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Desenvolvimento de materiais de alto desempenho a base de bambu através de processo de densificação.

Development of high-performance materials based on bamboo through densification process.

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DEDICATION

To my inspiring family, my Father, Mother, Sisters, and Brothers, for constantly supporting and encouraging me and pulling me up when I would be down.

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RESUMO

KADIVAR, M. **Desenvolvimento de materiais de alto desempenho a base de bambu através de processo de densificação**. 2020. 177 p. Tese de Doutorado – Faculdade de Zootecnia e Engenharia de Alimentos, Universidade de São Paulo, Pirassununga, 2020.

O bambu se tornou um material atraente para aplicações de engenharia em vista de aspectos relacionados a sua sustentabilidade, confiabilidade, excelentes propriedades físicas e mecânicas e facilidade de acesso. No entanto, vários problemas, como propriedades heterogêneas e questões de durabilidade, ainda impedem o uso generalizado do bambu como material de construção. Portanto, o bambu passa por tratamentos e processos especiais para resolver essas dificuldades. Os processos de densificação podem ser aplicados para diminuir a heterogeneidade dos colmos de bambu, melhorar seu desempenho mecânico e, consequentemente, auxiliar no uso mais eficiente do bambu como material industrial nas construções modernas. A revisão da literatura sobre densificação de bambu sugere que o teor de umidade inicial, o grau de densificação, a temperatura e a taxa de compressão são os principais parâmetros para a eficiência do processo. Os estudos experimentais nesta tese foram orientados para alcançar parâmetros ótimos para o processo de densificação. Primeiramente, a influência do teor de umidade inicial na densificação termo-mecânica (TM) do bambu foi investigada por meio de caracterizações mecânicas, químicas e físicas. Os resultados desta etapa mostraram que o processo de densificação aumenta a densidade e melhora o comportamento frente a solicitações de tração na flexão, a saber: módulo de ruptura (MOR), módulo de elasticidade (MOE), limite de proporcionalidade (LOP) e energia específica (SE) do bambu. As amostras densificadas com 10% de umidade inicial apresentaram as melhores propriedades de flexão, com um MOR médio, MOE e MOE dinâmico de 318 MPa, 27,7 GPa e 34,1 GPa respectivamente, com um aumento de 56% para MOR e 41% para MOE em comparação com as referencias não densificadas. A análise por microscopia eletronica de varedura (MEV) de cortes transversais de bambu mostrou o rearranjo das fibras, compactação densa dos feixes vasculares e fechamento parcial das cavidades como resultados da densificação. Análises de caracterização física revelaram que o processo de densificação foi prejudicial para a estabilidade dimensional do bambu. Portanto, em um segundo estudo, os parâmetros de modificação de TM (temperatura, taxa de compressão e tempo de prensagem) foram otimizados para alcançar a melhor estabilidade dimensional por meio de spring back (recuperação da espessura com o tempo após a densificação), absorção de água e medições de inchamento. De acordo com os resultados desta etapa, a o grau de densificação (DD) máxima alcançável em que nenhuma ruptura por cisalhamento e nenhuma deformação lateral ocorre é de cerca de 43,6%, que pode ser obtida densificando o bambu a 200°C com uma taxa de compressão de 2 mm/min. A análise de densitometria de raios X confirmou que o maior valor de densidade, 1,30 g/cm3, é obtido com um DD em torno de 50%. Os menores valores de spring back, absorção de água e inchamento em espessura, ou seja, 4,72%, 23,80% e 17,70% respectivamente, para o bambu densificado, são obtidos quando o processo de densificação é conduzido a 200°C com uma taxa de compressão de 6,73 mm/min. Por último, foram sugeridos dois exemplos de aplicação para bambu densificado; bambu planificado-densificado e painel sanduíche de bambu. Em conclusão, ganhos significativos na desempenho físico e mecânico do bambu podem ser obtidos com o processo de densificação.

Palavras chave: Bambu, Dendrocalamus asper, processo TM, densificação, caracterização física e mecânica, microestrutura.

ABSTRACT

KADIVAR, M. **Development of high performance materials based on bamboo through densification process**. 2020. 177 p. Tese de Doutorado – Faculdade de Zootecnia e Engenharia de Alimentos, Universidade de São Paulo, Pirassununga, 2020.

Due to its sustainability, reliability, excellent physical and mechanical properties, and ease of access, bamboo has become an attractive material for engineering applications. However, several problems, such as heterogeneous properties and durability issues, still hinder the widespread use of bamboo as a building material. Therefore, bamboo undergoes special treatments and processes to solve these difficulties. Densification processes can be applied to decrease the heterogeneity of bamboo culms, enhance its mechanical performance, and consequently help using bamboo more efficiently as an industrial material in modern constructions. Reviewing the literature on bamboo densification suggests that initial moisture content, densification degree, temperature, and compression rate are the main effective parameters of the process. Therefore, the experimental studies in this thesis have been oriented to achieve optimal parameters for the densification process. First, the influence of initial moisture content on thermo-mechanical (TM) densification of bamboo was investigated through mechanical, chemical, and physical characterizations. The results of this step showed that the densification process increases density and all related bending properties (modulus of rupture (MOR), modulus of elasticity (MOE), the limit of proportionality (LOP), and specific energy (SE)) of bamboo. The densified samples with 10% initial moisture content presented the best bending properties, with an average MOR, MOE, and dynamic MOE of 318 MPa, 27.7 GPa, and 34.1 GPa respectively, with an increase of 56% for MOR and 41% for MOE in comparison with un-densified counterparts. SEM analysis of bamboo cross-sections showed the rearrangement of fibers, dense compaction of vascular bundles, and partial closure of cavities as results of densification. Physical characterization analyses revealed that the densification process was detrimental to the dimensional stability of bamboo. Therefore, in a second study, TM modification parameters (temperature, compression rate, and pressing time) were optimized to achieve the best dimensional stability through spring back (change of thickness with time after densification), water absorption, and thickness swelling measurements. According to the results of this step, the maximum achievable DD in which no shear failure and no lateral deformation occurs is about 43.6%, which can be obtained by densifying bamboo at 200 °C with a compression rate of 2 mm/min. X-ray densitometry analysis confirmed that the highest value of density, 1.30 $g.cm^{-3}$, is achieved with a DD of around 50%. The lowest values of spring back, water absorption, and thickness swelling, i.e., 4.72%, 23.80%, and 17.70% respectively, for densified bamboo, are obtained when the densification process is conducted at 200 °C with a compression rate of 6.73 mm/min. Lastly, two examples of application for densified bamboo were suggested; flattened-densified bamboo and bamboo sandwich panel. In conclusion, significant gains in bamboo performance could be obtained with the densification process.

Keywords: Bamboo, Dendrocalamus asper, TM process, densification, physical and mechanical characterization, microstructure.

ABBREVIATIONS LIST

ASTM	American Society for Testing and Materials
BBP	Bamboo-based panels
EN	European Committee for Standardization.
FAO	Food and Agriculture Organization of the United Nations
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

FIGURE LIST

TABLE LIST

LIST OF PUBLICATIONS, AWARDS AND GRANTS DURING DOCTORATE

Published/ submitted journal papers:

KADIVAR, M.; GAUSS, C.; MÁRMOL, G.; DOMINIQUE, A.; SÁ, D.; FIORONI, C.; GHAVAMI, K.; SAVASTANO JR, H. The influence of the initial moisture content on densification process of D. asper bamboo: Physical-chemical and bending characterization. Construction and Building Materials, v. 229, p. 116896, 2019.

KADIVAR, M.; GAUSS, C.; GHAVAMI, K.; SAVASTANO JR, H. Densification of Bamboo: State of the Art. MDPI, Materials, v. 13, p. 4346, 2020.

KADIVAR, M.; GAUSS, C.; GHAVAMI, K.; SAVASTANO JR, H. optimization of bamboo thermo-mechanical densification process (submitted to the construction and building materials journal.)

GAUSS, C.; KADIVAR, M.; SAVASTANO JR, H. Effect of disodium octaborate tetrahydrate on the mechanical properties of Dendrocalamus asper bamboo treated by vacuum/pressure method. Journal of Wood Science, v. 65, n. 27, 2019.

GAUSS, C.; KADIVAR, M.; HARRIES, K.; SAVASTANO JR, H. Chemical modification of *Dendrocalamus asper* bamboo with citric acid: effects on the physical-chemical, mechanical and thermal properties. Journal of Cleaner Production, , v. 279, p. 123871, 2021.

GAUSS, C.; HARRIES, K. A.; KADIVAR, M.; AKINBADE, Y. A.; SAVASTANO JR, H.; Quality assessment and mechanical characterization of preservative-treated Moso bamboo (*P. edulis*). European Journal of Wood and Wood Products, v.78, p. 257-270, 2020.

GAUSS, C.; KADIVAR, M.; PEREIRA, R.; SAVASTANO JR, H. Assessment of Dendrocalamus asper bamboo treated with tannin-boron preservatives. (under revision in Construction and Building Materials Journal)

Contribution in the writing preface of a book " UTILIZAÇÃO DE RESÍDUOS AGROINDUSTRIAIS NA CONSTRUÇÃO", EDITORA UNICAMP, ISBN: 9786586253337, 2020.

Participation in events:

KADIVAR, M.; GAUSS, C., CHARCA, S., GHAVAMI, K., SAVASTANO JR, H. Physical Properties of Thermo-Hydro-Mechanically (THM) Flattened and Densified Bamboo (Moso and Dendrocalamus Asper). In: XVIII Brazil MRS Meeting - Proceedings, Balneário Caboriú, 2019.

KADIVAR, M.; GAUSS, C.; MARMOL, G.; FIORONI, C.; SA, A. D.; SAVASTANO JR, H. Evaluation of an optimized bamboo sandwich panel using layers of two different bamboo functional elements. In: XVIII Brazil MRS Meeting - Proceedings, Balneário Caboriú, 2019.

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PhD Student Scholarship by CAPES, Brazil, 2018, 2019.

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Coordinator of Bambusp Social Project (Centro comunitário de capacitação em bambu) at University of São Paulo, Brazil. July 2019-April 2021, Project approved and financed by the Culture and Extension Department of the USP.

Short academic visit grant by UHH foundation to visit Wood Science Department of the University of Hamburg, Germany, 2020.

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1. CHAPTER 1 - Introduction

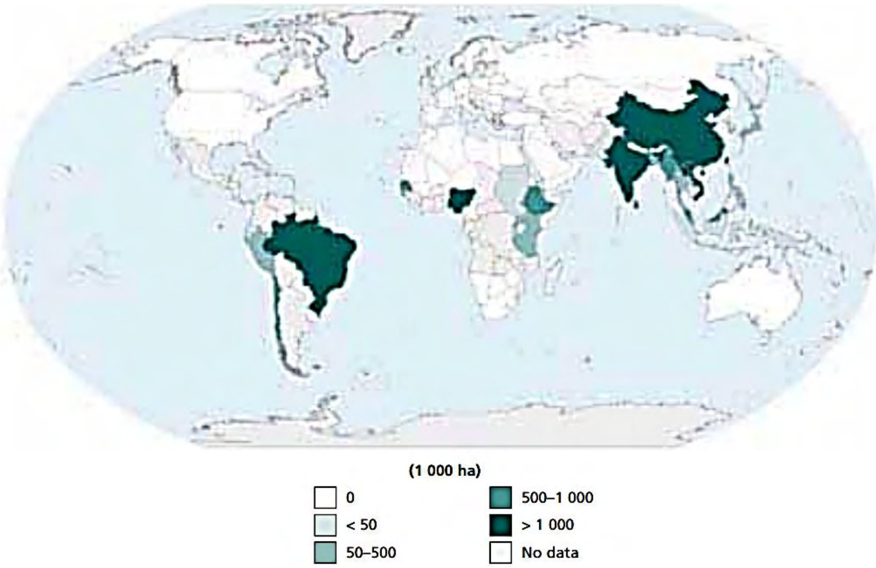
1.1. Contextualization and motivation

Environmental concerns related to different sectors of industry, such as the construction sector, which contributes to a large percentage of carbon dioxide emissions, force humans to substitute the energy-intensive materials to woody biomass, as low energy-demanding materials, may contribute to reducing greenhouse gas emissions. However, the world's forest resources are limit and are decreasing as a result of growing human populations and increasing demand for food and land. In these aspects, bamboo, which is a giant grass, can be an excellent option to complement timber.

Bamboo is a fast-growing plant of the Poaceae family (subfamily Bambusoideae) with around 1450 species that is part of the cultural and ecological landscape of many countries in Asia, America and Africa (CLARK; LONDOÑO; RUIZ-SANCHEZ, 2015; GOH; YAP; TONG, 2019). Bamboo forests are mainly found in the southern hemisphere, widely distributed in Asia (67%), Americas (30%) and Africa (3%), in regions with tropical, subtropical and temperate climate zones (HUANG; SUN; MUSSO, 2017a). Having the huge potential for bamboo with 9.3 million hectares of bamboo forest area (Fig.1.1), Brazil is the largest country in terms of bamboo resources (FAO, 2010).

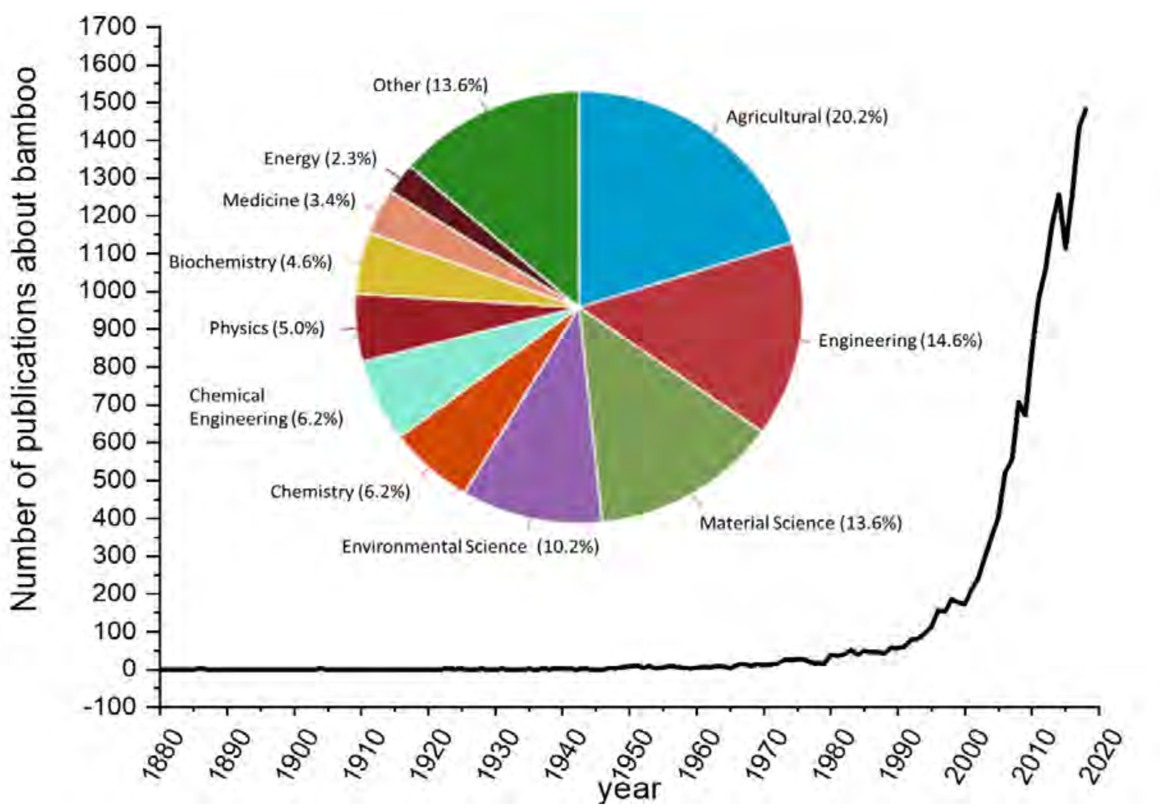
With the increased interest in environment and sustainability, bamboo as a sustainable material has been widely used in many applications (AWOYERA; UGWU, 2017; NIRMALA et al., 2018; DARIA; KRZYSZTOF; JAKUB, 2020; SILVA et al., 2020; SUN; HE; LI, 2020), and significantly in the last century, the bamboo industry has been developed rapidly to form a range of increasingly functional products, based on a combination of performance and sustainability requirements. Scientific articles related to bamboo (Fig.1.2) have been increasing over the past decade. According to the scientific research database Scopus, until the end of 2020, a total of 20749 publications related to bamboo have been published, concentrated in recent years. Most of these works were developed in the areas of agriculture, engineering, and materials science.

Figure 1.1- Area of bamboo by country



Source: (FAO, 2010)

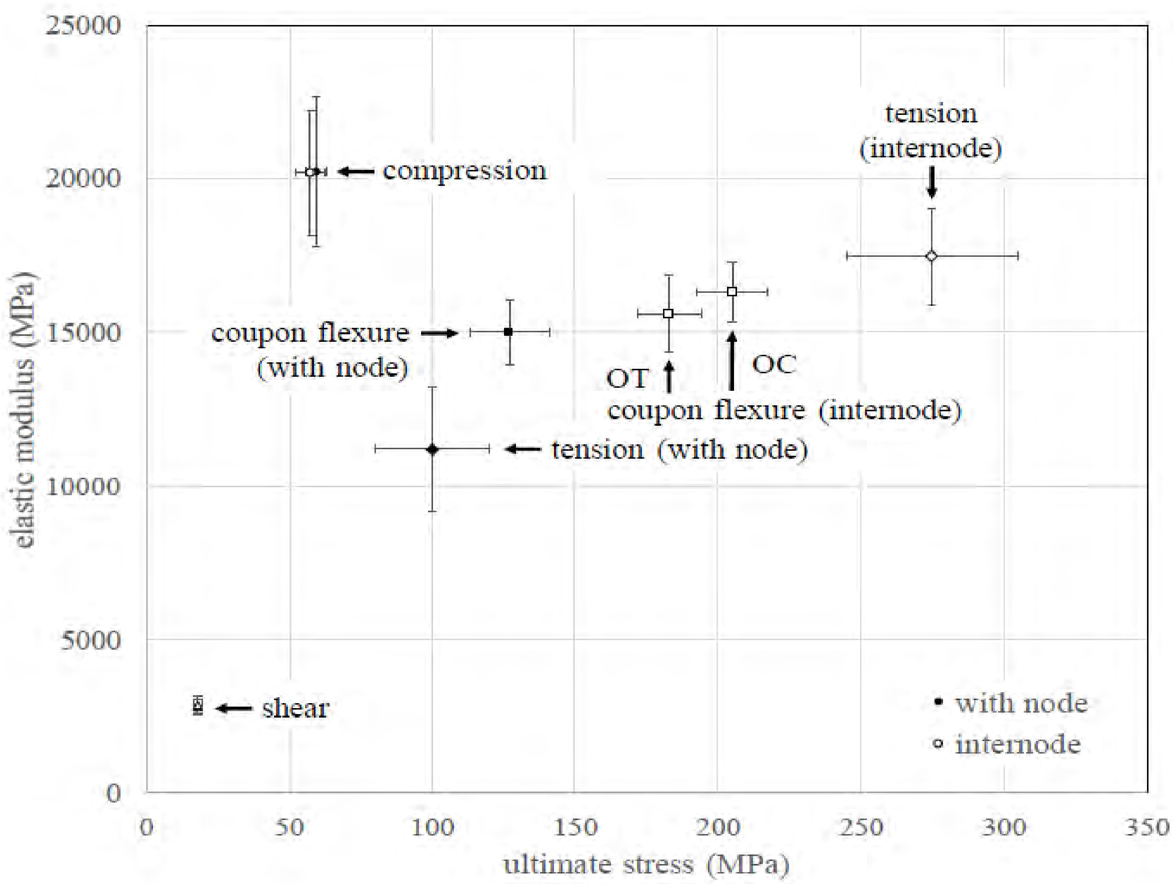
Figure 1.2 - Scientific articles related to bamboo and the corresponding areas of knowledge.



Source: Plots generated on Scopus® database platform (Elsevier B.V.)

Among its excellent advantages, availability, easy large-scale cultivation, rapid growth, high strength of bamboo combined with energy conservation, the reduction of CO2 emissions and carbon storage suggest that the material can play an important role in the construction industry. Its potential as a sustainable building material has been proven by several researchers (SHARMA; DHANWANTRI; MEHTA, 2014). Bamboo is distinguished by its excellent specific mechanical strength in comparison with conventional building materials, such as steel, cement, and wood (GHAVAMI, 1992). Figure 1.3. shows a summary of the mechanical characterization of Brazilian P. edulis bamboo using ISO 22157 test methods. It shows that bamboo is very strong in tension but weak in shear.

