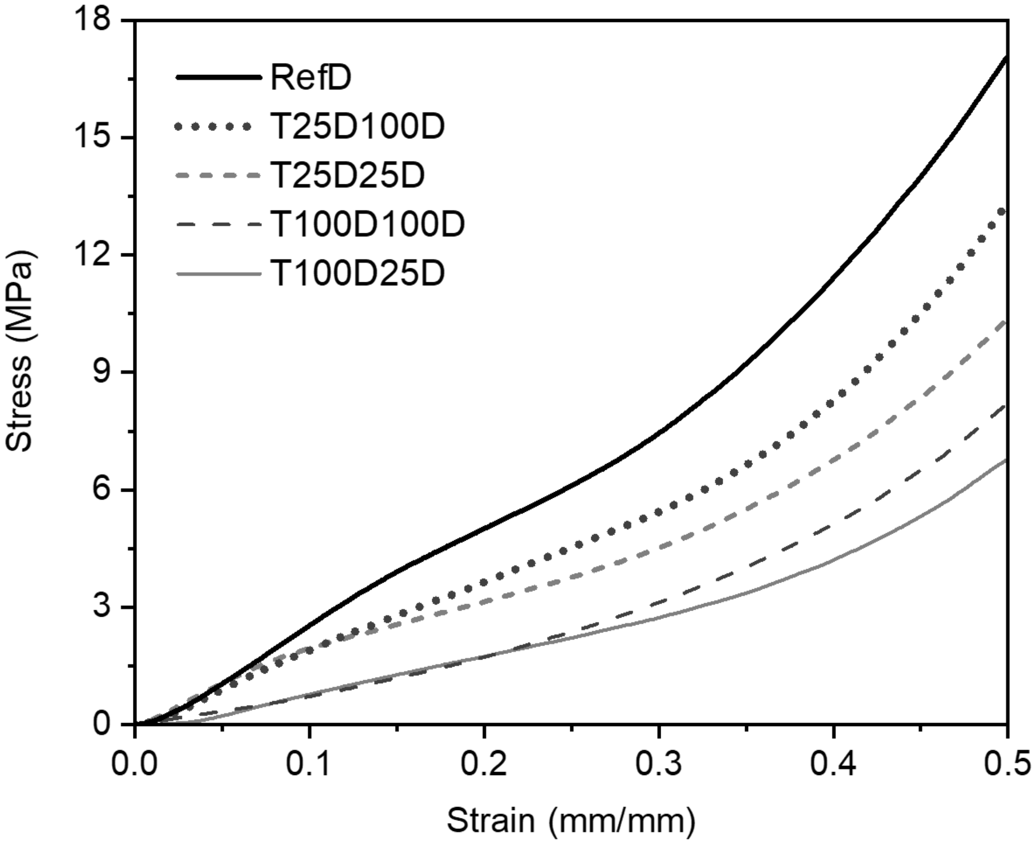


Source: Own authorship

4.3 Effects of pretreatment on densification process

The applied load during the bamboo densification process, up to a 50% reduction in thickness, was recorded and depicted in Figure 23. Alkali-treated bamboo exhibited a lower load requirement for achieving the desired densification, ranging from 7 to 13.4 MPa, as opposed to 17 MPa for the densification of untreated bamboo. This suggests that the removal of lignin and hemicellulose from the bamboo matrix eases compression and reduces energy consumption compared to non-treated bamboo.

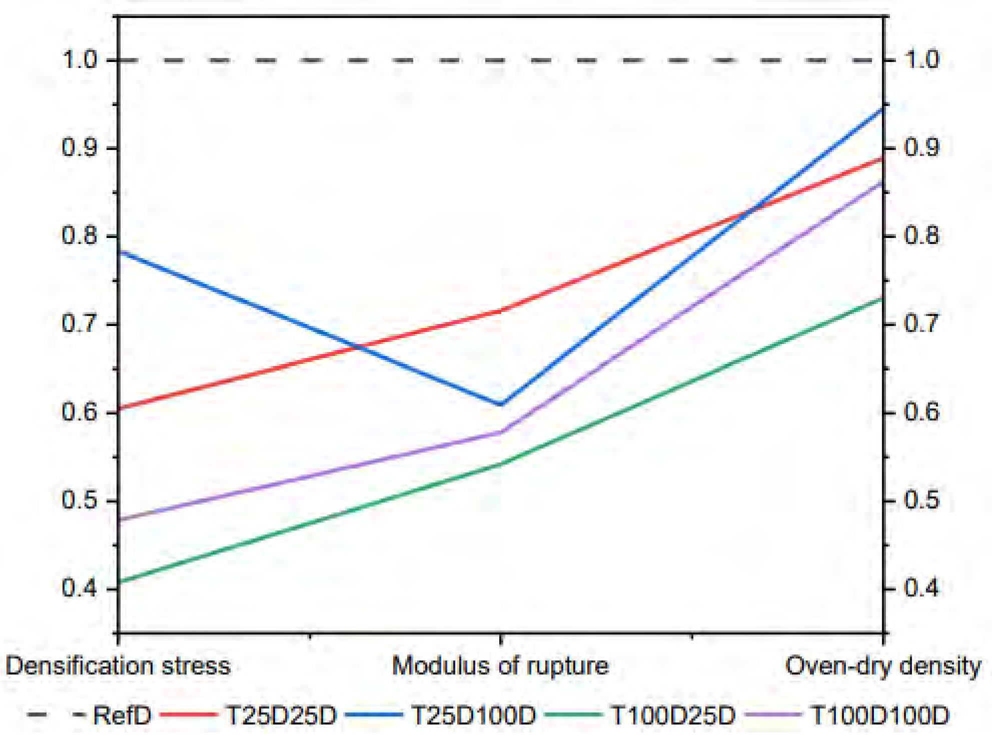
Figure 23: Stress-strain plot in the densification process.



Source: Own authorship

Notably, the reference group (RefD), which necessitated a higher compression stress (17 MPa), demonstrated a higher oven-dry density (1.08 g/cm³) and a greater modulus of rupture (266.7 MPa). In contrast, the T100D25 group, requiring a lower compression stress (7 MPa), exhibited a lower oven-dry density (0.79 g/cm³) and the lowest resistance in bending (144.6 MPa). This highlights the relationship between compression stress, density, and mechanical properties in the densification of bamboo. This implies that alkali treatment could enable a greater degree of densification with consistent energy consumption. Such an outcome has the potential to improve the material's mechanical properties and boost its density, given the observed correlation among these three properties— modulus of rupture (MOR), densification stress, and density (Figure 24).

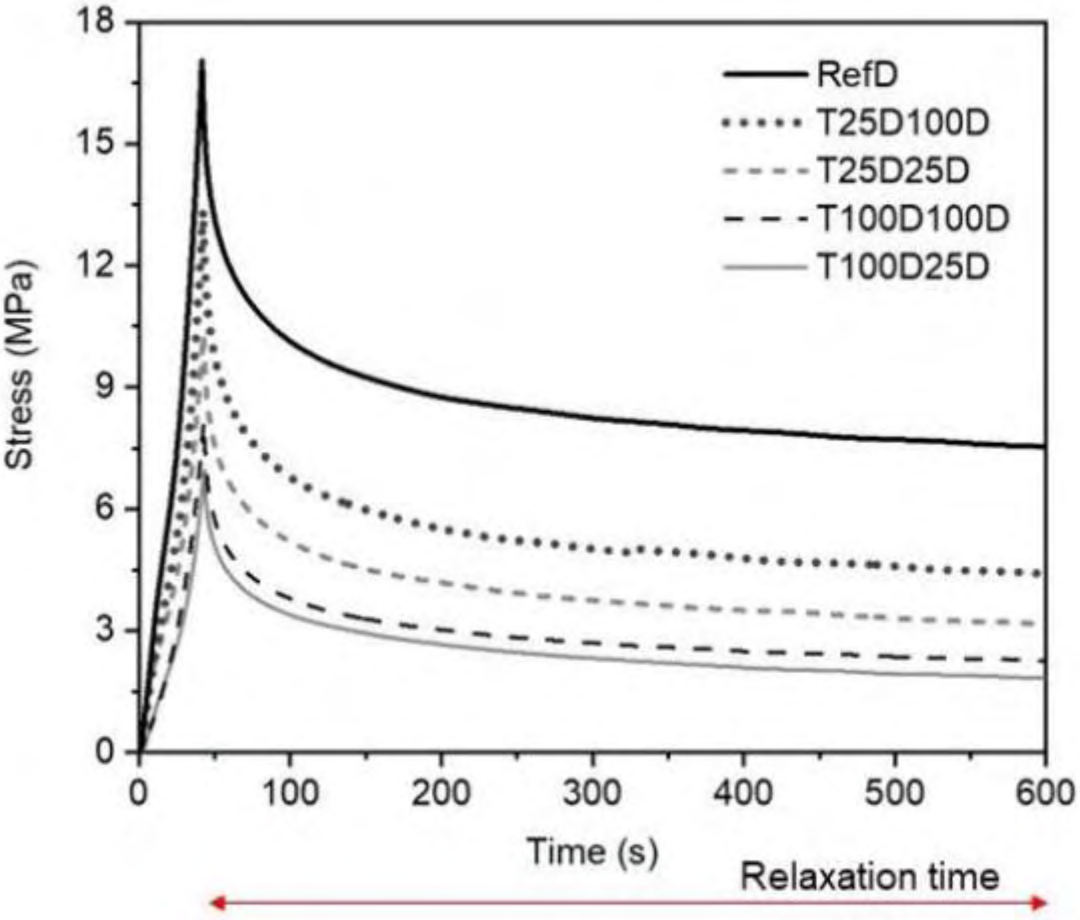
Figure 24: Correlation between normalized densification stress, modulus of rupture and density.



Source: own authorship

Figure 25 illustrates both the applied stress and the internal stress relaxation within the bamboo matrix during the densification process. It is evident that the Thermo-Hydro-Mechanical (THM) process serves as an intensive method for modifying bamboo, inducing internal stresses due to its structural transformation. This phenomenon is consistent across all samples. The relaxation process mitigated a portion of the stresses, displaying a rapid elimination rate within the initial 50 seconds for all samples before stabilizing toward distinct residual values. After 550 seconds, the residual stress for the reference group was approximately 8 MPa, and this value decreased with the application of pretreatments. Notably, the group of densified bamboo treated in boiling NaOH+NaSO3 solution and dried at room temperature approached a residual value of approximately 2 MPa.

Figure 25: Relaxation curve plot in densification process.



Source: Own authorship

This result suggests that the treated samples in the boiling solution undergo a more rapid accommodation of their structure after the densification process compared to the other groups, as can be seen in Figure 25, leading to a reduction in internal stresses, with the lower amount of residual stress ranging from 2 to 3 MPa.