Figure 1.3 - Mechanical characterization of P edulis: strength and modulus



Source: (GAUSS; SAVASTANO; HARRIES, 2019)

In its natural condition, bamboo culm has been utilized as an excellent structural element in the fabrication of rural houses. However, there are several challenges that have been barriers to the use of bamboo compared with concrete, steel, and wood. Bamboo culm grows as a hollow cylindrical pole composed of regions with aligned and continuous fiber bundles, called internodes, separated by parts with solid transversal diaphragms and interwoven fiber bundles in the wall thickness, called nodes. The ununiform distribution of fiber bundles across the wall thickness and differences in the culm diameter, thickness and internode length along the culm's height, are among barriers in the utilization of raw bamboo as an industrial structural material for modern constructions.

Designing and producing a series of bamboo-based panels (BBP) with different structures can overcome bamboo challenges and transfer it to a large flat surface stabilized product with excellent mechanical, physical, and aesthetical advantages. BBP started to appear in the construction industry in the 1970s (HUANG; SUN; MUSSO, 2017b), following the advancement of the modern timber industry (LI et al., 2019). A wide range of commercialized products and applications in the construction sector, i.e., floors, ceilings, and wall finishes can be found. However, more research and innovation are needed to develop a simple processing approach for high value-added bamboo products and to transfer it as an ideal material that fits actual market requirements.

Bamboo can be more efficiently used in structural bamboo products. Improved fundamental understanding of the structure and mechanical properties of bamboo, and of bamboo processing, could foster the adoption of structural bamboo products, extending the use of bamboo (DIXON et al. 2018). There are ways in which the properties of bamboo can be enhanced by modification through eco-friendly methods. Novel technologies on bamboo culm flattening and Densification have been reported and attempted during the last decades. Flattening can overcome the circular shape of bamboo and densification makes it homogeneous across the wall thickness. The combination of these two technologies makes it possible for inhomogeneous round shape bamboo to be substituted for uniform flat material so that commercially uninteresting feature of bamboo can be modified into high performance and high added-value products.

Densification of bamboo is one of the improvements that has been suggested as post-treatment of flattened bamboo culms to avoid cupping distortion and to close the indentations.

This method can also be used in the bamboo strips without the flattening step. The main aim of bamboo densification is to decrease its heterogeneity and improve its mechanical and moisture sorption properties since, in natural bamboo, density and mechanical strength gradually change from the outer to the inner layer of the bamboo transversal section.

One emerging eco-friendly method to achieve densification is the combined use of temperature, moisture, and mechanical action, the so-called thermo-mechanical treatment (THM). THM is an important process for bamboo products manufacturing, and could lead to densification. Bamboo densification indeed happens in processing scrimber and could be in other bamboo products. It can increase panels' density, improve mechanical properties, and make their structure uniform.

The wood densification has been studied and widely reported (LI et al., 2013; KUTNAR; COE; SERNEK, 2015; ESTEVES et al., 2017; KÚDELA et al., 2018). However, the information on basic properties of bamboo densification for supporting higher utilization of such materials are still lacking, especially those related to softening behavior, deformations and setting of compressed conditions(GAO; GUO; LUO, 2018). Although densified bamboo has not been considered as a product itself yet (DIXON et al., 2016a), the influence of the THM process has to be fully understood and explored since the quality of final products is affected by the experienced process.

Wood and bamboo have similar chemical compositions with comparable lignin contents which make the use of THM treatments feasible for bamboo(ARCHILA-SANTOS; ANSELL; WALKER, 2014). The strategy of THM process can be used for bamboo to increase the density and consequently mechanical properties of the material through non-destructive means. It is possible to make use of the viscoelastic behavior of bamboo, facilitating the flow (plasticization) of lignin by increasing the moisture content or using pre-treatment which might lead to the reduction of the glass transition temperature (Tg). It can have a positive effect on the modification of bamboo's macrostructure and microstructure when applying elevated temperature and pressure.

The aim of this work is to understand the deformation and softening behavior of bamboo, evaluate the densification process, and the main parameters, in order to apply this technology to the construction industry. Thermo-mechanical treatment (TM) which is a type of Thermo-Hydro- mechanical method (THM) has been applied to achieve densification. A comprehensive study has been performed to obtain the optimal parameters range for the process to be more effective, to shorten the processing time, and to lower the energy consumption. The influence of the densification process on natural bamboo in terms of physical-chemical and mechanical properties (density, dimensional stability, dynamic and static bending, fracture behavior, cellulose structure, and chemical composition) are evaluated.

1.2. General objectives

The general objective of this work is to develop products from bamboo *Dendrocalamus asper* with an improved physical and mechanical performance compared to the natural material through the densification process and to predict the required process parameters, in order to apply this technology to the construction industry.

1.3. Technical objectives

- To develop an experimental method for an advanced understanding of the influence of temperature on the stress-strain relationship in transverse compression of bamboo.

- To evaluate the influence of the densification process in physical and mechanical properties of bamboo.

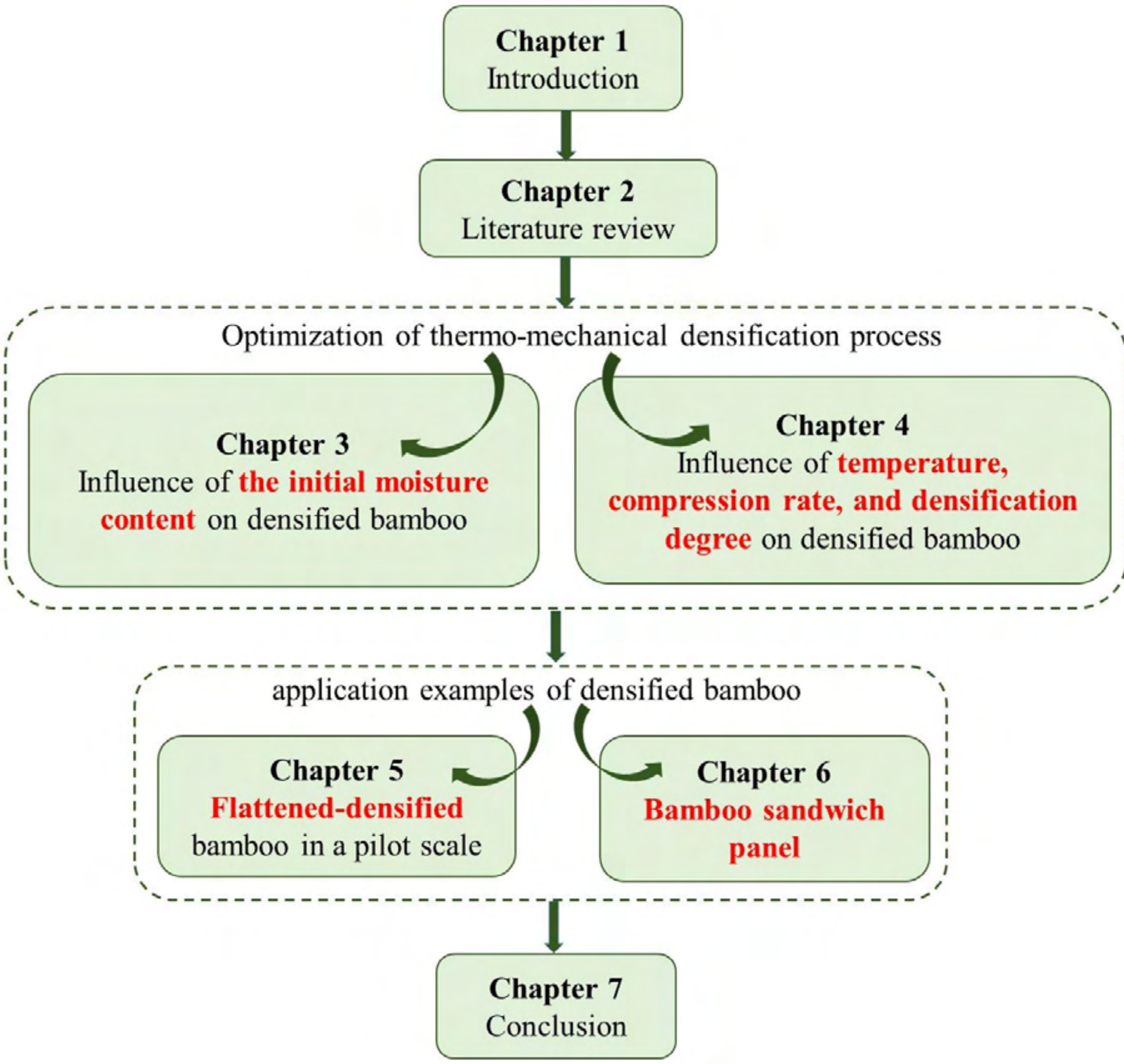
- Study of the effective parameters during the thermo-mechanical (TM) process; the influence of water content, densification degree, temperature, and the rate of compression.

- To sugest some examples of application for densified bamboo, flattened-densified bamboo and bamboo sandwich panel.

1.4. Thesis structure

This thesis has been structured in 7 chapters (Figure 1.2). Chapters 2,3 and 4 are presented in the format of fully published/submitted journal papers, and chapters 5 and 6 are the published conference papers.

Figure 1.4- Structure of the thesis.



Chapter 1: This chapter presents the context, motivation, and objectives of this work, along with the organization and structure of the thesis.

Chapter 2: This chapter presents the state of the art of densified bamboo and provide a literature review of the publications related to the effective factors for the bamboo densification process.

Chapter 3: This chapter presents the influence of densification process on physical-chemical and mechanical performance of bamboo and also an assessment of initial moisture content.

Chapter 4: This chapter focuses on the optimization of thermo-mechanical densification process of bamboo in terms of temperature, compression rate, and densification degree.

Chapter 5: This chapter examines bamboo densification in a pilot scale as a post-treatment of flattened bamboo, and compare the process for two different bamboo species.

Chapter 6: This chapter presents an application example of densified bamboo, as a functional element of bamboo sandwich panel development.

Chapter 7: This chapter provides the final remarks and a summary of the main conclusions and recommendations reached during the development of this thesis. Future activities related to this area of research are suggested at the end.

2. CHAPTER 2 - Literature review (Densification of Bamboo: State of the Art)

This chapter has been published in MDPI, Materials Journal, v. 13, p. 4346, 2020, (DOI: 10.3390/ma13194346).

Abstract:

Densification processes are used to improve the mechanical and physical properties of lignocellulose materials by either collapsing the cell cavities or by filling up the pores, consequently reducing the void volume fraction. This paper focuses on an extensive review of bamboo densification process, which is achieved by compressing the material in the direction perpendicular to the fibers using mainly two different techniques: an open system, thermo-mechanical (TM), or a closed system, viscoelastic-thermal-compression (VTC). The main aim of bamboo densification is to decrease its heterogeneity, as well as to improve its mechanical and physical performance. In addition, densification may occur during the manufacturing of bamboo products in which hot-pressing processes are used to mold bamboo panels. There are over 1600 publications about bamboo, concentrated in the recent decade, mainly about engineered materials. Although several papers regarding bamboo and wood densification are available, very few studies have comprehensively investigated the densification process solely through compression of natural bamboo culms. According to the literature, applying a combination of compression of 6–12 MPa at temperatures between 120–170 °C for 8–20 min can produce materials with higher strength in comparison to the mechanical properties of natural bamboo. The majority of research on bamboo densification indicates that the modified material results in improved properties in terms of density, hardness, bending strength, stiffness, and durability. This paper provides a review that consolidates knowledge on the concept of bamboo culm densification, discusses the roles of parameters that control the process, ascertains the best practice, and finally determines gaps in this field of knowledge.

Keywords: bamboo; densification; thermo-mechanical; viscoelastic-thermal-compression

2.1. Introduction

Sustainable materials are in high demand, particularly within the forest products industry, due to the increased need to decarbonize the built environment. Population and economic growth are straining the finite supply of suitable products. In addition, climatic changes and environmental impacts of industrial products are leading to the development of eco-friendly resources (SAVASTANO; WARDEN; COUTTS, 2005). According to life cycle assessment studies and considering the three pillars of sustainability (e.g., environmental, social, and economic impacts), bamboo has indisputable potential as a sustainable resource for a wide range of utilizations (LUGT; BREZET, 2009; VOGTLÄNDER; VAN DER LUGT; BREZET, 2010; YU; TAN; RUAN, 2011; ZEA ESCAMILLA; HABERT, 2014; AGYEKUM; FORTUIN; VAN DER HARST, 2017; ESCAMILLA et al., 2018). Bamboo as a raw material has a beautiful aesthetic, good mechanical strength, and is harvested in short rotation periods; however, it also has disadvantages (HUANG; SUN; MUSSO, 2017b). Variable cylindrical geometry, heterogeneity, and the variability of properties of full-culm bamboo generate challenges for its use in mainstream production. This has led to engineering the material to obtain standardized prismatic shapes and less variability in mechanical properties and performance. Consequently, a list of bamboo-based panels (BBP) having a large flat surface and different bamboo units have been designed and produced to overcome these disadvantages since the 1970s (HUANG; SUN; MUSSO, 2017b). Mechanical, physical, and aesthetical properties of BBPs can be controlled and engineered using specific species and processing methodologies. Although most methods follow the advancement of modern timber industry (LI et al., 2019), there are differences in the bamboo-based production due to the geometry and the material consistency.

Densification is used to manufacture bamboo scrimber and could also be used in the production of other bamboo products. Increasing the density of bamboo elements can lead to more efficient use of structural products because the quality of the final product depends on the performance of its components. Wood densification has been investigated and comprehensively reported (KUTNAR; ŠERNEK, 2007; LI et al., 2013; KUTNAR; SANDBERG; HALLER, 2015; ESTEVES et al., 2017; KÚDELA et al., 2018). Kutnar et al. 2015 summarized the state of the art and knowledge in the field of compressed wood processing and products (KUTNAR; SANDBERG; HALLER, 2015). Notwithstanding, there is still a lack of knowledge about the fundamental features of bamboo full-culm densification, particularly the softening behavior, physical properties, and effective processing parameters (GAO; GUO; LUO, 2018). Fundamental understanding of the bamboo element structure and engineering properties, as well as bamboo processing, can open up new advanced methodologies to promote its use for new applications (DIXON et al., 2016a). In this respect, flattening and densification are two new technologies that have been explored in the market and have recently been published in the academic literature. Flattening can overcome the circular shape of bamboo and densification makes it homogeneous across the wall thickness. A combination of these two technologies makes it possible for inhomogeneous round shape bamboo to be substituted by a uniform flat material so that commercially uninteresting features of bamboo could be altered. The objective of this paper is to present the state of the art of densified bamboo and provide a review of the publications related to the effective factors for the bamboo densification process.

2.2. Densification Concept

Densification can be defined as a process to increase the density and redesign the microstructure of the material. Chemical, thermal, or mechanical factors can influence this process. In the case of materials with cellular structure, densification has been defined as the last regime of stress-strain curves in compression after linear elastic and plateau region (GIBSON, 2005). In these types of materials like wood, the conventional understanding of the stress-strain relationship under compression identifies three distinct regions. In the beginning, the material exhibits elastic behavior, where load and deformation are linearly related. At the end of the elastic regime, the cells collapse and an inelastic behavior commences. In the inelastic region, strain increases rapidly with little or no change in stress, often called the plateau region. If compressive loading continues, all cells will collapse and the cell cavities are removed, transforming the material to function as a solid body, which will dramatically increase the stress. This region is called densification (BODIG, 1965; KENNEDY, 1968; TABARSA, 1999; GIBSON, 2005). The domain of these regions and their starting and ending points depend on the type and microstructural features of the material.

The mechanical behavior of a cellular structure, like bamboo and wood, can be modelled by different methods during a compressive force. The unit cell method considers the geometry of each cell structure in two or three dimensions. The method of dimensional analysis, on the other hand, relies on the correlation between the relative density and mechanical properties, which can be an easier and more accurate approach (GIBSON; ASHBY, 1997; GIBSON, 2005). Simulation of the material using finite element analysis (FEA) is also another method, which, considering the geometry and local effects, can be very accurate, and in some cases requires intense computations (GIBSON, 2005).

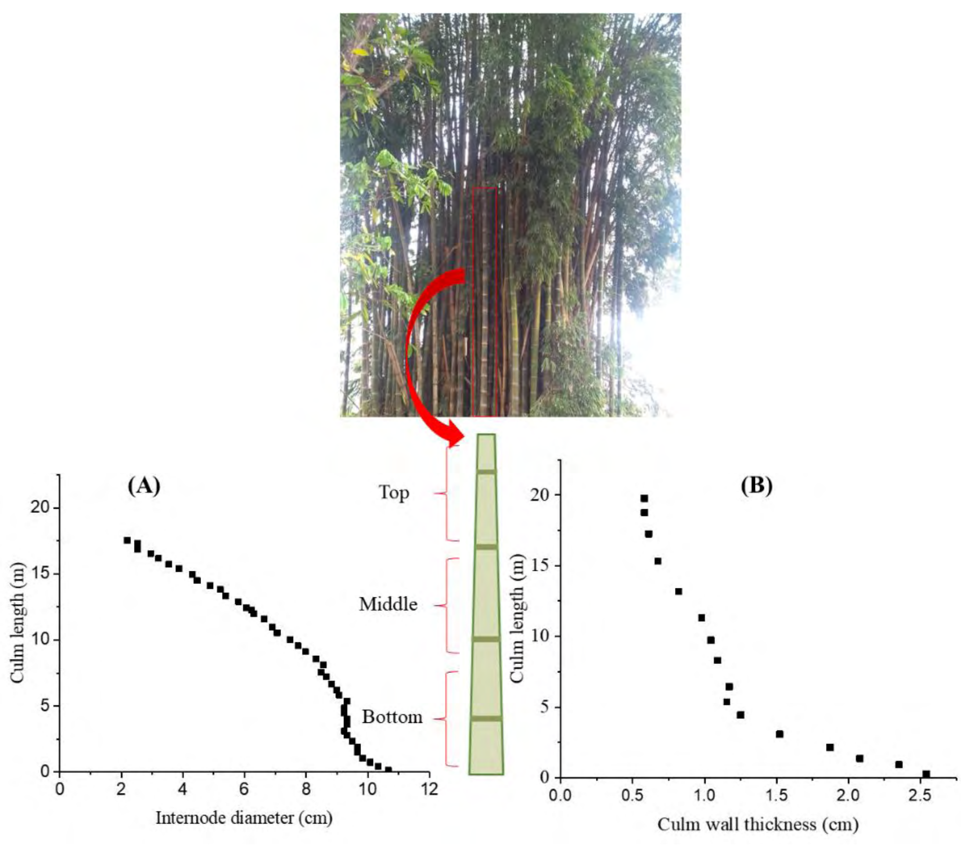
On the other hand, impregnating the material with additives is the other mechanism to achieve a greater weight and thereupon density. It is possible to fill the lumens and pores of a lignocellulose material with a suitable substance such as resins. This process causes the formation of new composite material with different physical, chemical, and mechanical properties. In some cases, both mechanisms may occur at the same time in some processes, like in the production of compressed wood-polymer composites (WOLCOTT, 2003) and bamboo scrimber (SHANGGUAN et al., 2016; HUANG; JI; YU, 2019a; ZHAO et al., 2019). The final density is controlled by the densification method. In addition, the physical, mechanical, and chemical properties of the raw materials are the key features. Porosity, for example, greatly affects the subsequent processing and mechanical properties of the final product.

This paper is focused on the first densification mechanism in which the volume of the material is changed using compression at elevated temperatures. The increase in the density of wood by compression at high temperatures has been done for at least a century, exploring different wood species (ZHONG et al., 2014). It worth mentioning that for lignocellulose materials like wood, when the utilized procedure involves high temperatures, not only does the wood structure change, but chemical modifications may also occur during the process, which affects wood properties. Bamboo, as a lignocellulosic material, follows the same model. However, due to the lack of sufficient data in this field, there is a gap in understanding the factors and the impact on the densification process. Further research is needed to quantify and predict the behavior of bamboo during the densification process accurately.

2.3. Bamboo

Bamboos are fast-growing woody grass plants that are subdivided into different families addressed in several reports (QISHENG; SHENXUE; YONGYU, 1980; LIESE, 1998), with more than 1200 species found globally. It is composed of the rhizome, which is buried underground, and the culm, which grows aboveground (QISHENG; SHENXUE; YONGYU, 1980). The part that is being used in the industry for the engineered bamboo production is the culm, which contains the woody material, formed with nodes and internodes. The internodes have a hollow cylindrical shape, while the nodes are thin diaphragms separating the internodes. The geometry of the culm is defined by the diameter, wall thickness, and internode length, which vary along with the height. Chaowana et al. (2017) (CHAOWANA; BARBU, 2017) showed this transfiguration of the bamboo culm and alteration of the macroscopic characteristics for four different bamboo species. From their results, it can be inferred that the middle part of a bamboo culm is more homogeneous than the upper and bottom parts (Figure 2.1). The difference in the structure of these three parts, top, middle, and bottom, leads to dissimilarity in physical and mechanical properties. Thus, bamboo manufacturing primarily utilizes the bottom and middle thirds of a culm.