4.4 Image analysis

The image analysis conducted through ImageJ software (refer to Table 10) revealed that the average fiber fraction for undensified groups increased after alkali treatment, with higher fractions observed in room temperature solutions. This aligns with expectations of lignin removal from the bamboo structure. However, for densified samples, treated groups exhibited lower values compared to the reference group. Notably, the volume of fiber fraction in the T100D100D group is similar to the undensified reference group, indicating a reduction of approximately 10%. This may be connected to a slight reduction in cellulose content, which becomes noticeable post-densification. However, it could result from the limited area sampled during SEM analysis of the fiber fraction, which may not accurately represent the entire cross-section. Consequently, despite the effectiveness of the pretreatment in facilitating the densification process, it adversely impacts bamboo by compromising its fiber content.

The mechanical results, as discussed in section 4.2, revealed a detrimental effect of chemical pretreatment on the mechanical resistance of densified samples. This negative impact is linked to a weaker interface between parenchyma and sclerenchyma, as well as a reduction in fiber content and the appearance of internal cracks due to the pressure applied during the densification process. The subsequent thermal analysis, which will be elaborated upon in the next section, further supports these findings by indicating a loss of cellulose following treatment and densification. This aligns with the observed trends in fiber fraction volume and mechanical properties. This may be due to the fact that the treatment enables a greater degree of densification, and since the reduction of up to 50% in the original thickness doesn't represent the proper densification degree for the treated groups, it leads to the appearance of several internal cracks, reducing the average fiber fraction in the area analyzed. It is also possible to analyze that samples densified with lower density values (T100D25 and T100D100) presented the lowest values of average fiber fraction and consequently lower mechanical performance, and the increase in internal cracks explain the smaller values compared to their corresponding undensified values.

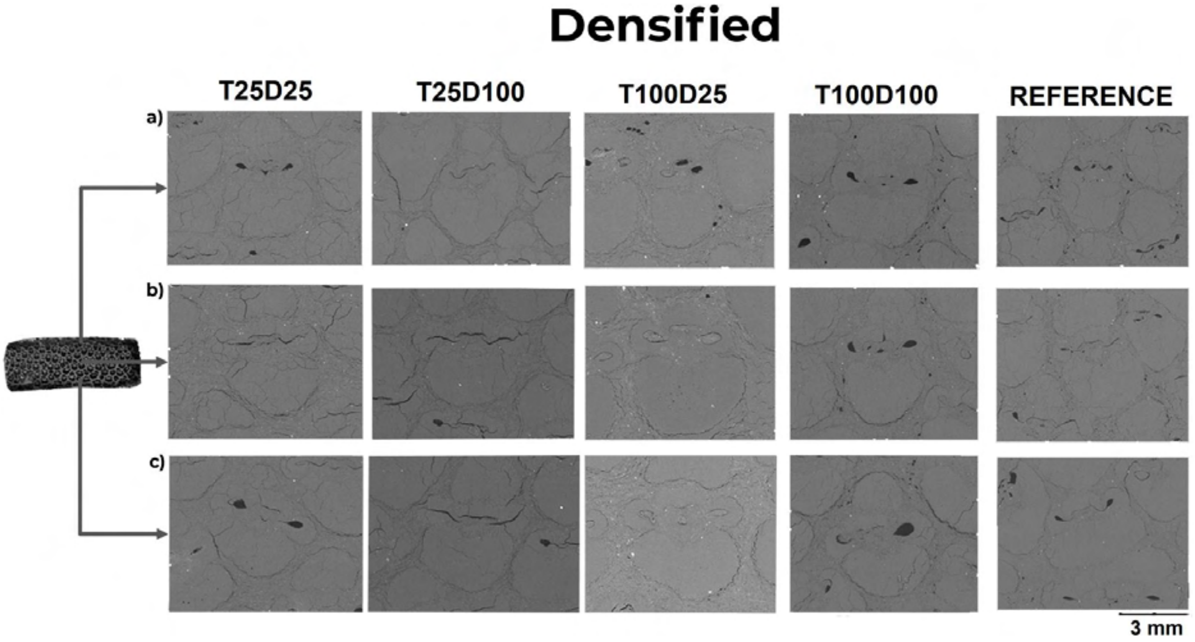
Table 10: Values of average fiber fraction for each group.

Treatment	Average Fiber Fraction
RefUN	52,13%
T25D25UN	65,98%
T25D100UN	63,29%
T100D25UN	60,92%
T100D100UN	59,71%

RefD	66,15%
T25D25D	61,64%
T25D100D	62,45%
T100D25D	54,84%
T100D100D	52,81%

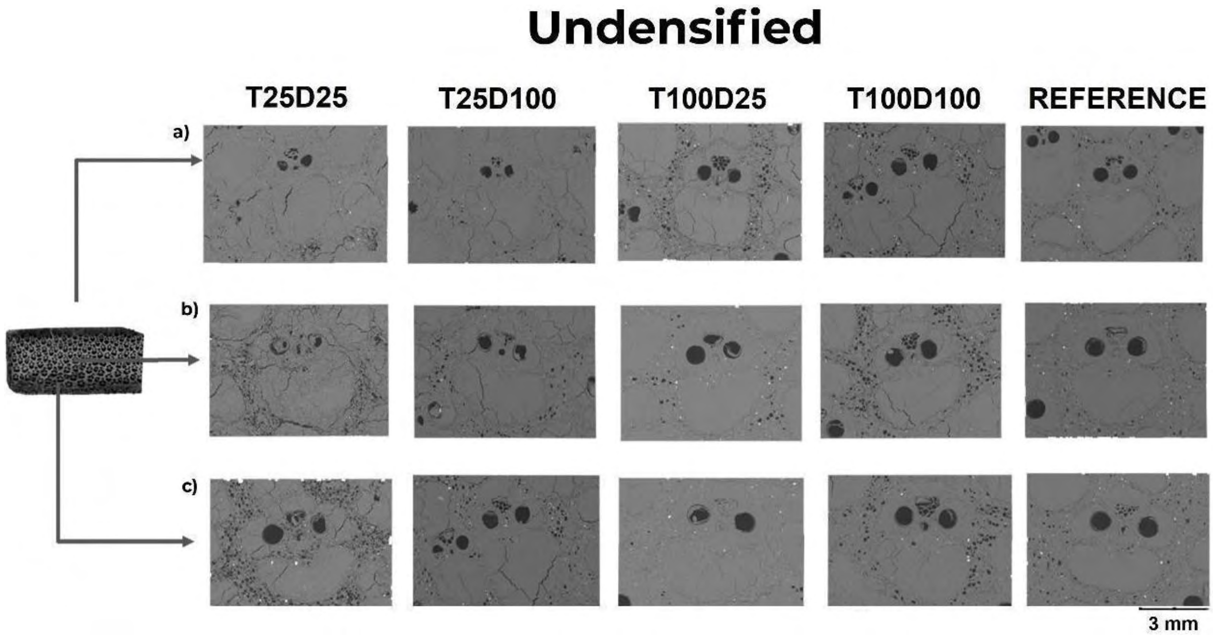
The images captured using the Hitachi TM-3000 Scanning Electron Microscope (SEM) provide information about the characteristics of the densification processes and the influence of the alkaline solution. These images are showcased in Figure 26 and Figure 27 for both densified and undensified samples. Images were taken from the inner, middle, and outer layers of the cross-section, offering a view of the structural changes induced by the densification process and the impact of the alkaline solution.

Figure 26: SEM images of densified bamboo samples. The images were extracted from the outer (a), middle (b) and inner layer (c)



Source: own authorship

Figure 27: SEM images of undensified bamboo samples. The images were extracted from the outer (a), middle (b) and inner layer (c)

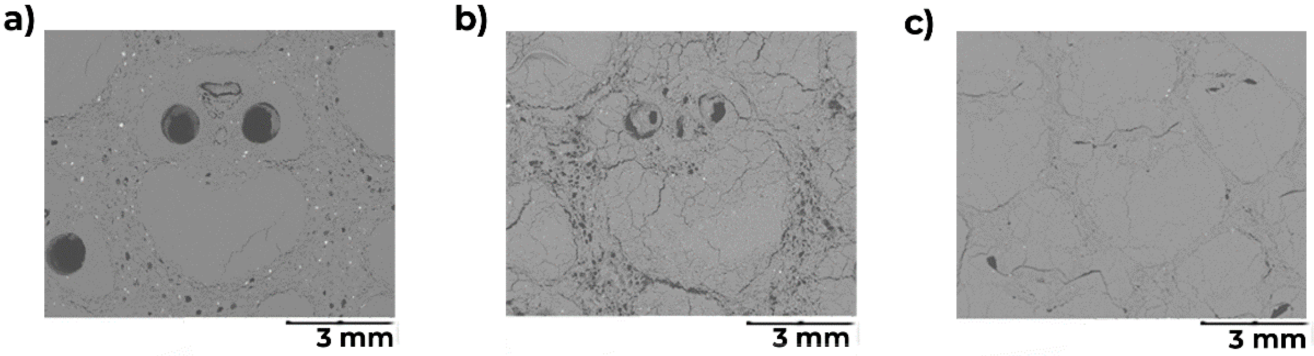


Source: Own authorship.

The SEM images, particularly Figure 28a, reveal the presence of vascular vessels in the undensified groups, highlighting the impact of the alkali treatment on bamboo microstructures. The T25D25 and T25D100 treatment appears to be more effective in closing the vessels until 50% of densification degree in which approaches the density of the reference group. Additionally, compared to the reference, there is a notable increase in the number of microcracks in the bamboo structure, as evident in Figure 28b. This occurrence can be attributed to the extraction of lignin and a higher densification degree to properly close the voids and eliminate the cracks.

In contrast, the samples that underwent densification exhibit closed voids resulting from the densification process, as shown in Figure 28c. The fiber parts, represented by the lighter regions, appear to be in closer proximity to each other. These observations provide visual characteristics of the structural alterations induced by both the alkali treatment and the densification process.

Figure 28: a) reference group without densification b) undensified sample that underwent alkaline treatment at room temperature and dried at room temperature (T25D25UN) c) densified reference group.