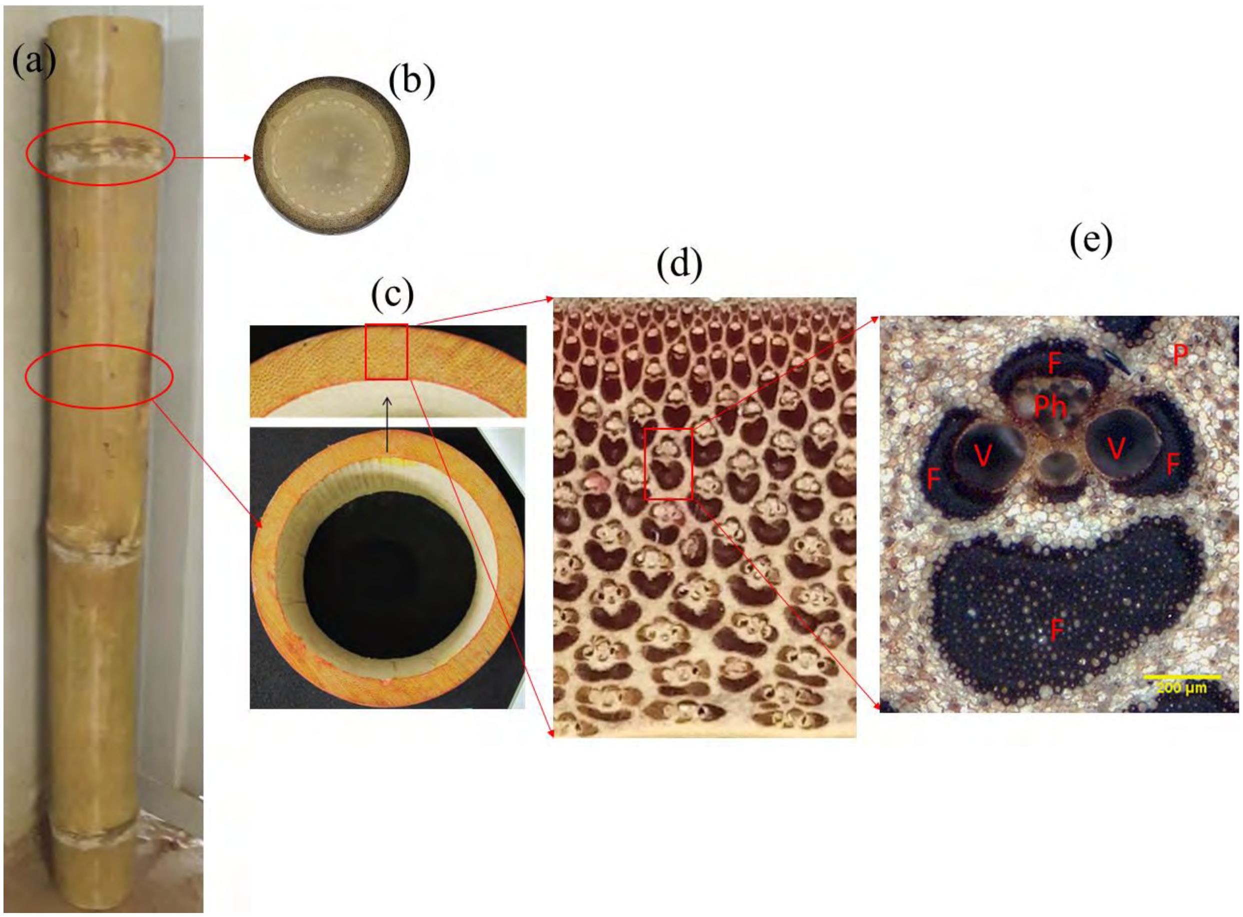
Figure 2.1 - The variation of macroscopic characteristics along the culm length, an example of the bamboo species *Dendrocalamus asper*, (**A**) internode diameter and (**B**) culm wall thickness



Source: (KADIVAR et al., 2020a)

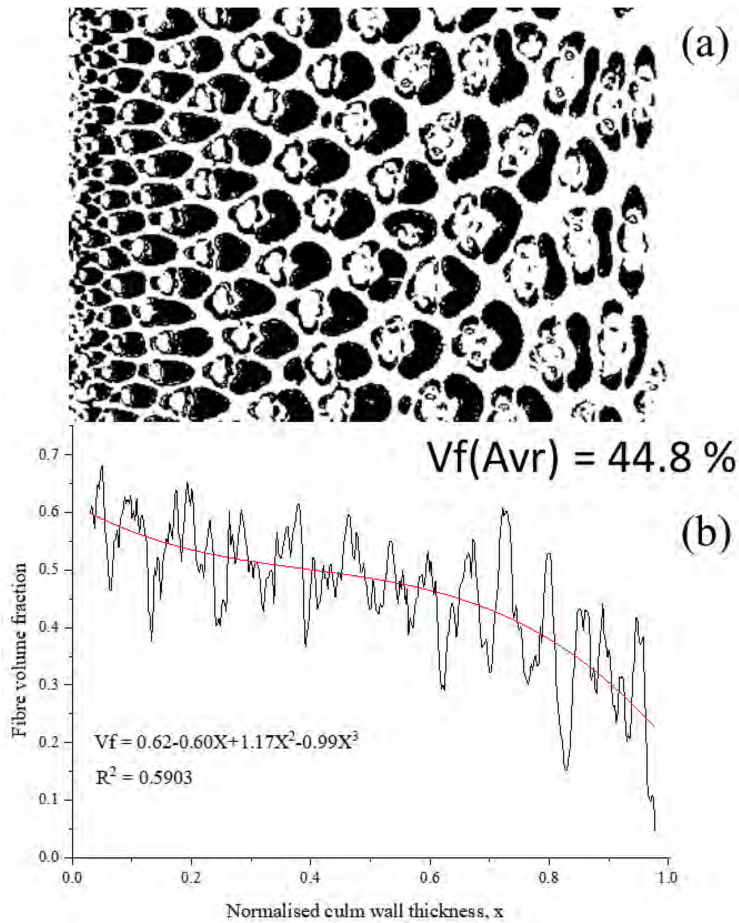
Anatomically, bamboo consists of fiber bundles (40%), parenchymal cells (50%), and vessels (10%), as shown in Figure 2 (LI et al., 1994; LIESE, 1998; AKINBADE et al., 2019). The mechanical properties of bamboo are correlated to its structure and fiber orientation. The fibers reinforce and support the matrix, which is composed of parenchyma. Therefore, at a macro scale, it is possible to consider bamboo as a uni-directional natural fiber reinforced composite. In contrast, at the meso-level, it can be seen through the bamboo culm wall thickness, as shown in Figure 3, that the fiber distribution is not homogenous. The density of fibers is functionally graded, increasing from inner to the outer region of the wall (RAY et al., 2005; CHAOWANA; BARBU; FRÜHWALD, 2015; AKINBADE et al., 2019). In addition, the fiber concentration is higher in the upper half of the culm than the bottom (LIESE, 1998; L.OSORIO et al., 2018). The lack of homogeneity across the bamboo thickness is one of the negative points of using bamboo culm directly in the construction industry.

Figure 2.2 - Overview of the morphological characteristic of bamboo culm. (a) Part of bamboo culm; (b) Cross section of bamboo showing node diaphragm; (c) Cross section of internode; (d) Section through culm wall; (e) Vascular bundle, V—Vessel, F—Fiber, Ph—Phloem, P—Parenchyma..



Source: (KADIVAR et al., 2020a)

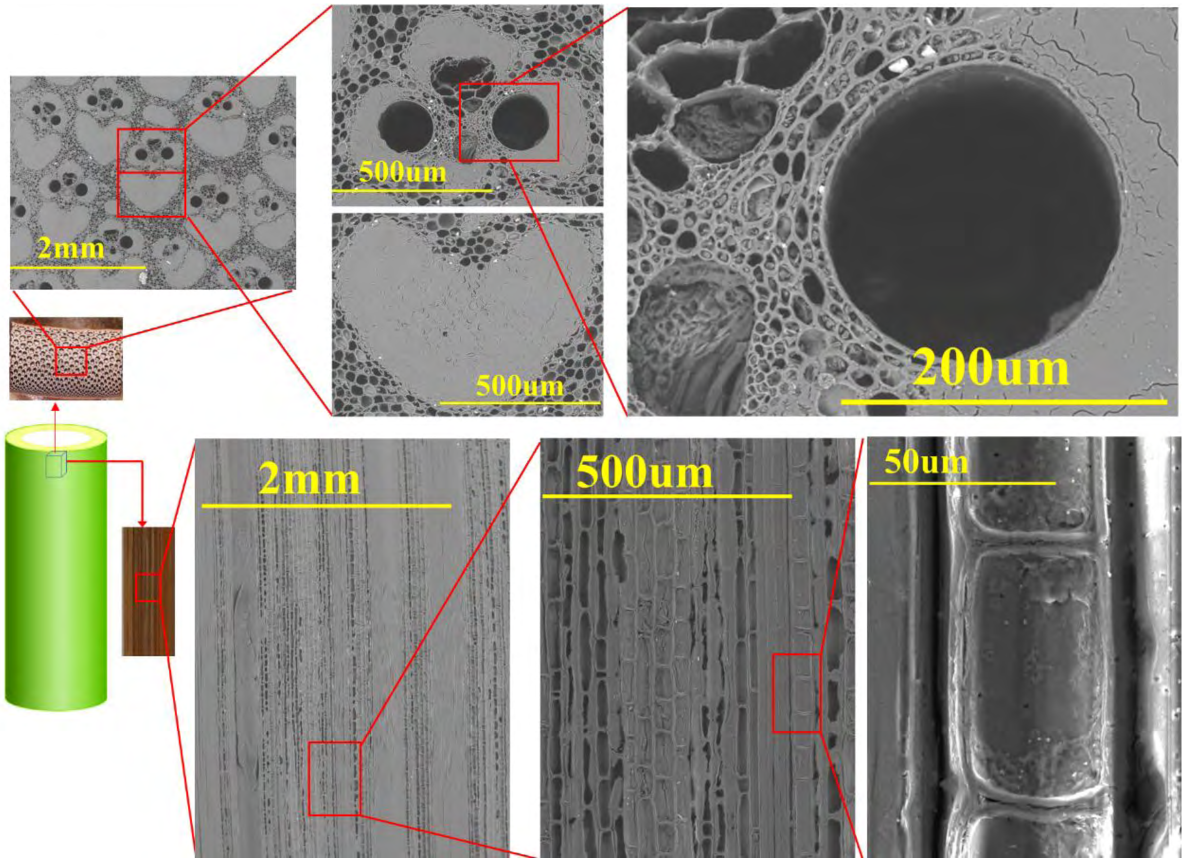
Figure 2.3 –Fiber distribution in the composite bamboo (*D. Asper*). (**a**) example of digital image analysis; (**b**) fiber volume distribution across the culm wall..



Source: (KADIVAR et al., 2020a)

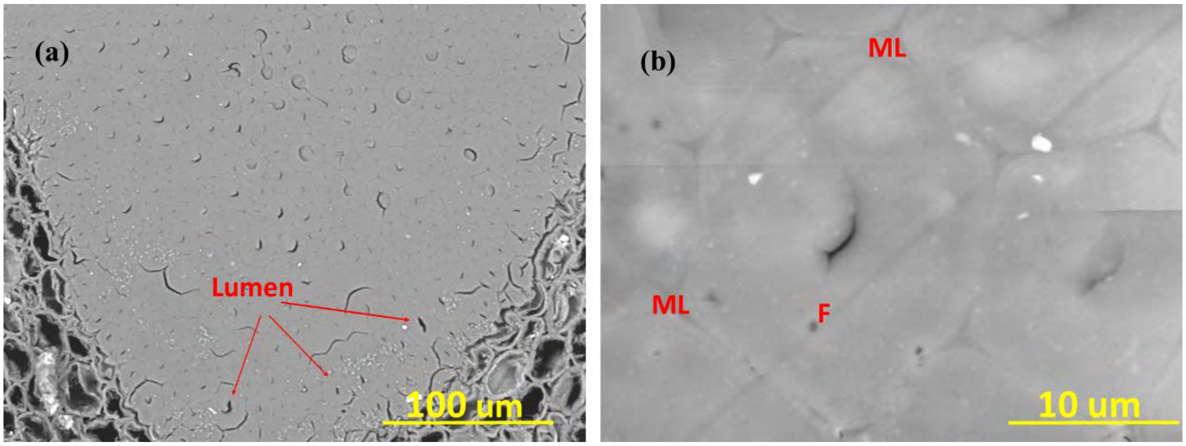
The oven-dried density of bamboo is around 400–900 kg/m3 (LIESE; KOHL, 1980) and varies by the species, age, growing conditions, and location along the length of the pole. There are many cavities within the bamboo vascular bundles, as presented in Figure 4. In addition, in bamboo fibers, which are bundles of elementary filaments bonded together by the middle lamellae (GOH; YAP; TONG, 2019) (Figure 5), the proportion of lumen tends to be higher towards the fiber bundle periphery. Therefore, theoretically, it is possible to compress bamboo close to a density of the cell wall density, which is approximately 1500 kg/m3 (DIXON; GIBSON, 2014). This compression could be in the radial or tangential direction to close the vessels, and the required force depends on the strength of the cell walls to buckle and varies by the species.

Figure 2.4 - Details of the microstructure of the bamboo *D.asper*..



Source: (KADIVAR et al., 2020a)

Figure 2.5 - **(a)** Bamboo fiber bundle, **(b)** Elementary bamboo fibers **(F)** bonded by middle lamellae (**ML**).



Source: (KADIVAR et al., 2020a)

These features of bamboo, as well as the variability of the geometric properties, have prevented the industry from considering raw bamboo as a mainstream building material. To address these limitations, the industry has shifted to manufacturing bamboo-based panels that are comparable to wood and engineered wood board products.

2.4. Densification in Bamboo-Based Panels

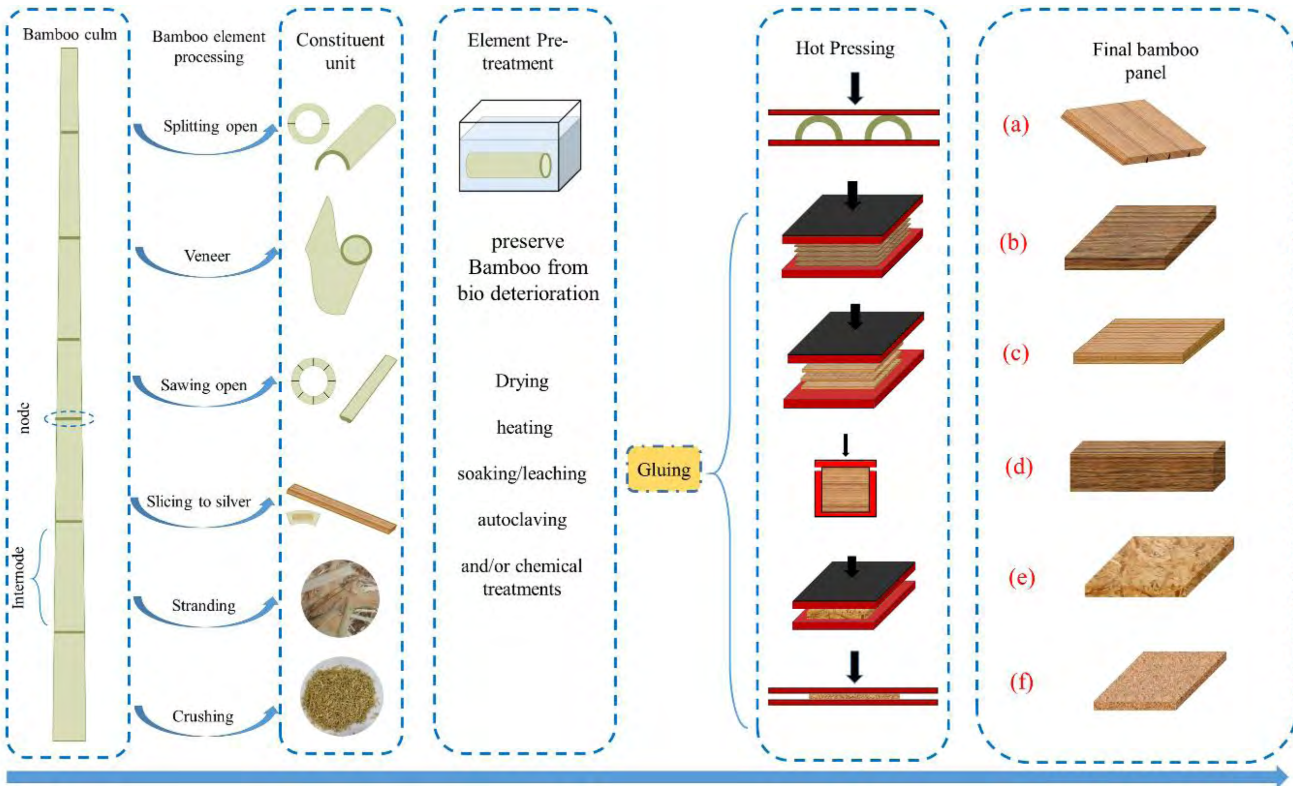
Bamboo based panels (BBPs) are widely applied in the field of construction, in surface applications (e.g., floors, ceilings, and wall finishes), as well as used in the furniture industry. BBPs are categorized and nominated based on the terminology standard. Referring to the Chinese standard "LY/T 1660-2006 Standard terminology for bamboo-based panels" (AKBEN, 2006), Liu et al. (2016) (LIU et al., 2016), and Huang (2019) (HUANG, 2019), standard BBP products are labeled as follows:

a) Flattened bamboo panel, formed by flattening the bamboo culm (FANG et al., 2018a).
b) Bamboo laminated lumber, constituted of bamboo strips (MAHDAVI et al., 2011).
c) Plybamboo, made of bamboo slivers, or bamboo mats, and curtains obtained by weaving (ANWAR, U.; HIZIROGLU, S.; HAMDAN, H.; LATIF, 2011).

d) Bamboo scrimber, by gluing the bamboo fiber bundle (QISHENG, Z.; SHENXUE, J.; HAI, L.; XUFENG, X.; HONGZHENG, L.; BIN, X.; WEN, 2009).
e) Bamboo particleboard, by molding the fine bamboo particles (BISWAS, D.; BOSE, S.K.; HOSSAIN, 2011; GAUSS et al., 2019).
f) Bamboo oriented strand board, by molding the oriented strand elements (SUMARDI, I.; ONO, K.; SUZUKI, 2007; TIANXIANG, 2008; MALANIT; BARBU; FRÜHWALD, 2011).

In the classified products, the flattened board is obtained from circular bamboo culm without using resin, while the others are developed by a "decomposition" and "recombination" process (HUANG; SUN; MUSSO, 2017b). The production processes of these bamboo-based panels are summarized in Figure 6. During decomposition, the bamboo culm is broken down into small segments (engineered bamboo elements) such as veneers, strips, slivers, strands, and particles. Through the use of resin and hot press, it is possible to recombine the small elements and produce a board.

Figure 2.6 - Summary of bamboo-based panel production processes. ((a) Flattened bamboo panel, (b) Bamboo laminated lumber, (c) Plybamboo, (d) Bamboo scrimber, (e) Bamboo oriented strand board, (f) Bamboo particleboard).



Source: (KADIVAR et al., 2020a)

It is possible to classify the bamboo elements required for the six mentioned bamboo-based panel products based on the geometry of the elements. This classification divides bamboo units into six major groups (Table 1). As shown in Figure 6, hot-pressing is a significant step for the manufacture of bamboo products in which bamboo elements bond together and might undergo compaction and increase their density. Among the BBPs mentioned, densification is more likely to occur in the flattened bamboo panels and bamboo scrimber, which alter the cell structures and improve the weak tissues.

Table 2.1 - The classification of bamboo elements used in densified products (specifically bamboo based panels (BBPs)).

Bamboo Element	Production Method	Size Range (mm)	Description	References
Half-split Culm (Half-Round Bamboo) 	Dividing a culm into two equal splits and removing the nodes.	The size of half-split culm depends on the original culm dimension	It has been used for a variety of traditional applications such as roof tiles and drainage ducts.	(LIESE; TANG, 2015a; FANG et al., 2018a)
Bamboo Veneer [51] 	Rotary cutting of bamboo culms	T =0.15–1.5 W = varied L = varied	A sheet-like folio which has the biggest bamboo industrial element size.	("ZAFU. LY/T 2222-2013 Sliced Bamboo Veneers, Zhejiang Agriculture and Forestry University, Standards Press China, Beijing.", 2014; LIESE; TANG, 2015a; LIU et al., 2016)
Bamboo Strip 	Cutting bamboo culm in longitudinal direction or by flattening half splits	T = 3–10 W = 15–25 L = varied	It is a long, thin piece of culm with uniform thickness and width.	("CAF. GB/T 265365-2011 Bamboo Strip, Chinese Academy of Forestry, Research Insititute of Wood Industry, Standards Press China, Beijing.", 2011; SHARMA et al., 2015a; LIU et al., 2016; FANG et al., 2018a)
 Bamboo Sliver	Cutting process Slicing to sliver	T = 0.5–3.5 W = 10–30 L = varied	Are also long bamboo elements consisting of flat surfaces, thinner than strips.	(QISHENG Z, SHENXUE J, 2002; CHAOWANA; BARBU, 2017)
Bamboo Strands	Produced by disk flaker	T: 0.3–0.8 W: 5–20 L: 50–90	Are consisted of flat, long bundles of fibers having parallel surfaces and	(HIDALGO-LÓPEZ, 2003; SUMARDI, I.; ONO, K.; SUZUKI, 2007; MALANIT; BARBU;

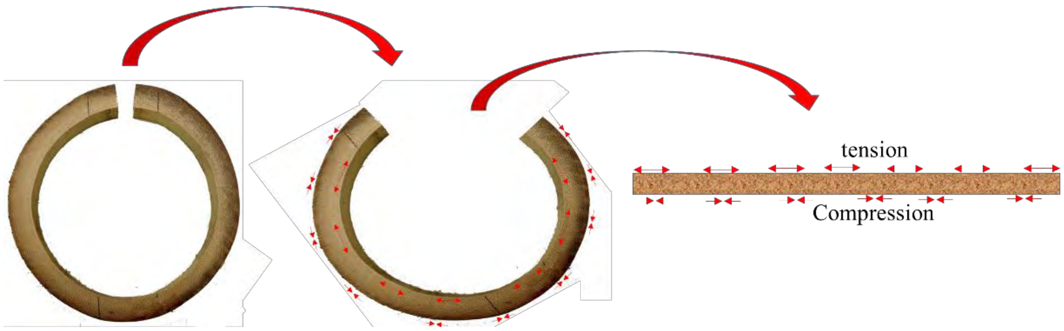
			short slivers of bamboo.	FRÜHWALD, 2011; LIU et al., 2016; CHAOWANA; BARBU, 2017; CHEN et al., 2018a)
Bamboo Particles 	Milling	T: 0.1–0.5 W: 1–5 L: 20–30		(WIDYORINI et al., 2014; ZAIA, 2015; LIU et al., 2016)

Source: (KADIVAR et al., 2020a)

2.4.1. Densification in Flattened Bamboo Panels

In the available literature on bamboo, there are generally three concepts of bamboo geometry modification: reformed bamboo, flattening, and densification. Reformed bamboo is a term which only exists in the literature from 1990s until 2003 and was a combination of flattening and densification (LI et al., 1994; ZENG et al., 1995; LI; LIU; YU, 2002). The concept of reformed bamboo changed after 2003 and divided into two different mechanisms of deformation: flattening and densification (LI et al., 1994; ZENG et al., 1995; LI; LIU; YU, 2002; TANAKA et al., 2006; TAKAGI et al., 2008; LIU et al., 2013; ARCHILA-SANTOS; ANSELL; WALKER, 2014; CHEN et al., 2014; KOREA, 2015; DIXON et al., 2016a; FANG et al., 2018a; GAO; GUO; LUO, 2018; ZHANG et al., 2019a; KADIVAR et al., 2019a). Bamboo flattening is a technique that softens and flattens the tubular bamboo culm directly into a flat board and to achieve that several different processes and types of equipment have been tested (LIU et al., 2013; KOREA, 2015; FANG et al., 2018a; ZHANG et al., 2019a). The method has some disadvantages, including the development of deep cracks during flattening. The mechanism of flattening, which is presented in Figure 7, occurs with the extension of the inner zone (tension) and contraction (compression) of the outer zone, along the tangential direction.

Figure 2.7 - The mechanism of flattening.

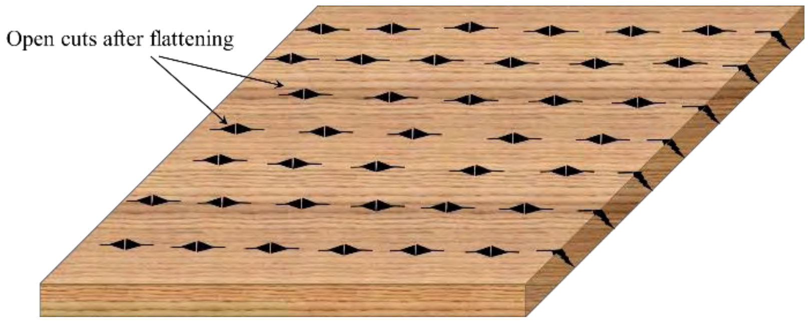


Source: (KADIVAR et al., 2020a).

Fang et al. (2018), (FANG et al., 2018a) reviewed the flattening methods and suggested two possible solutions to prevent the development of cracks. The first suggested solution is reducing the circumference differentiation of the internal and external layers, which is possible by extracting the skin or splitting the culm into several pieces or executing superficial scratches on the inner surface. The second method is to decrease the tangential stress by chemical treatment, heat treatment, or/and increasing moisture content. The combination of these two solutions, modification of the geometry and material treatment, can be more effective. Employing microwave heating (M. MAORI, (HYOGO-KEN. INDUSTRIAL RESEARCH INST., 1987) using chemical reagents (Z. QISHENG, 1988), hot oil immersion (T. PARKKEEREE, N. MATAN, 2015), and applying high-pressure steam (S.N.SHIMPEI; YOSHITANI; G.U, 2012; ZHANG et al., 2019a) are successful approaches for softening the half-split bamboo culm and flattening to non-cracked bamboo board. There are also several investigations concentrated on the technological factors and tools to facilitate the flattening process by counteracting the stress of the bamboo material, mainly focused on the internal culm wall during deformation (LIU et al., 2013).

Densification is one of the improvements that has been suggested as a post-treatment of flattened bamboo culms to maintain the new geometry and to reduce indentations. In the case of using the method of superficial scratches on the inner layer of bamboo, after just the flattening process, the small cuts will open (Figure 8) because of tension in this region. Therefore, densification will be required to fill up the grooves.

Figure 2.8 - Scratches on the internal layer of flattened bamboo.



Source: (KADIVAR et al., 2020a)

2.4.2. Densification in Bamboo Scrimber

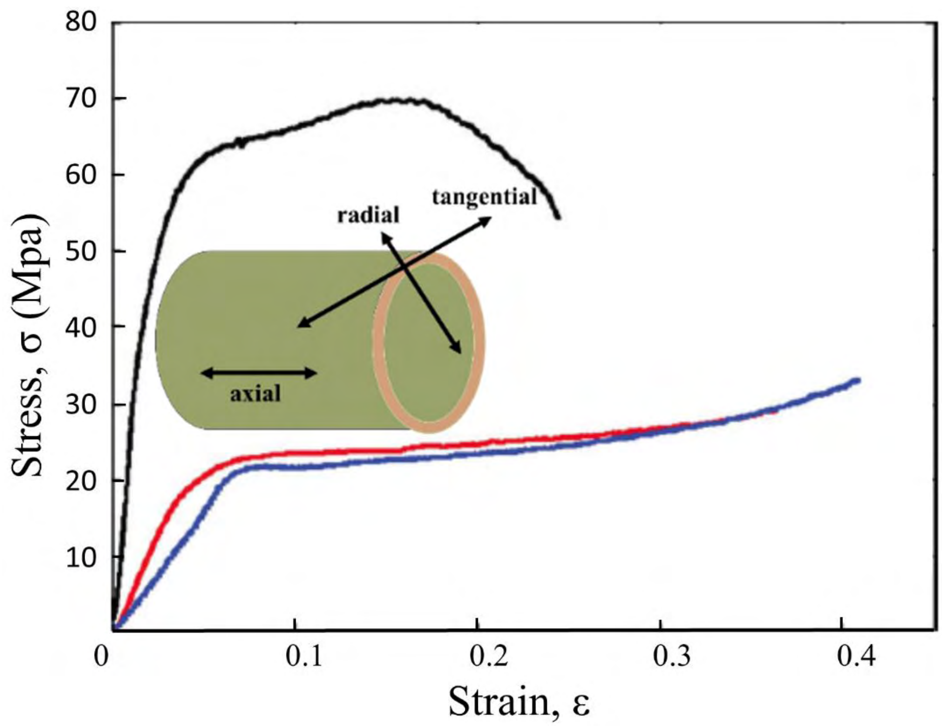
Bamboo scrimber, also known as reconstituted densified bamboo (KUMAR et al., 2016), utilizes fiber bundles dipped in phenol-formaldehyde resin, which is then compressed in a mold and heated to cross-link the resin (SHARMA et al., 2015b). Due to its excellent physical and mechanical performance compared to natural bamboo (SHARMA et al., 2015a; ZHU; ZHANG; YU, 2015; YU et al., 2017a), scrimber has attracted the attention of the bamboo industry in the 2000s (HUANG; SUN; MUSSO, 2017b). However, the quality of this engineered bamboo product depends on the bamboo species, media treatment, type and content of the resin, as well as the pressing parameters (KIM Y, ROH J, 2003; J. XIE et al., 2016; SHANGGUAN et al., 2016; LI et al., 2018), which are also related to the density. The density of a bamboo scrimber can be as high as 1050–1250 kg/m3 (KUMAR et al., 2016), which is almost twice the density of raw bamboo. Physical and mechanical properties of scrimber depend on the density. The higher the density, the higher the mechanical properties, and the lower the water absorption (J. XIE et al., 2016; KUMAR et al., 2016; RAO F, CHEN Y, LI N, ZHAO X, BAO Y, WU Z, REN D, XU J, 2017).

The increase of density may be related to the closure of bamboo voids such as vessels, parenchymas, and fiber lumens caused by high-pressure and hot-pressing processes (YU et al., 2017a; HUANG; JI; YU, 2019b), or the presence of solid resin particles in the cavities. The microstructural analysis of engineered bamboo scrimber at different densities conducted by Yu (2017) (YU et al., 2017a), shows that when the scrimber density is low, for example at 850 kg/m3, the structure is irregular, which led to deficient fiber bonding. Some of the big vessels are open, and just a few parenchyma cells are compacted. By increasing the density of bamboo scrimber to 1300 kg/m3, the structure gets more compact and the fibers bond more effectively. On the other hand, part of this increase in density is due to the addition of the resin.

2.5. Densified Bamboo

Densified bamboo utilizes the full-culm (or strips with whole thickness of bamboo) with the fiber bundles maintained in the parenchyma matrix, which is then compressed to densify the culm wall. This product can be used as a single lamina or be laminated into a multiply board with or without adhesive. The main aim of the bamboo densification is to decrease its heterogeneity and improve its mechanical properties since, in raw bamboo, most of the characteristics change through the bamboo transversal section. The densification process can change the distribution of fibers through the bamboo cross-section and, consequently, homogenize the distribution of physical and mechanical properties. The method can be achieved by pressing bamboo perpendicular to fibers, which can be transversal or radial direction (the directions are shown in Figure 9). Flattening bamboo results in compression in both transverse and radial directions. However, in this study, the densification of the culm wall is only considered in the radial direction.

Figure 2.9 - Typical compressive stress-strain curves of bamboo (adapted from (DIXON; GIBSON, 2014)).



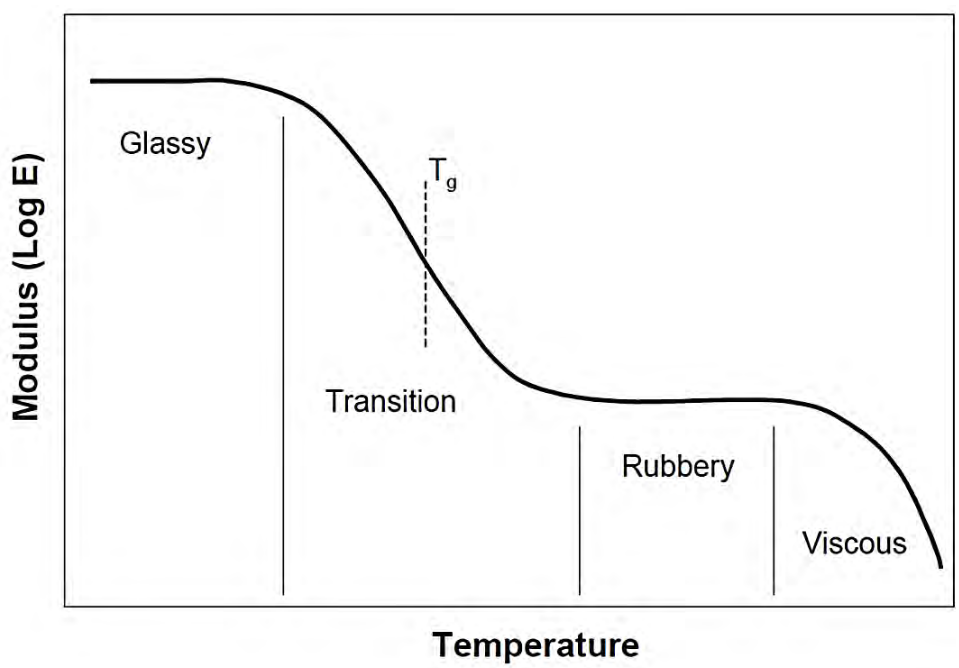
Source: (KADIVAR et al., 2020a)

To densify bamboo in the radial direction, the applied force should overcome the compressive strength of bamboo to buckle the cells and pass the elastic region. Typical compressive stress-strain curves of Phyllostachys edulis (Moso) bamboo are shown in Figure 9 (red line). Although the compression test had been carried out at room temperature, it is still possible to understand that using more than 30 MPa of pressure can cause a deformation higher than 40%. Elevated temperatures and using the same pressure can lead to a higher densification degree (DD). To facilitate bamboo deformation (for both flattening and densification mechanisms), its viscoelastic behavior must be considered. Similar to other viscoelastic materials, temperature and glass transition temperature (Tg) are the most significant parameters for geometric deformation.

2.5.1. Softening Behavior of Bamboo

Bamboo contains natural polymers based on hemicellulose, lignin, and non-crystalline cellulose components, which are amorphous phases. Therefore, it is considered a viscoelastic material (AMADA, S. AND LAKES, 1997; W.G. GLASSER et al., 1998; MATAN; KYOKONG; PREECHATIWONG, 2007). The mechanical behavior of bamboo is between linear elastic solids and viscous fluids, similar to other viscoelastic materials such as polymers and wood. Figure 10 presents the different stages of amorphous polymers and the resulting behavior with increasing temperature. In this figure, the Tg limit, or the softening temperature stage, describes the softening characteristics of amorphous polymers in which many qualities of the material change sharply. For example, the molecular movement and damping properties increase while the strength and elastic modulus fall dramatically (M. P. WOLCOTT, F. A. KAMKE, 1990; SKYBA, 2008; T. ZHAN, J. LU, H. ZHANG, J. JIANG, H. PENG, 2017; WANG et al., 2018). At temperatures below Tg, bamboo behavior is glassy, while at higher temperatures, it behaves like a rubbery or viscous material (C.A.LENTH, 1999).

Figure 2.10 - Variation of relaxation modulus with temperature for an amorphous polymer (C.A.LENTH, 1999).

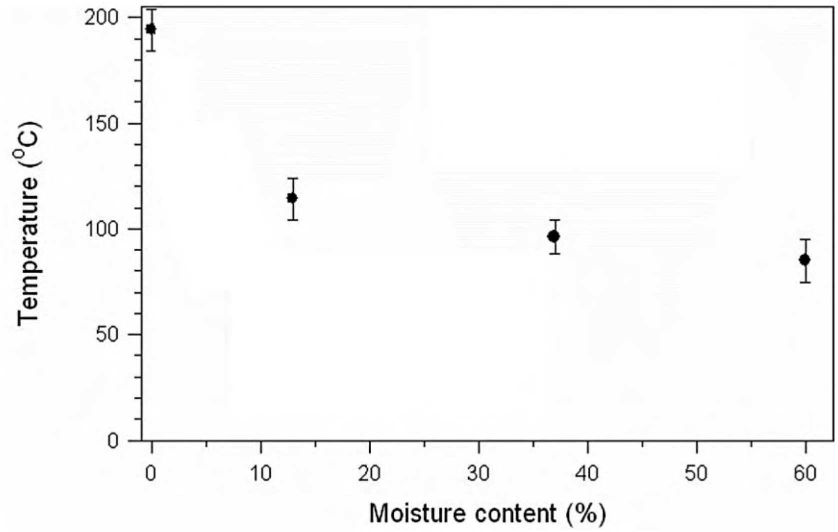


Source: Adapted from (C.A.LENTH, 1999).

The glass transition in these viscoelastic materials is dependent on the moisture content, chemical composition, and testing method. For wood, as an example, the Tg ranges from 60 °C to 235 °C (C.A.LENTH, 1999). Higher moisture contents lead to a decrease in the glass transition temperature. As presented in Figure 10, the increase in temperature makes the amorphous material constituents to display viscous behavior. However, this stage is not reached in wood before the start of thermal degradation, and the amorphous wood components will degrade at elevated temperatures (M. P. WOLCOTT, F. A. KAMKE, 1990; MATAN; KYOKONG; PREECHATIWONG, 2007). Bamboo also experiences similar stages as wood when exposed to elevated temperatures, and the mechanical properties are also dependent on temperature and moisture.

In relative terms, at short durations, low temperatures, and low moisture content, amorphous bamboo constituents stay in a "glassy state" and exhibit high strength and modulus. By increasing the temperature, moisture content, and duration, bamboo exhibits a rubbery behavior, which makes it easier to be deformed. Therefore, the softening stage needs to be considered in any geometry deformation mechanism of bamboo. Matan et al. (2007) (MATAN; KYOKONG; PREECHATIWONG, 2007) showed the glass transition temperature dependence on the bamboo's initial moisture content for Dendrocalamus asper bamboo (Figure 11). According to their study, the Tg of bamboo approaches a constant value with initial moisture contents above 13% (between 100 and 120 °C) (MATAN; KYOKONG; PREECHATIWONG, 2007).

Figure 2.11 - The moisture dependence of the glass transition (Tg) for bamboo (data from (MATAN; KYOKONG; PREECHATIWONG, 2007)).



Source: (KADIVAR et al., 2020a)

2.5.2. Densification Methods

Selection of the right method for densification requires accurate knowledge of the material, such as its anatomical characteristics, softening behavior, and also loading direction. Applying an improper method, using solely compression, for example, can cause problems in the final product. Spring back effect, which occurs when the sample does not maintain the targeted thickness after decompression, is one of the main problems associated with the densification process. It can be eliminated by using an appropriate method. An appropriate method is a process in which the optimized parameters apply to the material. Several methods that utilize steaming or heating, which can induce permanent fixation of the compressive deformation, have been used in the wood and bamboo industry (KADIVAR et al., ; BOONSTRA; BLOMBERG, 2007; KUTNAR; ŠERNEK, 2007; L, 2012; DIXON et al., 2016b; ESTEVES, 2017; ZHANG et al., 2019b). The best densification method is the one in which plasticization of the material and stabilization of the final product is adequately taken into account.

There are approximately 1000 patents on bamboo densification, using different methods, mostly from China and Japan (TAKAO FUJIKAWA, 2008; FANG CHANGHUA ALAN KRUTIYE JIANG ZEHUI FEI BENHUA SUN ZHENGJUN, 2017). However, less than ten published studies of bamboo culm densification are published to date (presented in Tables 2 and

3). These studies focused on thermo-mechanical (TM) method, which is conducted in an open system using heat and pressure, and viscoelastic-thermal-compression (VTC) process approach, using heat and pressure and pre-softening with steam in a closed system.

Table 2.2 - Bamboo densification process parameters.

Reference	Bamboo species	Size of samples (mm)	Temperature (°C)	Pressing time (min)	Method of press	Pressure MPa	Densification degree (DD) (%)
(TANAKA et al., 2006)	*Phyllostachys edulis*	I + M + O	120	-	Thermo-hydro-mechanical (THM)	8	-
	P. bambusoides	I + M + O					
(TAKAGI et al., 2008)	*P. bambusoides*	10 × 200 I + M + O	25–220	10	Thermo-mechanical (TM)	50	-
(K.E. SEMPLE, F.A. KAMKE, A. KUTNAR, 2013)	*P. edulis*	5 × 20 × 150 M + I	170	13.3	Viscoelastic-thermal-compression (VTC) (steam pressure 775 kPa)		20–67
(ARCHILA-SANTOS; ANSELL; WALKER, 2014)	*Guadua angustifolia*	M	150	20	TM	6.2	42.51
(DIXON et al., 2016a)	*P. edulis*	90 ×20 × (3–5)	170	13.3	VTC (steam pressure 775 kPa)	-	50
(KADIVAR et al., 2019a)	*Dendrocalamus asper*	I+M+O	140	20	TM	4.34	31.2

I: inner layer, M: middle layer, O: outer layer, DD: Densification Degree.

Source: (KADIVAR et al., 2020a)

Table 2.3 – Summary of the mechanical and physical properties of bamboo, before and after densification process.