Source: Own authorship

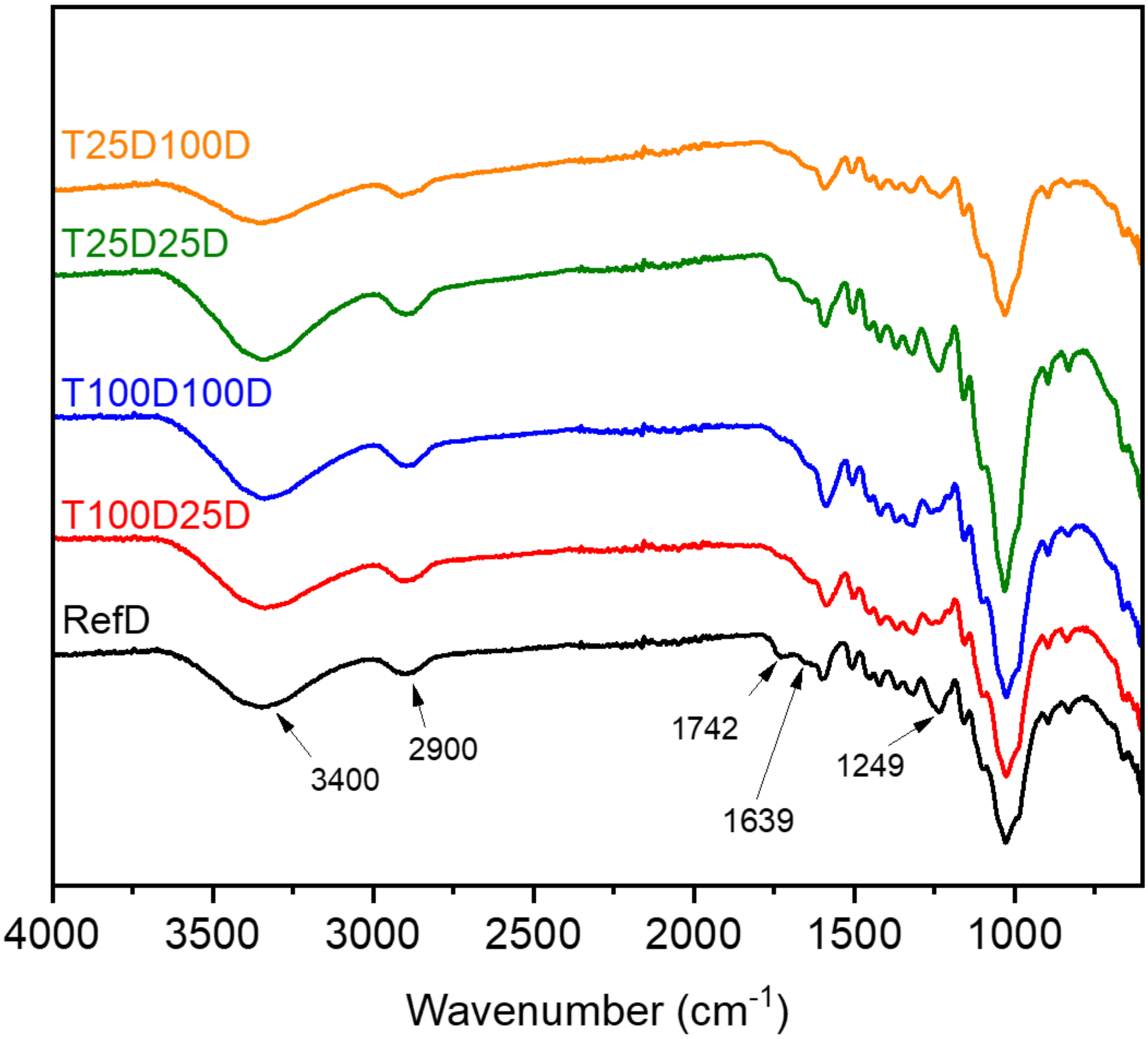
The utilization of a boiling solution increased the formation of cracks and spaces, facilitating the material's ability to accommodate displacement during the densification process. However, this comes at the cost of reduced mechanical properties in densified bamboo. The boiling solution induces the degradation of cellulose and hemicellulose in addition to lignin, resulting in a weakened bond between the matrix and fibers that can be solved in a closed system densification process with higher densification levels.

4.5 Chemical and thermal analysis

FTIR analysis

The FTIR spectra of bamboo is presented in Figure 29.

Figure 29: FT-IR spectra for densified bamboo samples.



Source: Own authorship

The bands around 1742 and 1249 cm-1 exhibited a decrease following alkaline treatment. The band at 1742 cm-1 is associated with the C=O stretching vibration in hemicellulose in bamboo, as reported by Kabir et al. (2013). Post-treatment, this band is no longer discernible in the samples, underscoring the effectiveness of alkaline attack in removing hemicellulose from bamboo. While temperatures exceeding 150 °C can also lead to hemicellulose removal (Pelaez-Samaniego et al., 2013; Kadivar, 2019), the more pronounced decrease in intensity observed in treated samples emphasizes the significant impact of the chemical treatment.

The bands around 1600 and 1505 cm-1 are linked to the stretching of the benzene ring and -OCH3 groups in lignin, as reported by Tserki (2005). The prominent absorption band at approximately 1049 cm-1 is associated with the C-O/C-C stretching vibrations, according to Bessadok (2009) and John et al. (2010). Furthermore, the band around 1249 cm-1 is attributed to the acetyl groups of lignin and exhibited a decrease in intensity following treatment with NaOH, as noted by Gierlinger et al. (2008). This decrease in intensity is indicative of the removal of lignin from the bamboo structure, with a more pronounced reduction observed in treatments involving boiling alkali solution.