Reference	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
(TANAKA et al., 2006)	*P. edulis*	JIS Z2101. (1994) (JIS Z 2101, 1994)	680	1334	8.5–36.5	-	12.0 (T)	32.0 (T)	80 (T)	310 (T)
	P. bambusoides		600	1100		-	8.5 (T)	19.0 (T)	170 (T)	220 (T)
(TAKAGI et al., 2008) (*P. bambusoides*)	25 °C	(10*100*(3-5)) mm	714	1000	5 ± 3	-	6 (B)	8.5 (B)	117 (B)	160 (B)
	160 °C			1280				20.0 (B)		320 (B)
	220 °C			1380				27.0 (B)		190 (B)
(K.E. SEMPLE, F.A. KAMKE, A. KUTNAR, 2013) (*P. edulis*)	80% C. R	ASTM D1037-06a (2006) (AMERICAN SOCIETY FOR TESTING AND MATERIALS INTERNATIONAL, 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)
	33% C. R			1261		7.7		13.6 (B)		219 (B)
(ARCHILA-SANTOS; ANSELL; WALKER, 2014) (*G. angustifolia*)	dry	ISO 22157 (2004) (STANDARDIZATION, 2008; ISO-INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, 2009)	540 (Oven dried)	810 (OD)	-	-	16.2 (T)	22.8 (T)	-	-
	Pre-soaked			830 (OD)	-	-		31.0 (T)		-
(DIXON et al., 2016a) (*P. edulis*)	-	70 × 5 × (1–3) mm	450 to 600	800 to 1200	7	5	2.5–12.1 (B)	5.4–23.0 (B)	47–140 (B)	74–296 (B)
(KADIVAR et al., 2019a) (*D.Asper*)	0% MC	ASTM D7264—15 (2015) (D7264-15, 2015)	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: Test in tensile, B: Test in bending, OD: Oven dried, JIS Z2101: Japanese Standards Association, ASTM: American Society for Testing and Materials International, ISO: International Organization for Standardization

Source: Source: (KADIVAR et al., 2020a)

2.5.2.1. Thermo-Mechanical (TM)

In thermo-mechanical (TM) densification, an open system with temperature and pressure is used to densify the material. TM operation processes have been applied between 140–200 °C, at 40%, 50%, and 60% compression rates using different moisture contents (KUTNAR; ŠERNEK, 2007). TM densification is usually performed in open systems (e.g., hot-press) in which controlling the sample moisture content (MC) or the relative humidity (RH) of the environment during the process is not possible. Therefore, in this method, special attention should be paid to the initial MC; low MC makes it difficult for the material to be densified, and high MC can cause an explosion. Stabilizing wood at 13% MC (HEGER et al., 2004), and bamboo at 10% (KADIVAR et al., 2019a) is suggested to be enough to facilitate the process. Some commercial products developed during the 1950s were produced through this method (R.M.SEBORG; M.A.MILLETT; A.J.STAMM, 1945).

Research has shown that both wood and bamboo materials exposed to TM densification have higher bending resistance and modulus of elasticity than the natural one (KADIVAR et al., ; TABARSA, T.; CHUI, 1997). The influence of effective parameters involved in TM process on the final densified material has been studied (GONG, M. AND LAMASON, 2007; DUBEY, 2010). The reason for increasing the strength after TM densification process is related to the decrease in porosity and the increase in density (ULKER; IMIRZI; BURDURLU, 2012). However, at low temperatures and high compression rates, cellular cracks occur more often, which may decrease strength (TABARSA, T.; CHUI, 1997).

2.5.2.2. Viscoelastic-Thermal-Compression

Densification of bamboo by viscoelastic-thermal-compression (VTC) is similar to that of wood, and includes the following manufacturing steps (KUTNAR; ŠERNEK, 2007):

1) Elevating the temperature to exceed its glass transition temperature.
2) Causing rapid steam decompression and removal of the bound water in the cell wall.
3) Densification by compressing the material perpendicular to the grain.
4) Relaxation of the remaining stresses. This step promotes the thermal degradation of the hemicelluloses in the material component.

5) Cooling the material by conditioning that to the ambient temperature and humidity.

The critical point in the VTC process is softening in a high-pressure vapor environment. Yet, the material must have some initial moisture content, which changes during the pressing process due to the steam pressure in the closed space. For example, the initial moisture of wood to be VTC processed is preferable to be around 15–30% (S.ŞENOL; M.BUDAKÇI, 2016). The desirable temperature and pressure levels depend on the initial density and species of the wood (S.ŞENOL; M.BUDAKÇI, 2016; SANDBERG; KUTNAR; MANTANIS, 2017). For low-density woods, the best temperature is between 160–175 °C and pressures of 650–2000 kPa (KUTNAR; ŠERNEK, 2007). However, for a higher density, a temperature of 175–225 °C with pressures of 2000–4000 kPa are required (KUTNAR; ŠERNEK, 2007). In the case of VTC process for bamboo, because of lack of information, the process parameters are not optimized yet. According to the available literature of wood and bamboo, the VTC densification method improves the mechanical properties of the material and enables dimensional stabilization (A. LENTH; A. KAMKE, 2001; S.ŞENOL; M.BUDAKÇI, 2016; YU et al., 2017b).

2.5.2.3. Mechanisms of deformation

The deformation in the reviewed densification methods is controlled by three mechanisms:

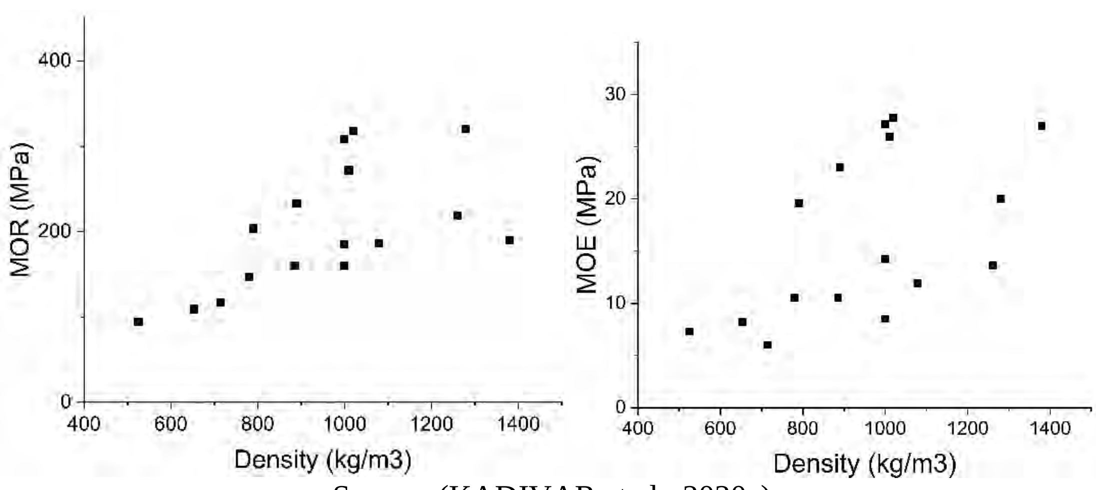
1) Purely mechanical densification, mainly from rearrangement of fiber bundles, brittle crushing, cell wall buckling, and subsequent moisture content drainage during the compaction.
2) Materials shrinkage and plastic yielding due to elevated temperature (at a temperature higher than 160 °C, the chemical changes also cause additional deformation).
3) Compaction resulting from intercell steam pressure, generally induced by stress and heat, which causes intercell cracks (even in TM method, the moisture content and elevated temperature can generate steam).

Therefore, it is possible to consider the VTC and TM as two types of THM (thermo-hydro-mechanical method), which are conducted in closed and open systems, respectively. There are other new densification methods such as thermo-vibro-mechanic (TVM), which is a new application using heat pressure and vibration (KUTNAR; ŠERNEK, 2007). However, very few studies applied this method to densify wood, and in the case of bamboo, there is no published literature at the time of writing.

2.6. Mechanical and Physical Performance of Densified Bamboo

The mechanical and physical qualities of densified bamboo subjected to the process parameters presented in Table 2 are summarized in Table 3. In general, three-point bending (B) and tensile tests (T) are used to evaluate the mechanical performance of densified bamboo. Based on the results in Table 3, without considering the effect of different species for both the natural and densified bamboo using different processing methods, the bending modulus of rupture (MOR) and modulus of elasticity (MOE) appear to be linearly correlated to the density (Figure 12). Dixon et al. (2016) (DIXON et al., 2016a) also showed the linear relationship of bending and density for natural and densified bamboo. The authors evaluated the bending characteristics in the longitudinal orientation for un-densified and THM densified bamboo (Moso species) and concluded the increase of MOE and MOR with densification. However, comparing the un-densified and densified bamboo with equal density, raw bamboo shows higher strength (DIXON et al., 2016a). Due to the small number of specimens tested in tension, it is not possible to indicate any correlation for the tensile strength of densified bamboo.

Figure 2.12 - Bending-density results of data presented in Table 3.



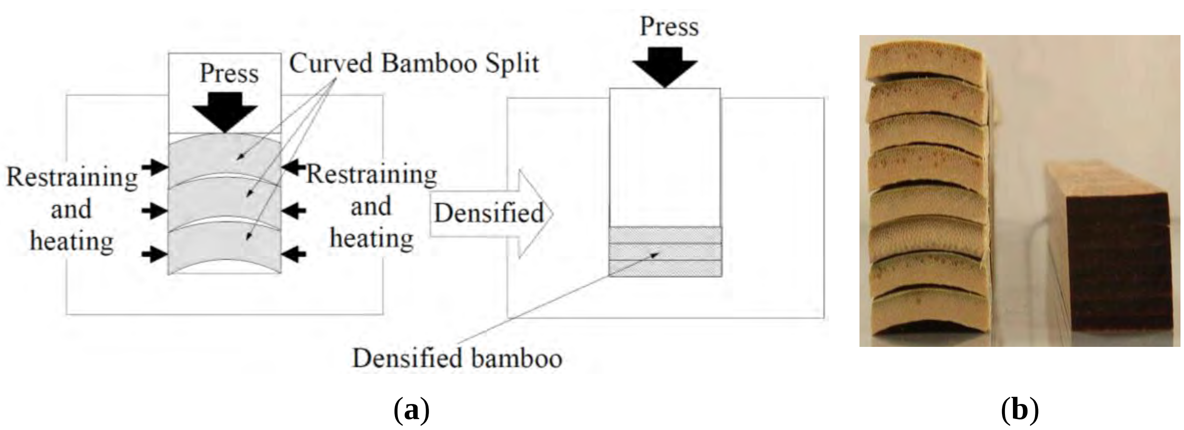
Source: (KADIVAR et al., 2020a)

2.7. Discussion of the Effective Parameters

2.7.1. The Influence of Bamboo Species

Bamboo species have varying physical and mechanical properties, related to the wall thickness, density, and fiber distribution. Accordingly, the densification mechanism varies by species. Tanaka et al. (2006) (TANAKA et al., 2006) adopted the densification method, which is presented in Figure 13, to produce the bamboo connector (for developing a new connecting system) using two different bamboo species, Moso and Phyllostachys bambusoides (Madake). To densify the material, the authors compressed sections of the bamboo culm wall using the press parameters shown in Table 2. As a result of this process, the density, tensile strength, and modulus increased. Although the values of tensile strength for the two bamboo species improved significantly by the densification process, the relative improvement was different. By using the same procedure, Moso tensile strength improved by 287.5% and reached 310 MPa. However, Madake tensile strength changed only by 29.41% from 170 MPa to 220 MPa (TANAKA et al., 2006). The initial density of the natural Madake is less than that of the natural Moso, and it is expected to result in a higher density after the same pressure. However, different initial compression strengths not explored in the study may be the cause of the lower density for Madake bamboo. Different fiber content can be a justification to explain the different relative improvement of these two bamboo species.

Figure 2.13 - (a) Densification process used by Tanaka et al. (2006) (TANAKA et al., 2006) and (b) bamboo samples before and after the densification process (TANAKA et al., 2006).



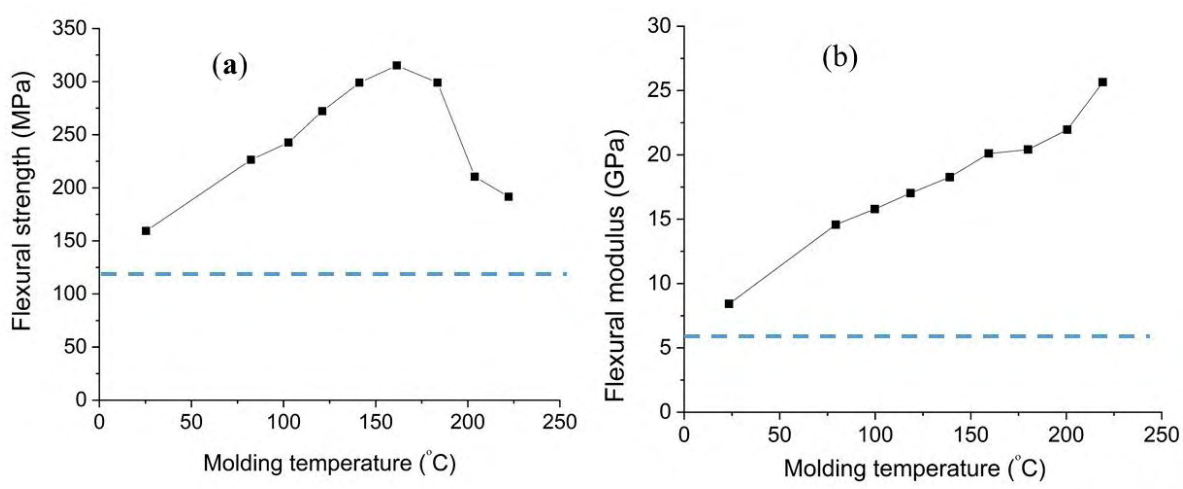
Source: (TANAKA et al., 2006)

2.7.2. The Influence of Pressing Temperature

As discussed in Section 5.1, an elevated temperature is needed in the bamboo densification process to facilitate deformation without generating a high incidence of cracks. There is the question of how much temperature is required and what the appropriate temperature range is for the bamboo press process. The answer to this question requires understanding the effect of temperature on bamboo material. The temperature must be high enough to make the bamboo plastic, but not too much to reduce mechanical strength and cause thermal degradation.

To evaluate the impact of pressing temperature on the mechanical strength of densified bamboo, Takagi et al. (2008) (TAKAGI et al., 2008) prepared bamboo strips without nodes and placed them in an environment of 5 °C after soaking in water. Then, they dried samples at 50 °C for 22 h prior to the process to regulate the MC of samples. The authors used hot pressing at various temperatures of up to 220 °C for enhancing the flexural, compression, and impact resistance of bamboo. According to their results, the optimum process temperature was 220 °C in terms of obtaining the best density and flexural modulus, while 160 °C identified to be the optimized temperature in terms of flexural strength (as shown in Figure 14) (TAKAGI et al., 2008).

Figure 2.14 – **a)** Flexural strength, and b) modulus as a function of molding temperature (the dashed line shows the flexural strength and modulus of un-densified bamboo), data from (TAKAGI et al., 2008).

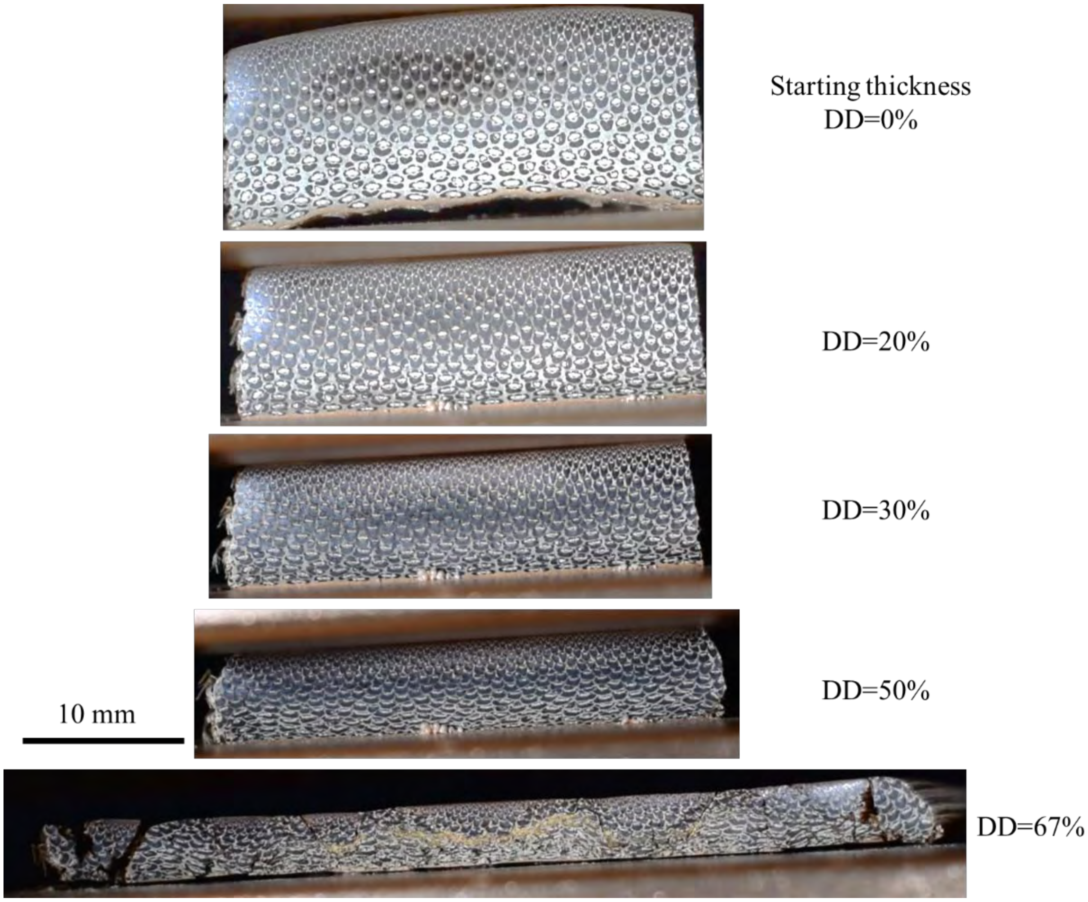


Source: (KADIVAR et al., 2020a)

2.7.3. The Influence of Densification Degree

THM pressure is one of the most significant determinants for the densification of bamboo. Semple et al. (2013) (K.E. SEMPLE, F.A. KAMKE, A. KUTNAR, 2013) clearly showed this effectiveness by densifying small specimens of Moso bamboo strips to produce samples with four different densification degrees using the THM method. Based on the study, the higher the compression ratio, the higher the density and MOR. By compressing the bamboo to 50% of the original thickness (50% densification degree), the method increased the MOR by 71% and the MOE by 31% (Table 3). However, strips compressed to 50% of the initial thickness generated a partial side spread of the tissue, and lateral expansion happens. Compressing to 33% (67% densification degree) causes notable tissue displacement and distortion. Figure 15 shows similar behavior for bamboo D.Asper. Therefore, a densification degree (DD) around 50% has been suggested to be the optimum compression ratio (CR) for the elimination of collapsible void space.

Figure 2.15 - Cross sections of D.Asper strips densified at 160 °C.



Source: (KADIVAR et al., 2020a)

2.7.4. The Influence of Water and Initial Moisture Content

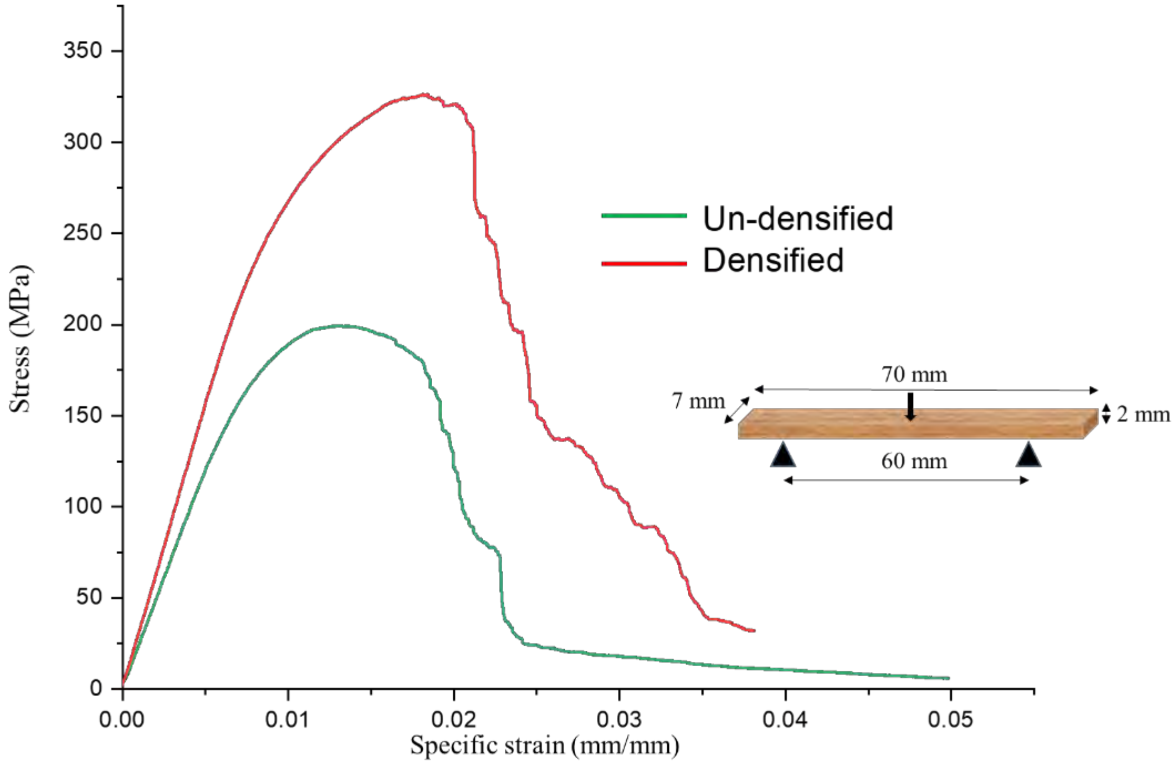
In Figure 11, the graph demonstrates that the softening behavior depends on initial moisture content. Water can facilitate bamboo softening and consequently the densification because of material plasticization (AMADA, S. AND LAKES, 1997; ARCHILA-SANTOS; ANSELL; WALKER, 2014). Santos et al. (2014) (ARCHILA-SANTOS; ANSELL; WALKER, 2014) immersed Guadua angustifolia Kunth in water and densified it using thermo-hydro-mechanical (THM) treatments. The authors examined this pre-treatment by evaluating the tensile properties of THM modified Guadua and comparing it with the tension strength of undensified samples. The results showed the improvement of specific stiffness of the material by a factor of 1.25 as the result of pre-soaking the specimens.

Kadivar et al. (2019) (KADIVAR et al., 2019a) used an open thermal press to densify bamboo D. asper in its radial direction using various initial MC, from 0 to 20%. The study evaluated the effect of starting MC on bending and physical-chemical characteristics of the material. Applying the TM parameters presented in Table 2, the material achieved a maximum of 31.2% DD. According to the results, the initial MC is one of the effective parameters that needs to be considered for bamboo densification. Bamboo samples with less than 5% MC manifested cracks in TM processing, and consequently, the final product does not perform well in the presence of water. A high MC prevents uniform densification (Figure 16), and in this case, the samples need more time to release internal gas pressure. Based on the results, initial moisture content of approximately 10% was observed to be optimal, which can be high enough to satisfy the softening requirements for TM process and homogeneity in the densified product. Using this optimized MC, the densified bamboo achieved a 56% increase in ultimate stress in bending (Figure 17). However, in terms of physical properties and dimensional stability, the densified samples in all samples resulted in decreased performance compared to the natural bamboo.

Figure 2.16 - Bamboo sample densified with 20% MC.

Source: (KADIVAR et al., 2020a)

Figure 2.17 – Stress-strain bending plot of non-densified and densified samples with 10% of moisture content (data from (KADIVAR et al., 2019a)).

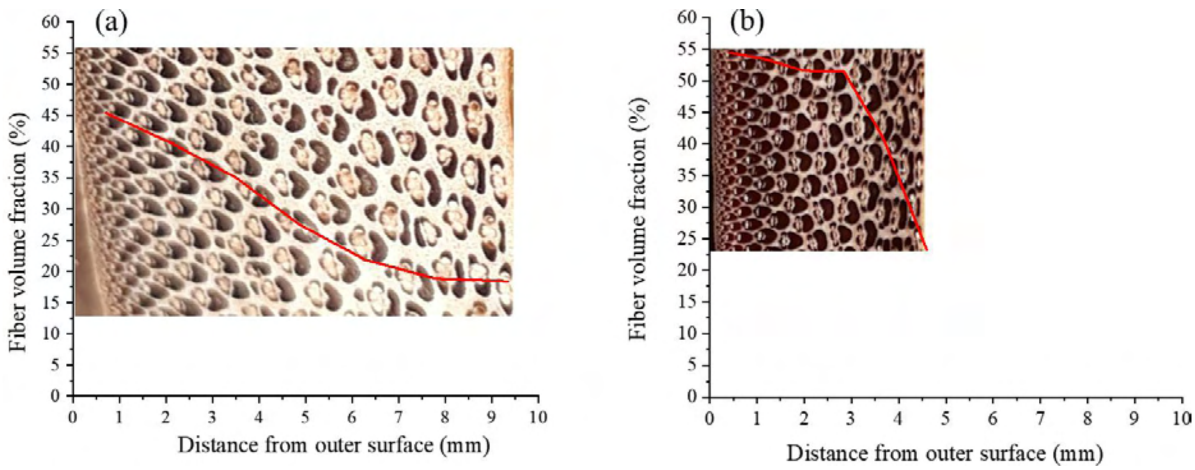


Source: (KADIVAR et al., 2020a)

2.7.5. The Microstructure of Densified Bamboo

Li et al. (1994) (LI et al., 1994) produced reformed bamboo using a process including softening, followed by compressing and fixing the material and studied its microstructure. The study included optical microscopy of bamboo cross-sections before and after the process which revealed the rearrangement of fibers, dense compaction of vascular bundles, disappearance, and closure of cavities, as well as deformation of the components configuration. The authors used an Automatic Image Analyzer for estimating the ratio of fibers to the total area, and their fiber volume fraction (Vf) (Figure 18). For the natural bamboo, Vf gradient declines along the thickness. After the reforming process, the Vf is uniformly distributed, about 50% through the thickness. However, adjacent to the inner layer, the fiber distribution appears not to change due to the process (LI et al., 1994).

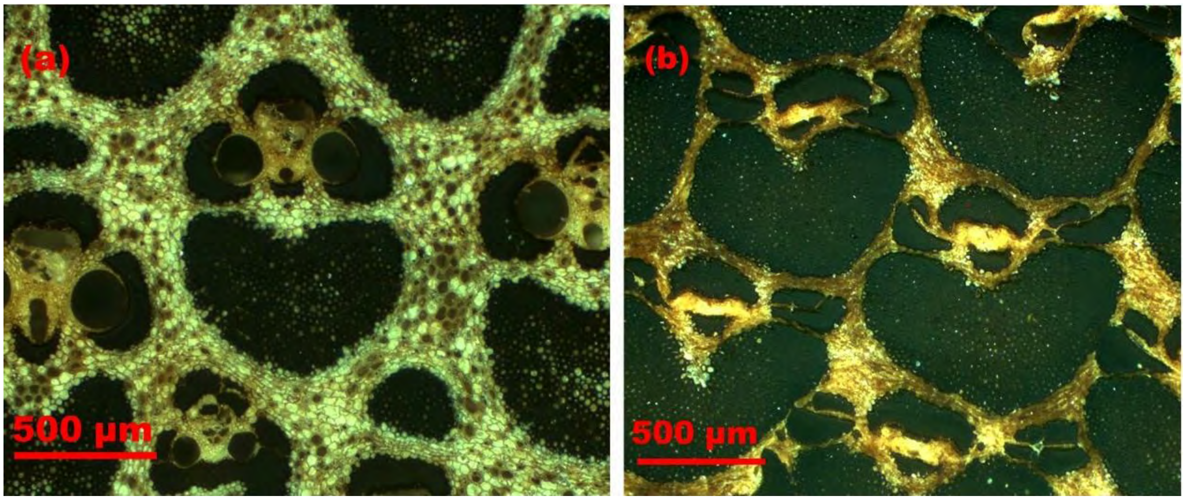
Figure 2.18 – Fiber volume fraction of (**a**) normal bamboo, and (**b**) reformed bamboo (data from (LI et al., 1994)).



Source: (KADIVAR et al., 2020a)

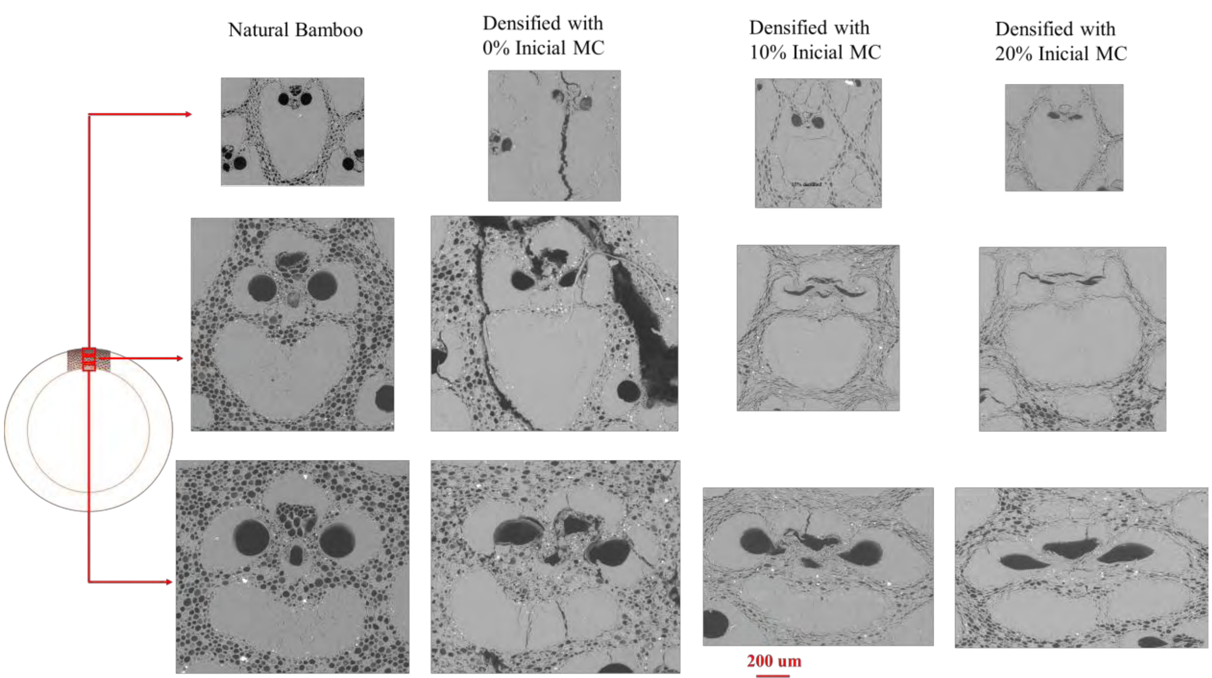
Several researchers have reported the microstructure of densified bamboo (ARCHILA-SANTOS; ANSELL; WALKER, 2014; DIXON et al., 2016a; KADIVAR et al., 2019a; ZHANG et al., 2019a). According to the microscopic results of Archila-Santos et al. (2016) (ARCHILA-SANTOS; ANSELL; WALKER, 2014), the vessels (vascular conduits) close as the result of densification. The breakdown of vacant spaces, such as protoxylem and phloem are also recognized (Figures 18–20). Figure 20 shows the Scanning electron microscope analysis of the crosswise surface of non-densified and densified bamboo (D. asper) along with the specimens' thickness and at different initial MC levels. The images show the closure of the cavities and the compaction through the cell wall thickness. It is also possible to see the different compaction of bamboo components such as fiber bundle and parenchyma. Kadivar et al. (2019) (KADIVAR et al., 2019a) stated that the densification happens principally in the central layer of the samples section (KADIVAR et al., 2019a).

Figure 2.19 – Optical microscope images (**a**) before densification and (**b**) after densification.



Source: (KADIVAR et al., 2020a)

Figure 2.20 – Scanning electron microscope (SEM) images of the non-densified and densified samples in different initial MC (from 0% up to 20%).



Source: (KADIVAR et al., 2020a)

2.7.6. Best Practices

The bamboo species, densification degree, temperature, and initial moisture content are the suggested effective parameters for the densification process. By combining the mentioned studies, there is an optimized range for each parameter, and using a parameter outside this range causes defects in the performance of the final product.

According to the available literature, although the best practice (in terms of bending and tensile resistance and also physical properties) depends on the bamboo species, it is possible to approximate the best water content as 7–10%, the best temperature between 140 to 160 °C, and densification degrees of 30–50% for the bamboo modification.

2.7.7. Gaps in Knowledge

There are some important gaps regarding other parameters such as pressure, time, and rate, which are also effective parameters that are scarcely informed in the publications. Based on the theory, the pressure rate is correlated with moisture content, since low rates can dry the material while high rates can also govern the incidence of cracks. On the other hand, in terms of the mentioned effective parameters, theoretically, there should be correlations that need to be identified. Identifying these correlations helps the process optimization, which will also lower the energy consumption and consequently reduce the associated costs and environmental impacts from manufacturing.

Existing studies have evaluated the process mostly in flexural strength and some physical properties. Information regarding the tensile properties of densified bamboo is scarce. Therefore, it is difficult to make a general conclusion about the mechanical properties of densified bamboo. Regarding some physical properties, such as spring back, swelling, and water absorption, at the best of the authors' knowledge, little information is available, which accordingly, densification harms the dimensional stability (KADIVAR et al., 2019a). Therefore, it can be an important topic for future investigations in this field to solve the problem.

Regarding the densification process, other methods that have been used for wood can be utilized for bamboo to solve some challenges, such as swelling and water absorption. Last but not least, using chemical modification as a pre-treatment can also facilitate the process.

2.8. Conclusions

In this review chapter, the development of densified bamboo according to different processes recently introduced in the literature was discussed. Applying thermo-mechanical (TM), thermo-hydro-mechanical (THM), or viscoelastic-thermal-compression (VTC) methods allow the densification of bamboo specimens in the radial direction to approximately 20–67% of densification degree, increasing the density by 20–100%.

The densification process can decrease the heterogeneity and enhance bamboo mechanical performance. However, some physical properties, such as dimension stability, swelling, and water absorption, are reported to be compromised. Moreover, the process has an influence on chemical components of bamboo only at temperatures higher than 160 °C. The efficiency of densified bamboo can vary depending on the process parameters. Bamboo species, moisture content, hot-press temperature, pressing time, and pressure are the main factors that affect the densification process.

Though several aspects of these modifications are known, the fundamental influence of the process on the performance of densified bamboo has yet to be explored for the development of bamboo modification technologies. Further investigation is needed to design effective parameters for the processing and densification of bamboo (KADIVAR et al., 2020a).

3. CHAPTER 3 –The influence of the initial moisture content on densification process of D. Asper bamboo: physical-chemical and bending characterization

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Abstract

Densification process aims to improve the physical and mechanical properties of wood and bamboo products. However, its processing parameters were not yet thoroughly investigated for bamboo. In this study, Dendrocalamus asper bamboo was densified in its radial direction in an open thermal press with different starting moisture content (MC), from 0 to 20%, to evaluate its effect on bending and physical-chemical properties. A maximum densification degree of 31.2% was achieved. Physical characterization and three-point bending tests showed that densification process increases density and all related bending properties (modulus of rupture (MOR), modulus of elasticity (MOE), the limit of proportionality (LOP), and specific energy (SE)) of bamboo, producing a more homogeneous material. The densified samples with 10% MC presented the best bending properties, with an average MOR, MOE and dynamic MOE of 318, 27,754 and 34,120 MPa respectively, with an increase of 56% for MOR and 41% for MOE in comparison with un-densified samples.

SEM analysis of fractured samples showed an improvement of the fibers-parenchyma interface after thermo-mechanical modification, confirmed by the presence of unitary fiber failure. XRD analysis revealed that although densified bamboo had higher cellulose crystallinity compared to un-densified samples, the starting moisture content did not affect on the cellulose structure. FTIR showed that there are no significant changes in the chemical composition in all the analyzed conditions.

However, the samples with moisture content below 5% presented cracks during the thermal-mechanical process, which resulted in higher thickness swelling and water absorption. Additionally, when samples with 20% MC are densified, an excess of water entrapped in the middle of the samples causes heterogeneous densification.

The control of the initial moisture content of bamboo is a strategic parameter to improve the efficiency of the densification process. An initial moisture content around 10% is recommended for bamboo, which can guarantee enough plasticization and at the same time homogeneous properties in the final product.

Keywords: Bamboo, Thermo-mechanical modification, Microstructural analysis, Three-point bending test.

3.1. Introduction

The construction sector is the most significant contributor to environmental impacts (KLIJN-CHEVALERIAS; JAVED, 2017), which is responsible for approximately 40% of the total energy consumption and 36% of the entire universal CO2 emission (CHAU et al., 2012; KUMAR et al., 2017). Accordingly, to reduce the environmental impact, construction materials are of great importance (ROSSELLÓ-BATLE et al., 2015; ZHANG; WANG, 2017; HOSSAIN; POON, 2018). On this respect, bamboo has been widely discussed and reviewed as a bio-based construction material in which life cycle assessment studies show its indisputable potential to reduce carbon emissions of the construction sector (VOGTLÄNDER; VAN DER LUGT; BREZET, 2010; YU; TAN; RUAN, 2011; CHANG et al., 2018; ESCAMILLA et al., 2018).

Bamboo culm grows as a hollow cylindrical pole composed of regions with aligned and continuous fiber bundles, called internodes, separated by parts with solid transversal diaphragms and interwoven fiber bundles in the wall thickness, called nodes. The uniform distribution of fiber bundles across the wall thickness and differences in the culm diameter, thickness and internode length along with the culm’s height, make bamboo a functionally graded material developed to resist wind forces and weather conditions. However, these morphological aspects are also barriers in the utilization of raw bamboo as an engineered structural material. Designing and producing a series of bamboo-based panels (BBP) with different structures can overcome bamboo disadvantages and transfer it to a large flat surface stabilized product with excellent mechanical, physical, and aesthetical advantages.

BBP started to appear in the construction industry since the 1970s (HUANG; SUN; MUSSO, 2017b), following the advancement of modern timber industry (LI et al., 2019). A wide range of commercialized products and applications in the construction sector, i.e., floors, ceilings, and wall finishes can be found. These BBP, including bamboo-plywood, bamboo particleboards, bamboo oriented strand board(OSB), glued laminated bamboo, bamboo scrimber, and flattened bamboo panels, as the mainstream bamboo products in the market, are formed through ’decomposition’ and ’recombination’ processes that improve raw material utilization efficiency (HUANG; SUN; MUSSO, 2017b). Decomposition requires breaking down bamboo culms into small components such as veneers, strips, slivers, strands, and particles, and recombination involves adhering these small units together mainly using an adhesive with hot pressing.

Thermo-hydro-mechanical treatment (THM) is the main feature to achieve densification. It is an essential process for increasing panels’ density, consequently improving mechanical properties, and for making their structure uniform. Although densified bamboo has not been considered as a product itself yet (DIXON et al., 2016a), the influence of the THM process has to be fully understood and explored because the quality of final products is affected by raw bamboo elements.

Similar to wood, the performance of compressed bamboo is affected by a range of factors such as its species, size of the specimen, moisture content, pressing temperature and time, pressing or the degree of compression. The wood densification literature shows that the densified wood physical and mechanical properties are significantly influenced by the interaction between moisture content and temperature (NAVI; SANDBERG, 2011; ZHAO, 2017) and the effect of temperature is pronounced under high moisture content (KÚDELA, 2018). As a result of heating, wood is momentarily transformed into a soft and rubbery structure from a stiff and glassy material. At short times, low temperature, and low moisture content, wood exhibits glassy behavior. At long times, high temperature, and high moisture content, wood exhibits rubbery behavior (ULKER; IMIRZI; BURDURLU, 2012).

Wood undergoes elastic-visco-plastic deformation during compression and due to the softening of hemicelluloses and lignin in the presence of water. It has been found that the pliability of wood improves as its moisture content (MC) increases (GAO; GUO; LUO, 2018). On the other hand, high wood MC can also have negative impacts, such as explosion (caused by steam release) and dimensional heterogeneity (HEGER et al., 2004). The mechanisms of bamboo densification, however, have not been fully recognized yet and pressing parameters that can guarantee appropriate dimensional stability, as well as other properties of the pressed bamboo, are still unknown.

Few recent studies about densified bamboo showed that during bamboo compression at elevated temperature, the presence of water serves to plasticize cell wall components (ARCHILA-SANTOS; ANSELL; WALKER, 2014; DIXON et al., 2016a) and reduce the glass transition temperature (Tg) (MATAN; KYOKONG; PREECHATIWONG, 2007). Archila-Santos et al. (2014) densified Guadua Angustifolia Kunth (Guadua) using THM treatments and evaluated the influence of water soaking as a pretreatment on tension strength of densified bamboo. After THM treatment processing of dry and water-saturated Guadua samples, they compared the tensile strength of THM modified Guadua with un-densified samples. Their results show mechanical properties of the material were improved; e.g. the specific stiffness of the species of Guadua was increased by a factor of 1.25 for pre-soaked samples (ARCHILA-SANTOS; ANSELL; WALKER, 2014). According to Matan et al. (2007), the Tg of bamboo with higher initial moisture content was lower than that of bamboo with lower initial moisture content (MATAN; KYOKONG; PREECHATIWONG, 2007). Additionally, the maximum and minimum Tg of the bamboo Dendrocalamus asper Backer were (194 ± 10)°C and (85 ± 10)°C, respectively, and were obtained from oven-dried bamboo and water-saturated bamboo respectively. Gao et al. (2018) investigated the changes in compressed Moso bamboo (Phyllostachys pubescens) after hot-press molding and confirmed that water has a plasticizing effect (GAO; GUO; LUO, 2018).