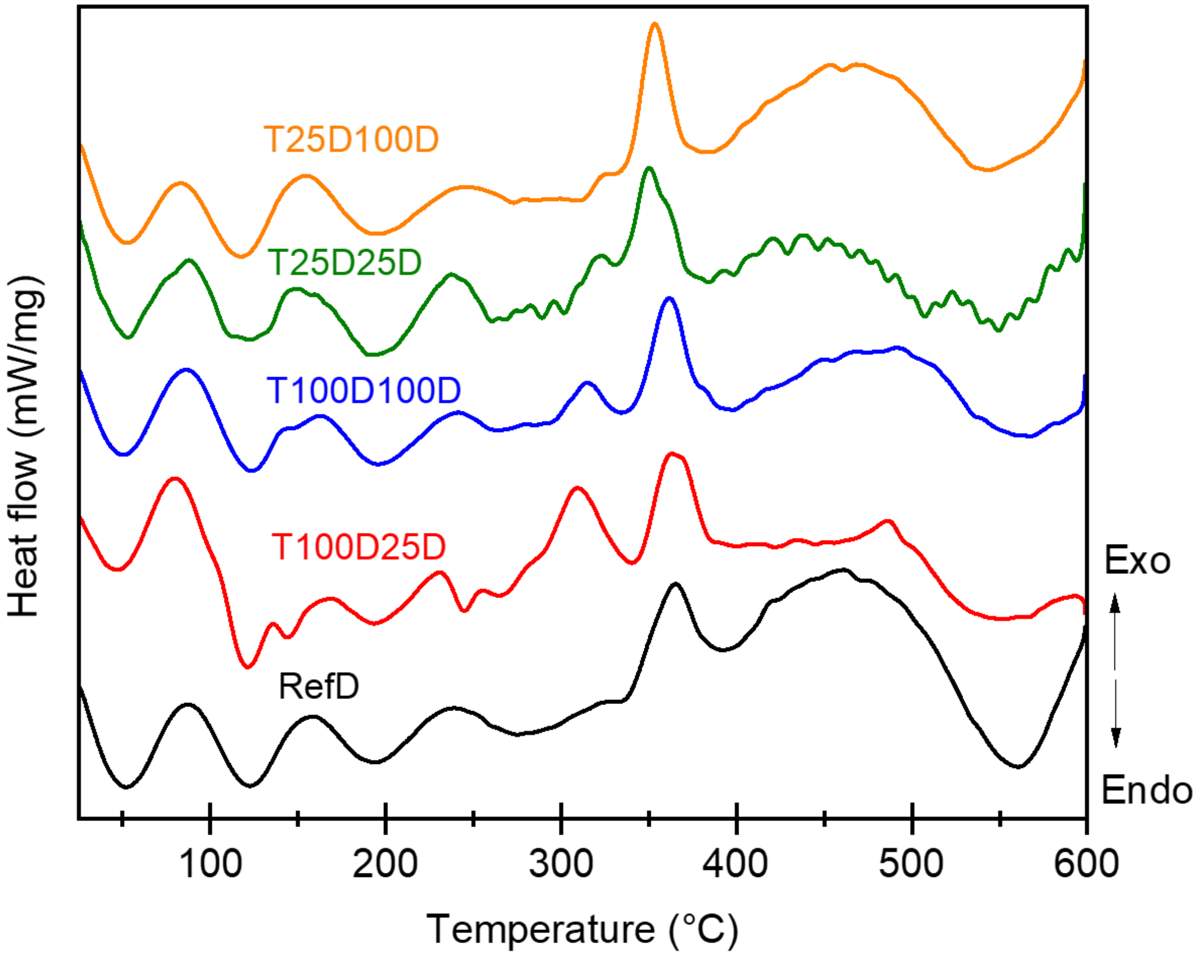
In the regions near 2900 cm-1, the band is attributed to the symmetric and asymmetric stretching of CH2 groups. The band at 3400 cm-1 corresponds to the -OH stretching vibration from the cellulose in bamboo fibers. Additionally, the band associated with water adsorbed on the fibers is observed at wavenumbers close to 1639 cm-1. Notably, in all the regions related to water absorption, no significant changes were observed. This aligns with the results obtained from the water absorption test conducted after 24 hours of immersion for densified samples.

Thermogravimetric analysis

Bamboo fibers primarily consist of hemicellulose, cellulose, and lignin, with the possibility of other substances being present as components or impurities on the fiber surface. The thermal decomposition of bamboo fibers can be categorized into four main stages: moisture evaporation, hemicellulose decomposition, cellulose decomposition, and lignin decomposition, as highlighted by Zakikhani et al. (2015).

Hemicellulose decomposition takes place at lower temperatures, followed by further decomposition of cellulose. Lignin, characterized by the highest decomposition temperature, exhibits the lowest rate of mass loss within the employed heating rate, according to studies such as Zhou et al. (2014). Figure 30 illustrates the differential scanning calorimetry (DSC) curves of both untreated and treated bamboo fibers.

Figure 30: DSC curves of untreated and treated bamboo samples.