The moisture content plays a key role in the plasticizing effect of bamboo in the presence of temperature and pressure. A comprehensive study is required to understand the optimal moisture content range for the TM process to be more effective. Therefore, in this work, the initial moisture content influence in the TM process has been evaluated through physical-chemical and mechanical characterization (density, dimensional stability, dynamic and static bending, fracture behavior, cellulose structure, and chemical composition) to optimize and understand the underlying mechanisms of bamboo densification.

3.2. Materials and methods

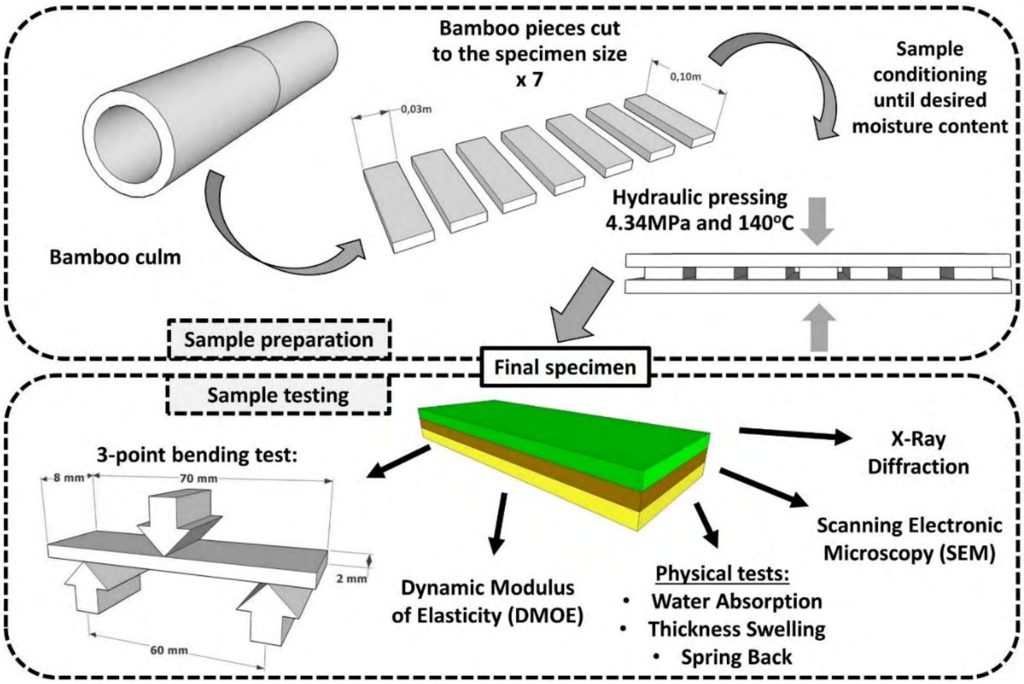
3.2.1. Sample preparation

Mature bamboo culms, three years old, of Dendrocalamus asper species, were harvested at the experimental field in the University of São Paulo, USP campus, at Pirassununga, Brazil (21°58'53.5"S 47°26'03.3"W). Then, the harvested culms with an average diameter of 18 cm were stored in a protected environment at room temperature for four months until reaching the equilibrium state with the MC 10.67% (COV 7.03%). A total number of 70 specimens of (30 × 100 × 11) mm3 were extracted from different internodes originally located at the middle part of the culms. The wall thickness of all the samples varied between 10-13 mm. The obtained material was gradually dried to expose the samples to a less temperature stress, e.g. firstly bamboo specimens were oven-dried at 60°C for five days and then at (105 ± 2)°C for two days- zero moisture content – to achieve the dry mass m0. After drying the samples had an average shrinkage of 3.81% (COV 15%). Then, the samples were conditioned in a climate chamber (30°C, 75% RH) for four days until reaching a target MC of 5% (average: 5.72, COV: 4.28%), and for 18 days until a nominal MC of 10% (average: 9.46, COV: 6.23%). After conditioning, 14 samples were placed in room conditions to be used as the reference samples (without densification). A desiccator containing water (25°C, 98% RH) has been used for conditioning samples for 19 days to obtain a nominal MC 20% (average: 19.14, COV: 5.28%) MC. For each condition including the reference, 14 specimens were used, with a total number of 70 samples submitted to the densification process.

3.2.2. Densification process

The acclimated samples were placed and compressed in the radial direction, inside an open thermo-hydraulic press system. All samples were compressed following the same TM procedure at 140oC, 4.34 MPa for 15 min (Figure 3-1). Before applying the pressure, the samples were placed in the press equipment, in contact with the hot plates for 5min.

Figure 3.1 - TM process and characterization schedule.



Source: (KADIVAR et al., 2019a)

Before pressing, the specimens were weighed with an accuracy of 0.01 g and measured with an accuracy of 0.01 mm. The densification degree (DD) was calculated by measuring the thickness of the test specimens in the pressing direction two times: before and immediately after the removal from the pressing equipment, according to the Equation (1). Where t_o and t_1 are the thickness of the samples before and after densification respectively

$$DD = (t_o - t_1)/t_o \times 100\% \qquad \text{Eq. 3.1}$$

3.2.3. Physical Properties

3.2.3.1. Moisture content (MC)

The MC for both densified and un-densified bamboo samples in specified condition were based on the dry mass and was calculated using Equation 2. For the dry mass, the samples were oven-dried at (105 ± 2)°C for 48 h until reaching a constant weight, as per ISO 22157:19 (22157:2019,).

$$MC = (m1-m_o)/m_o \times 100\% \qquad \text{Eq. 3.2}$$

Where m_0 refers to dry mass and m_1 is the masses after conditioning.

3.2.3.2. Apparent density

The apparent density (ρ) of the specimens in three different layers, inner, middle, and outer layer (Figure 3-1) was determined according to Equation 3.3, where m and v are the mass and volume of the sample at a specific MC.

$$\rho = \frac{m}{v} \qquad \text{Eq. 3.3}$$

3.2.3.3. Dimensional stability

3.2.3.3.1. Spring-back

The spring-back (SB) of the densified samples was determined after conditioning at (20 ± 2)°C and (65 ± 3)% RH for one week and calculated using Equation 3.4. Where t_1 is the thickness immediately after densification and t_2 is the thickness after conditioning the samples at (20 ± 2)°C and (65 ± 3)% RH.

$$SB = (t_2-t_1)/t_1 \times 100\% \qquad \text{Eq. 3.4}$$

3.2.3.3.2. Thickness swelling and water absorption

The water absorption (WA) and thickness swelling (S) tests were carried out based on ASTM D1037-12 for the bamboo specimens before and after densification (D1037-12, 2012). First, the specimens with the dimension of (30 × 30 × t) mm were dried at (105 ± 2) oC before measuring the weight and volume. Afterward, the specimens were fully immersed in water at (22 ± 1)oC, and the weight and thickness changes were measured after 1 h and 24 h. Subsequently, S and WA were determined using the Equations (3.5) and (3.6) respectively. Where, m0 and m2 are the masses before and after the immersion of the samples in water after 1 h and 24 h respectively, and t2 and t3 are the thicknesses before and after the test respectively.

$$WA = (m_2-m_o)/m_o \times 100\% \qquad \text{Eq. 3.5}$$

$$S = (t_3 - t_2)/t_2 \times 100\% \qquad \text{Eq. 3.6}$$

3.2.4. Excitation pulse non-destructive test (NDT)

Un-densified and densified specimens were sliced into prismatic pieces with nominal dimensions of (2 × 7 × 70) mm from outer, middle and inner regions, which are presented in Figure 1 with different colors. The NDT tests were performed according to the ASTM E1876-15 Standards using excitation pulse testing machine Sonelastic® to determine the dynamic elastic modulus (E1876-15,). A total number of 210 samples from three positions of the bamboo sections have been tested.

3.2.5. Three-point bending test

The static elastic modulus was determined for each sample subsequently after NDT. Three-point bending tests were conducted in a Servohydraulic Test System MTS Landmark with a 1 kN load cell, a 60 mm span and a displacement-controlled testing speed of 2 mm/min, based on Dixon et al. (2016) and recommendations of ASTM D7264 – 15 (D7264-15, 2015; DIXON et al., 2016a). For the test, the specimens were intentionally placed so that the bamboo outer layer was on the compression side. The modulus of rupture (MOR) and modulus of elasticity (MOE) were determined with the equations (3.7) and (3.8):

$$MOR(MPa) = \frac{3F_{max}L}{2bd^2} \qquad \text{Eq. 3.7}$$

$$MOE\ (MPa) = \frac{F_e L^3}{4\delta b d^3} \qquad \text{Eq. 3.8}$$

Where F_{max} is the maximum load in (N), L is the testing span (mm), b is the width and d is the thickness of the specimen (mm). Fe is the load difference in (N) between the upper and lower boundary loads within the proportional limit, and d is the mid-span deflection (mm) of the specimen under Fe.

3.2.6. X-ray diffraction

An X-ray diffractometer Horiba LA-960, with CuKα radiation generated at a voltage of 40 kV and a current of 30 mA was used to scan between 5–65° 2θ at 10°/min. The sample crystallinity of cellulose was calculated using the Segal peak height method (NAM et al., 2016), according to the crystallinity index (CrI) in Equation (3.9).

$$CrI = \frac{I_{002} - I_{am}}{I_{002}} \, x \, 100 \qquad \text{Eq. 3.9}$$

Where, I_{002} represents the maximum intensity of the (002) plane reflection of the cellulose I structure at approximately $2\theta = 22.7^\circ$ and I_{am} the intensity of the amorphous reflection at $2\theta = 18^\circ$.

3.2.7. Fourier transform infrared spectroscopy (FTIR)

The monitoring of the chemical modifications involved during the bamboo densification process, using different MC before densification, was carried out through Fourier transform infrared (FT-IR) spectroscopy. In order to prepare specimens for this analysis, the bamboo samples were ground into powder and passed through a 100-mesh sieve. Samples were analyzed on a PerkinElmer brand Spectrum On equipment with the ATR (Attenuated Total Reflectance) universal sample accessory. For each analysis, 32 scans were used in the spectral region of 4000-600 cm^{-1} with a resolution of 4 cm^{-1}.

3.2.8. Microstructural characterization

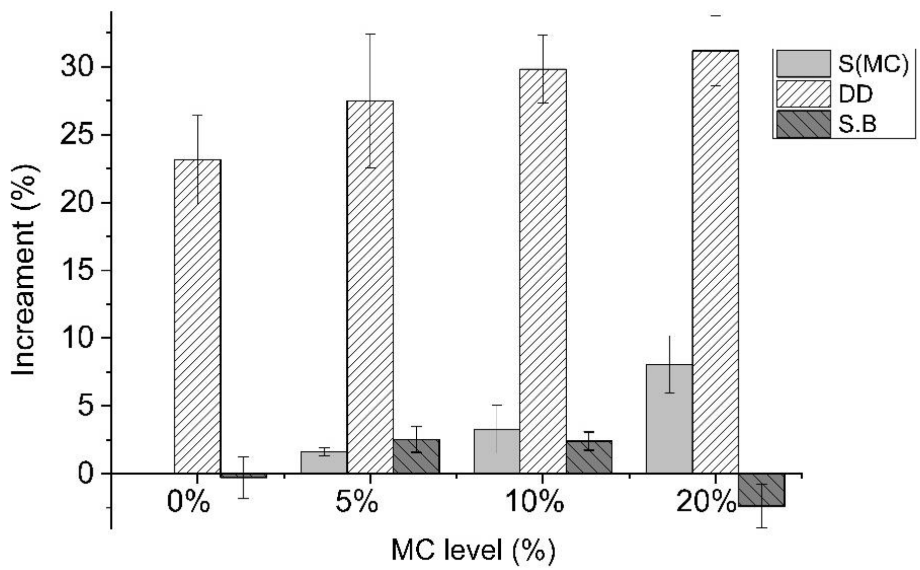
The bamboo's microstructure was investigated by scanning electron microscopy (SEM), using a HITACHI TM-3000 SEM model with an acceleration voltage of 15 kV and using backscattered electron detector mode. This analysis was performed on two different sets of samples. The first set of images was on the cross-sections (transversal) of both un-densified and densified bamboo. Samples were polished before microscopic observation by gradually reducing the sandpaper sizes and using the soft polishing cloth and diamond suspensions of 6, 3 and 1 μm. The second set of SEM analysis was on the fracture region of bamboo samples after the static bending test in which no polish was needed before image analysis.

3.3. Results and discussion

3.3.1. Physical properties

The results of the thickness measurement during the samples preparation to achieve specific MC show that as the MC rises, the thickness increases. This thickness swelling, (S(MC)), which occurs during the conditioning of samples until achieving 5, 10, and 20 % of MC, is presented in Figure 3-2.

Figure 3.2 - Effects of MC on the swelling of un-densified bamboo (S(MC)), densification degree (DD) and spring back (SB) of densified bamboo



Source: (KADIVAR et al., 2019a)

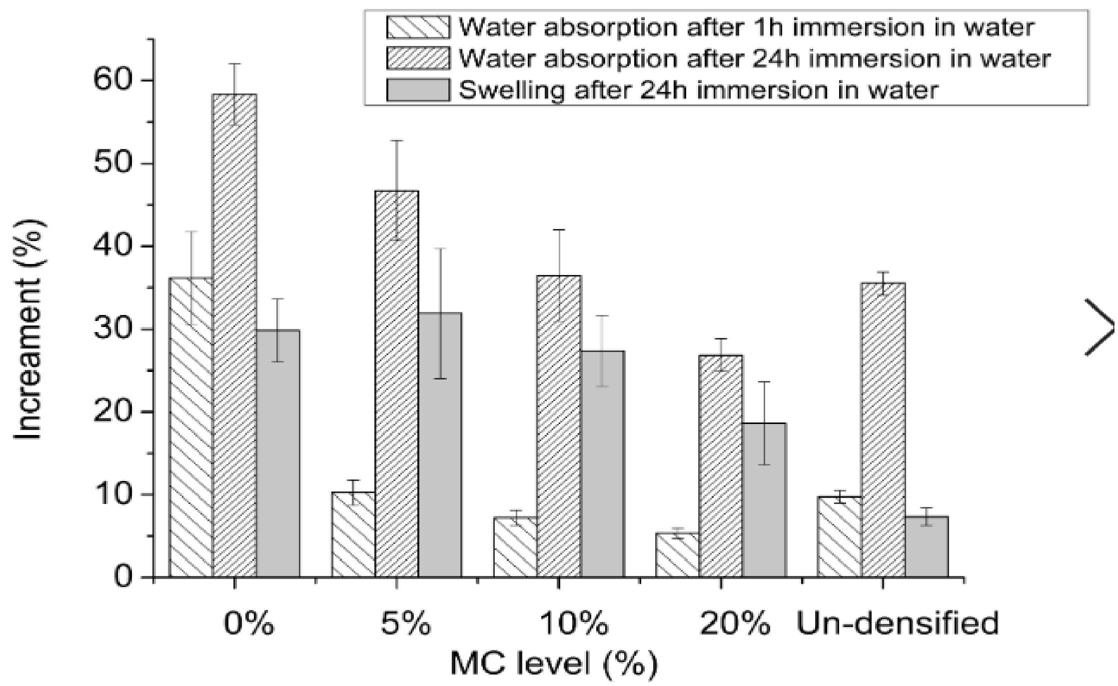
The DD results of densified bamboo at different MC conditions (Figure 3-2) revealed that the DD varies with the initial moisture content. The higher the water content, the higher the DD. This trend may be related to the higher densification capacity of the material due to the increase of the lignin plasticity, caused by a direct correlation between the moisture content and its glass transition temperature (MATAN; KYOKONG; PREECHATIWONG, 2007). It can be noted from Figure 3-2 that there is negligible spring back effect in all the conditions. The negative values obtained for the 20% MC condition is related to the inhomogeneous geometry of the samples after densification, which showed a pronounced curvature in the inner layer (Figure 3.3). This effect can be explained by insufficient time of pressing during the densification process, trapping water inside the middle part of the samples. Therefore, this problem might be solved by increasing the pressing time or controlling the compression rate.

Figure 3.3 - Bamboo samples ; a) un-densified, b) densified with 10% MC, c) densified with 20% MC

Source: (KADIVAR et al., 2019a)

The thickness swelling and water absorption of densified and un-densified bamboo after 1 h and 24 h are presented in Figure 3.4. After immersion for 24 h, densified bamboo samples presented higher thickness swelling values compared to un-densified samples. This behavior is explained by the expansion of the inner material structure due to the water filling of the bamboo cavities. The higher thickness swelling is displayed by densified samples with 5% MC, followed by densified samples without initial moisture content and 10% initial moisture content. The lowest swelling for densified samples is presented by 20% moisture content samples. The results of the water absorption after 24 h also reveal an adverse effect of densification at all different MC levels. However, the water absorption decreases by increasing the MC. The water absorption results in short term immersion time, after 1 h, shows a higher value for un-densified bamboo compared to 20% and 10% MC densified samples. Among all, samples densified with 0% MC have the highest water absorption for both short and long-term immersion time. This is because of a high number of cracks that happened during the densification.

Figure 3.4 - Water absorption and thickness swelling after 1 h and 24 h of un-densified and densified samples with different initial MC

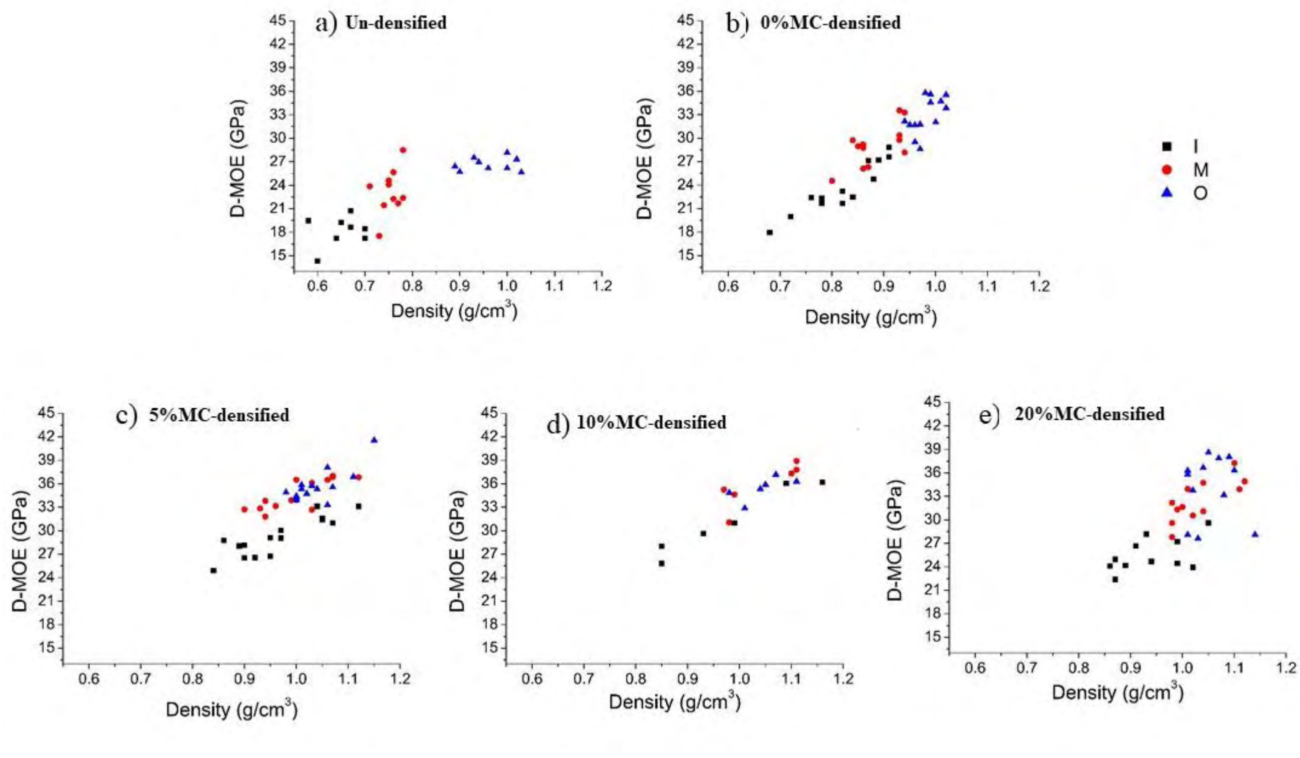


Source: (KADIVAR et al., 2019a)

3.3.2. Excitation pulse non-destructive test (NDT)

Before doing the test, all the samples exposed to identical environmental humidity conditions, in the equilibrium of moisture content. Figure 3.5 shows the results of the dynamic modulus of elasticity. It is possible to establish a clear correlation between density and DMOE for un-densified samples, where the higher the density, the higher are the DMOE values. Inner layer samples present both the lowest density and DMOE values, while the outer samples exhibit the highest values. This trend also remains for densified dry samples, although they display higher density and DMOE as a consequence of densification. According to these results, the presence of water within the samples during densification also increases bamboo density for every fraction of the bamboo section. The main difference attributed to the increased moisture content is that DMOE is not as affected by density as it is for dry samples. Besides, DMOE values dispersion is lower compared to the dispersion of density values. Moreover, the performance of the middle layer and outer layer samples becomes more similar to the increase in moisture, suggesting a uniform distribution of DMOE across the bamboo thickness.