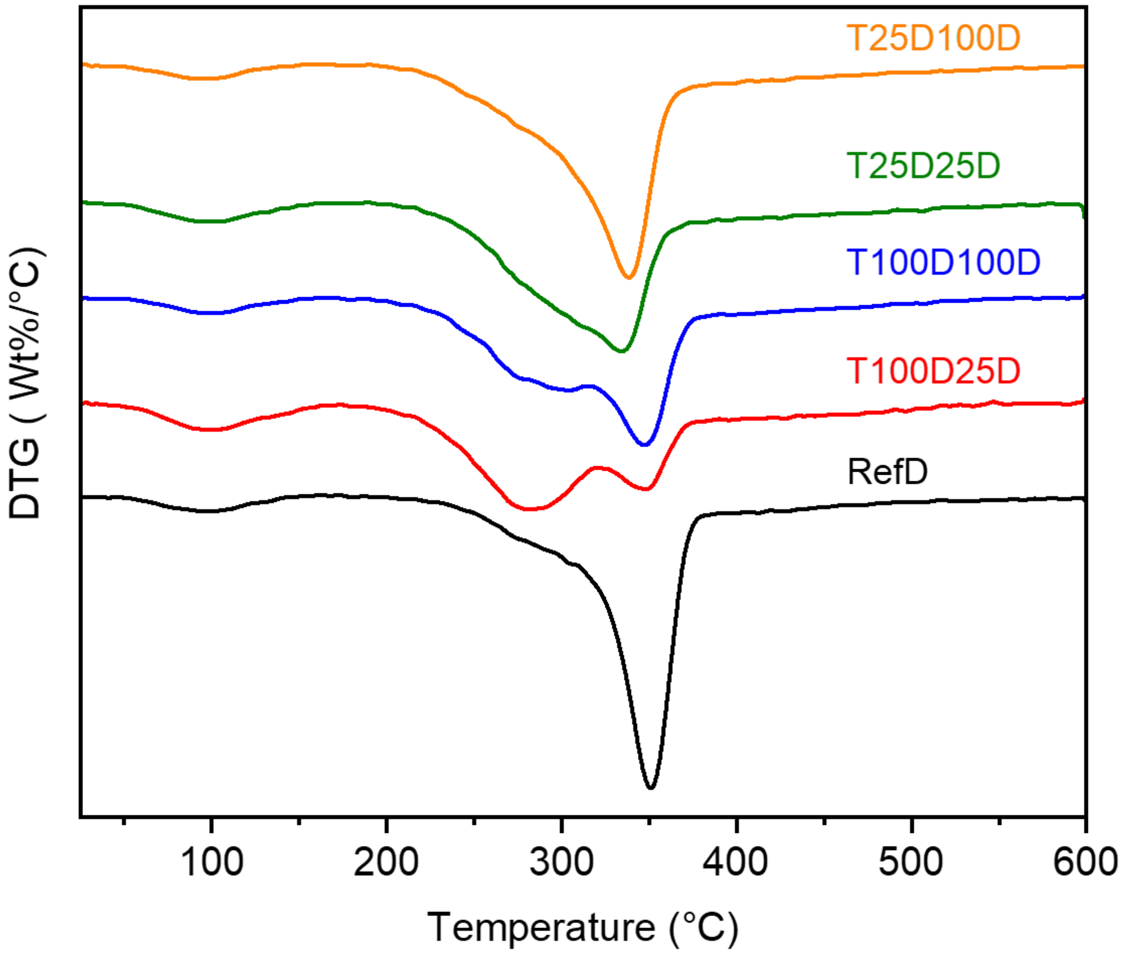


Source: Own authorship.

At temperatures lower than 200 °C, endothermic peaks corresponding to the evaporation of moisture present on the fiber surface are observable. Cellulose decomposition is evident at temperatures close to 350 °C, with an endothermic peak evident in all bamboo fiber samples. Notably, there is a decrease in peak intensity for treated groups at room temperature (T25D100D and T25D25D), which could be linked to the alkaline chemical treatment impacting bamboo cellulose. Two exothermic phenomena are observed at 275 °C and 365 °C, associated with the decomposition of hemicellulose and lignin, respectively. A reduction in peak intensity at 365 °C for the T100D25 group is apparent. This result may be attributed to enhanced lignin removal when treating bamboo fibers with NaOH at 100 °C and drying at 25 °C, as reported by Chen et al. (2017), Kabir et al. (2013), and Tserki (2005).

The differential thermogravimetric (DTG) curves of untreated and treated bamboo (refer to Figure 31) reveal a small mass loss for all groups in the temperature range between 25 and 200 °C, associated with moisture removal.

Figure 31: DTG curves of untreated and treated bamboo samples.



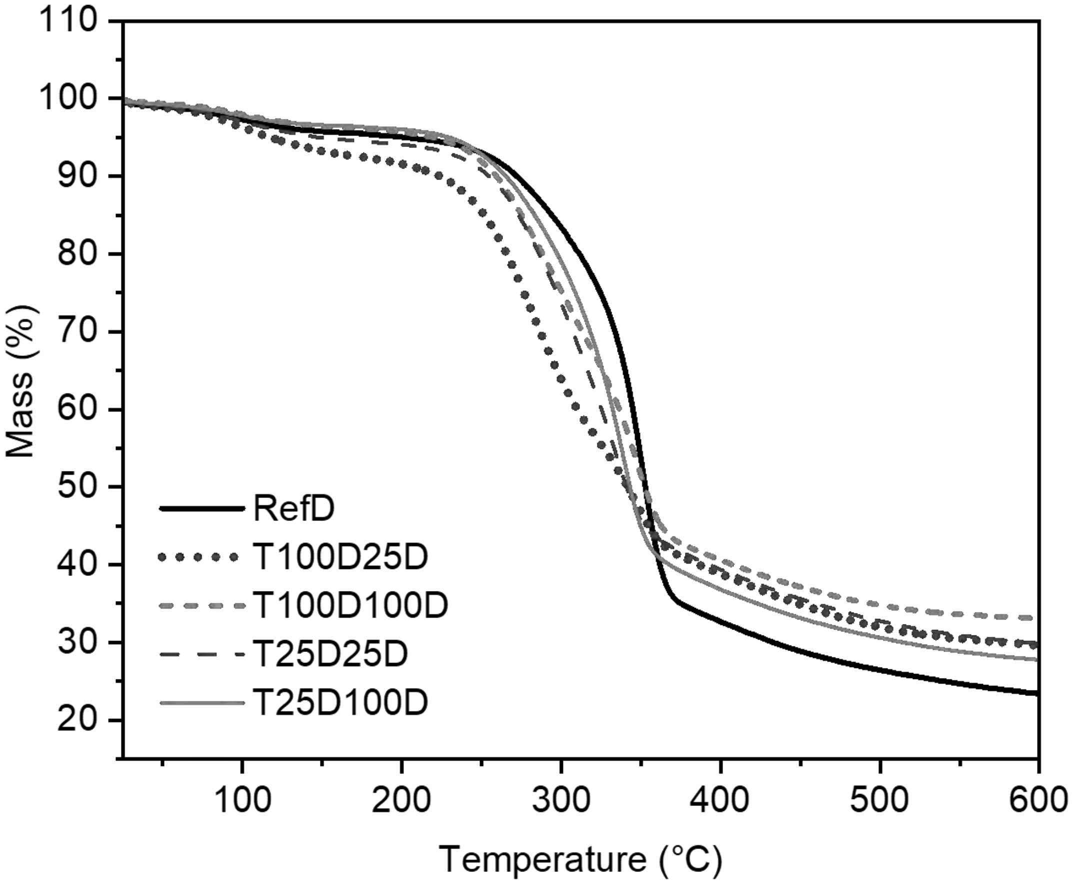
Source: Own authorship

A peak close to 300 °C is linked to the decomposition of hemicellulose, while a peak close to 350 °C is associated with the decomposition of cellulose in the fibers. Notably, in samples without alkaline treatment, the peak between 300 °C and 350 °C is more intense than in samples treated with NaOH.

The reduction in cellulose content in treated samples aligns with the observed loss of mechanical resistance after pretreatment. This indicates that the alkali treatment not only caused the expected removal of lignin content but also resulted in the degradation of the interface between parenchyma and sclerenchyma and the removal of cellulose content.

Figure 32 illustrates the thermogravimetric (TG) curves of untreated and treated bamboo samples. The mass loss before and after treatments is notably similar, with a greater mass loss observed for the untreated fiber. This result may be attributed to the presence of alkalis on the surface of the treated samples, which do not evaporate within the studied temperature range.

Figure 32: TG curves of untreated and treated bamboo samples.



Source: Own authorship.

5 Conclusions

The impacts of alkali treatment and pre-densification drying processes on Dendrocalamus asper bamboo species were evaluated, and the findings can be summarily described as follows:

(1) The utilization of a NaOH+Na2SO3 solution for bamboo treatment exhibited positive effects on compressibility during the densification process. This treatment proved advantageous by extracting lignin content and mitigating internal stresses, thereby reducing the energy and load necessary to attain a specified densification level. Notably, samples subjected to the boiling solution and subsequent room temperature drying required only 40% of the stress applied to non-treated bamboo. This outcome signifies a substantial reduction in the energy and load requirements for achieving a 50% densification degree. This implies that alkali treatment could enable a greater degree of densification with consistent energy consumption. In a closed pressing system it would be possible to increase the densification degree within the same amount of energy, which could lead to enhanced mechanical properties with the potential to improve the material's mechanical properties and boost its density, given the correlation among these properties

(2) Anticipated improvements in the physical performance of densified bamboo through pretreatment were not observed as both treated and untreated bamboo exhibited comparable behavior in terms of physical properties in the densified samples.

(3) The results of the three-point bending test revealed that the pre-treatment had an adverse effect on the modulus of rupture, limit of proportionality, and elastic modulus of densified bamboo. Specifically, the modulus of rupture exhibited a decline ranging from 29% to 46%, the limit of proportionality decreased by 32% to 48%, and the modulus of elasticity experienced a reduction within the range of 18% to 33%

(4) Thermal analysis, Fourier-transform infrared (FT-IR) tests, and image analysis of fiber volume fraction collectively indicated a reduction in hemicellulose and

cellulose content within bamboo, accompanied by the removal of lignin subsequent to alkaline treatment and the appearance of internal cracks that persisted with a densification degree of 50%. Was analyzed an enhanced lignin removal when treating bamboo fibers with NaOH at 100 °C and drying at 25 °C

To summarize, subjecting bamboo to this concentration of alkali solution in an open system with a densification degree of 50% is not enough to have a proper densification compromising the mechanical properties of the material. Additionally, a qualitative analysis was conducted on the efficiency of delignification solutions, with further testing required in the future for a quantitative analysis of lignin removal. Further exploration using a closed pressing system to optimize the parameters involving the use of delignification prior to densification and to achieve a higher densification degree and different lignin extraction levels are necessary to investigate the potential of these pre-treatments in enhancing the physical and mechanical characteristics of densified bamboo.

6 Future research suggestions

For future research, it is important to analyze closed thermo-mechanical pressing systems, achieving a higher thickness reduction, increasing the average fiber fraction of the densified bamboo, and closing the voids and eliminating the cracks, while the current study could only analyze a reduction in 50% of the original thickness, being able to a better understanding on how the bamboo densification behaves in a more controlled environment, analyzing how the process affects the structure and strength of the bamboo.

Additionally, more samples should be analyzed with different concentrations of the solution and in different immersion times for a better understanding of the delignification process. Also, the drying conditions should be considered for a fully understanding of the conditions of the two-step delignification and densification. For a better understanding of the properties of the delignified-densified bamboo, studies about the fire resistance and durability with aging techniques should be carried out.

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