Figure 3.5 - Dynamic modulus of elasticity. Un-densified samples (a). Densified samples (b) 0%MC, (c) 5%MC, (d) 10%MC and (e) 20%MC.



Source: (KADIVAR et al., 2019a)

3.3.3. Three-point bending test

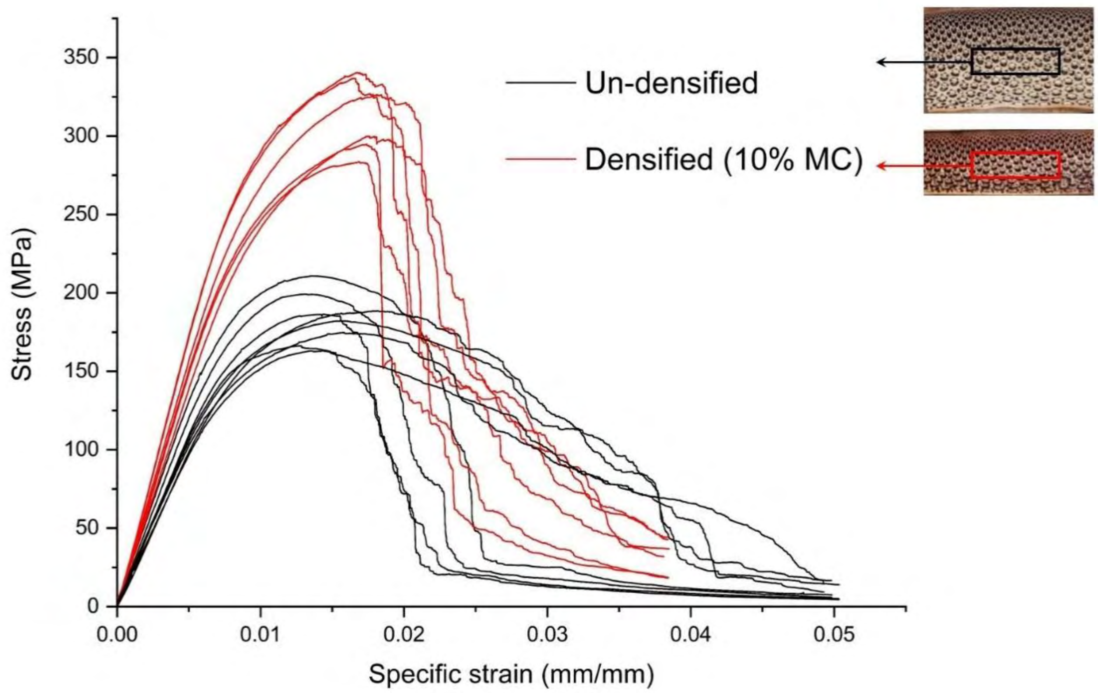
The authors observed an increase of density of each layer, compared with the un-densified samples (Table 3.1). For the 10%MC, there was a density increase of 56% for the inner layer while for the outer layer, there was an increase of only 8%. Similar behavior can be observed in the mechanical properties. According to the results presented in Table 1, the outer layer for all un-densified and densified samples presented the highest MOR values followed by middle and inner layers. Additionally, regardless of the initial MC, the outer layer for every densified sample has higher MOR values than un-densified bamboo. However, the increase in moisture content has no influence on the MOR of the outer layer for any of the assessed samples. The MOR of the inner and middle layers is only increased with the presence of moisture within the sample since un-densified samples, and dry densified samples show identical average MOR values. The samples with initial MC of 10% had the highest MOR for the inner and middle layers, while samples with 20% of water content presented the lowest values. It seems the initial moisture content around 10% can be enough for the required plasticization before densification. Figure 3.6 presents a comparison in terms of the stress-strain curve of un-densified and densified bamboo with 10% MC. This comparison, which has been performed only for the middle layers, clearly shows the bamboo bending improvement using TM bamboo densification process.

Table 3.1 - Three-point bending results of un-densified and densified samples extracted from the inner (I), middle (M) and outer (O) layers of the bamboo wall thickness.

Condition	Thickness position		ρ (g/cm³)	MOR (MPa)	LOP (MPa)	MOE (MPa)	SE (kJ/m²)	SMOR (MPa/ρ)
Un- densified	I	Average	0.65	143.2	108.6	15,406	24.50	220.2
		COV%	6.69	8.86	8.41	12.03	26.63	7.41
	M	Average	0.76	183.99	131.11	20,523.1	28.86	243.12
		COV%	3.07	8.76	9.83	10.26	23.72	7.13
	O	Average	0.96	283.27	202.84	22938.6	44.97	294.88
		COV%	5.73	2.72	6.27	3.29	22	6.98
	All - average		0.79	203.50	147.52	19622.8	32.78	252.72
Densified - 0% MC	I	Average	0.82	147.82	117.67	17520.6	21.72	179.81
		COV%	8.90	22.89	25.26	18	17.14	2.03
	M	Average	0.88	199.81	149.65	23371.8	28.16	230.6
		COV%	5.58	23.33	18.99	11.72	14.36	2.09
	O	Average	0.98	351.39	254.13	28074.3	48.47	357.87
		COV%	2.60	5.36	5.77	5.82	5.98	3.76
	All - average		0.89	233.01	173.82	22988.9	32.78	256.09
Densified - 5% MC	I	Average	0.96	262.65	176.76	23073.12	37.31	272.08
		COV%	8.68	11.48	10.46	9.52	24.84	7.42
	M	Average	1.00	298.43	209.04	28157.04	39.67	299.34
		COV%	6.38	5.68	6.14	5.76	15.09	5.11
	O	Average	1.04	364.02	257.27	30199.56	50.82	353.14
		COV%	4.57	6.35	5.08	7.58	8.5	2.10
	All - average		1.00	308.37	214.36	27143.2	42.60	308.19
Densified -10% MC	I	Average	0.98	268.57	187.86	24824.79	34.53	274.06
		COV%	13.00	15.4	14.35	12.85	27.98	6.45
	M	Average	1.04	313.67	218.59	28627.13	42	300.51
		COV%	6.68	7.63	7.83	8.64	13.29	1.91
	O	Average	1.04	371.7	264.08	29811.83	50.94	356.28
		COV%	4.35	4.95	5.47	3.47	12.76	2.79
	All - average		1.02	318.0	223.51	27754.6	42.49	310.28
Densified - 20% MC	I	Average	0.94	214.82	148.99	23683.66	30.35	193.44
		COV%	7.28	51.59	55.07	62.76	43.51	16.80
	M	Average	1.03	250.29	190.31	25741.84	29.45	242.77
		COV%	5.07	9.61	9.71	9.56	16.63	8.16
	O	Average	1.05	350.74	243.66	28343.27	49.38	332.58
		COV%	3.99	7.88	7.98	10.58	11.75	6.16
	All - average		1.01	271.95	194.32	25922.9	36.39	256.26

Source: (KADIVAR et al., 2019a)

Figure 3.6 - Stress-strain bending plot of un-densified and densified samples with 10% MC



Source: (KADIVAR et al., 2019a)

Analogous behavior is observed for the MOE results in comparison to the MOR values. Densification increases MOE values for every sample with no difference of the moisture on this parameter for the outer layer. However, inner and middle layers of samples with 5, 10, and 20% MC have a higher MOE compared to dry samples. For the outer layer, the highest MOE was achieved by samples containing 5% of MC, while for inner and middle layers it was achieved by the samples with 10% MC.

The same trend has been observed for the limit of proportionality (LOP) and specific energy (SE). The SE was calculated as the area under the stress-strain curve divided by the cross-section of the samples. Samples with 5% and 10% MC had the highest energy absorption and limit of proportionality. This observation confirms that the densification process not only does not degrade the fibers but also improves its quality to support the load and improve the interface of fiber and matrix. By analyzing the ratio of the MOR over apparent density, named here as specific MOR (SMOR), the densification degree is more pronounced in the inner layer followed by middle and outer layers. For example, comparing the densified samples with 10% MC and un-densified samples, SMOR had an increase of 24.5 % 23.6 and 21% for the inner, middle, and outer layers, respectively. Observing the average densities of all the analyzed layers (from the un-densified and densified conditions), this different densification degree along the thickness is evident. It is worth mentioning that the densified samples present a more uniform density distribution in comparison with the un-densified ones. Therefore, the densification process enables the increase of performance in an efficient way, increasing the MOR and density of the weakest regions.

The high coefficient of variation in the inner layer for MOR, MOE, LOP, and SE of the densified samples with 20% MC is a sign of non-uniform bending properties in this region. The reason is that the samples were not densified in a homogenous way, so most of the samples after densification showed some pronounced curvature in the inner layer, as shown in Figure 3.

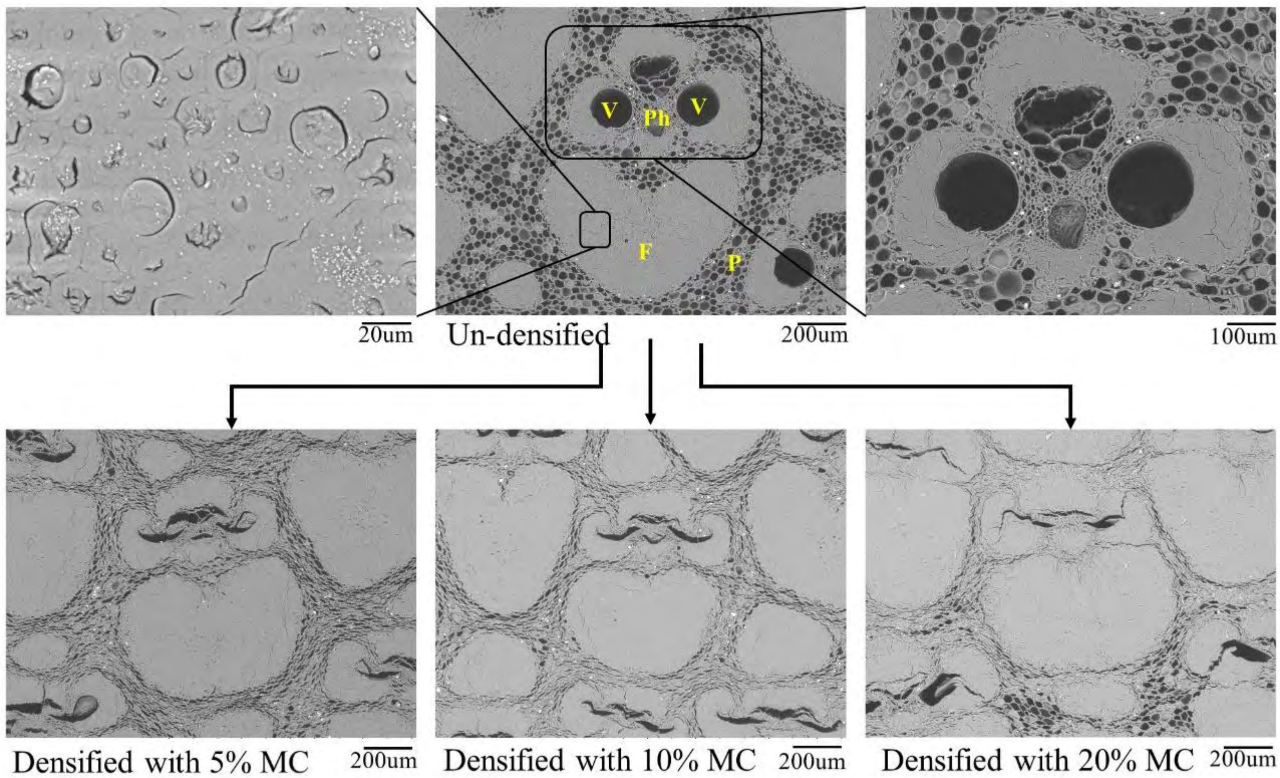
In general, the increase in the bending properties after densification is mainly related to the increase of density, caused by the collapse/compaction of the large vessels and parenchyma. Furthermore, it is possible to infer that the interaction between fiber bundles and parenchyma plays a key role in the bending behavior of the densified bamboo, as discussed in section 3.4, through SEM analysis of the fractured samples.

3.3.4. SEM analysis

3.3.4.1. Morphologies of bamboo cross-section

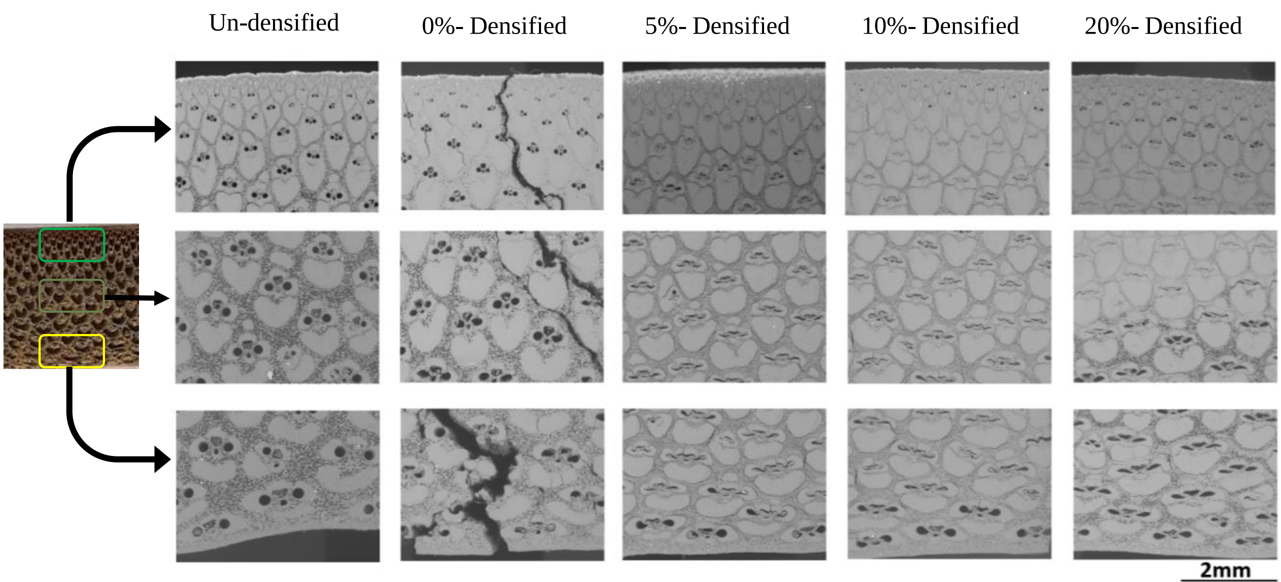
As shown in Figures 3.7 and 3.8, the densified samples with 5% and 10% MC underwent relatively good densification in terms of the parenchyma and vessels compaction. These observations corroborate with the density results shown in table 1.

Figure 3.7 - SEM images of un-densified and densified bamboo samples. The images were extracted from the middle layer. V=Vessels; Ph=Phloem; F=Fiber bundles; P=Parenchyma.



Source: (KADIVAR et al., 2019a)

Figure 3.8 - SEM images of the un-densified and densified samples.



Source: (KADIVAR et al., 2019a)

It can be observed that bamboo samples come across a more effective plasticization when the MC is increased. However, high MC is an obstacle, which prevents achieving effective densification by trapping water in the pores during the process. Therefore, the structure of samples densified with 20% MC had become more porous in some sections compared to the samples densified with 5 and 10% MC.

The SEM analysis was performed in three different regions of specimens' cross-section (outer, middle, and inner layers) to examine the characteristics of the deformed bamboo microstructure. Figure 8 presents a visual comparison of the transverse surface of un-densified and densified *D. asper* bamboo at different initial MC levels. According to what Archila-Santos et al. (2014) and Dixon et al. (2016) observed before, the pores (parenchyma and conducting vessels) have been collapsed by the densification process (ARCHILA-SANTOS; ANSELL; WALKER, 2014; DIXON et al., 2016a). Fiber bundle compaction and densification of the parenchyma are other evidence from the micrographs in Figure 8. Since the microstructure of un-densified bamboo is different along the different cross-section regions (percentage and morphology of fiber bundles and parenchyma), the microstructure of densified bamboo is also different in these three regions. Furthermore, the SEM images show that the densification occurred mainly in the middle layer in the samples with MC between 5 and 20%. This image shows a higher number of closed vessels in the middle and inner layers, especially in the samples with 10% MC.

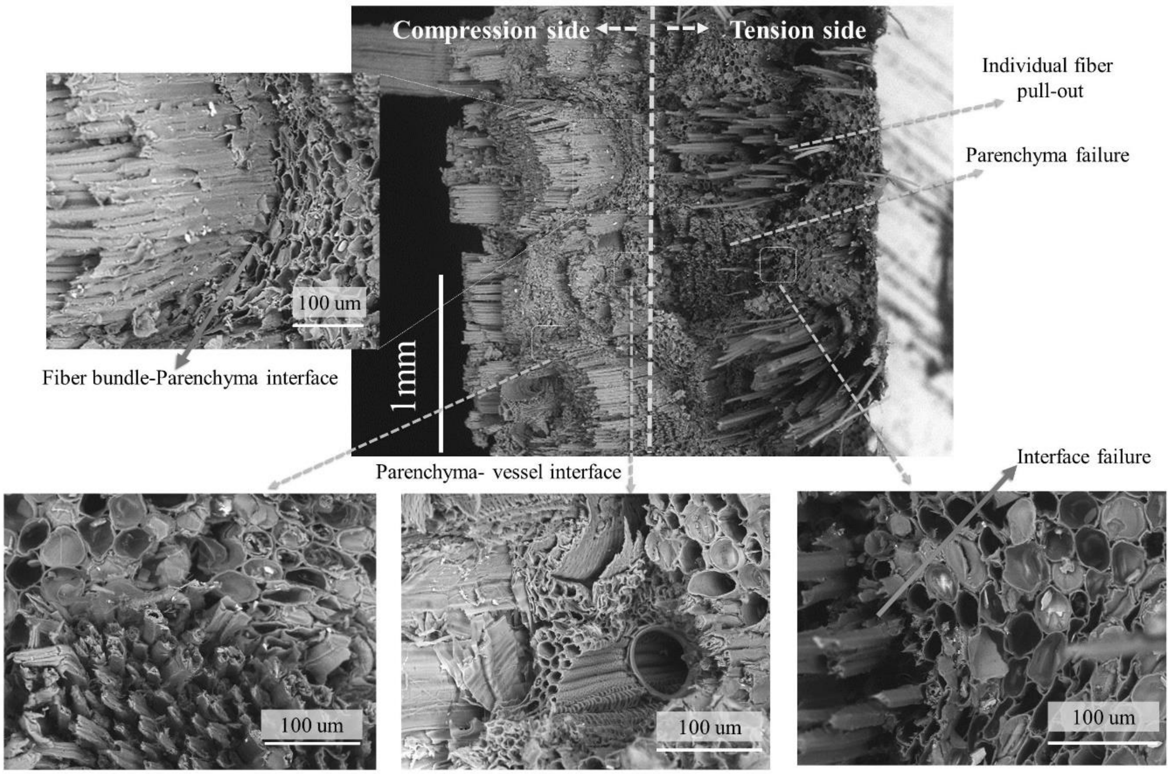
Samples with 0% MC have undergone many cell walls fracture without noticeable pores closure. It seems that dry bamboo samples have high friction and it is not easy to be densified without moisture. Additionally, since the presence of water reduces the Tg of the lignin (MATAN; KYOKONG; PREECHATIWONG, 2007), the temperature used for the process was not sufficient to plasticize the bamboo components. Therefore, only a few samples of all those pressed with 0% MC presented enough integrity to be tested.

According to the Figure 3.4, the water absorption and thickness swelling results indicate that equilibrium moisture content is increased with densification since samples with 0, 5, and 10% initial moisture content are able to absorb more water after reaching the equilibrium. This increase in equilibrium moisture content is associated with modification in the bamboo microstructure during the pressing. This fact is confirmed by water absorption tests, in which every densified sample except those with 20% initial moisture content presented higher absorption values compared to un-densified bamboo. The high standard deviation hampers any statistical conclusion and no significant difference is noted between densified samples with 0, 5 and 10% moisture content.

3.3.4.2. Bending-fractured microstructural morphologies

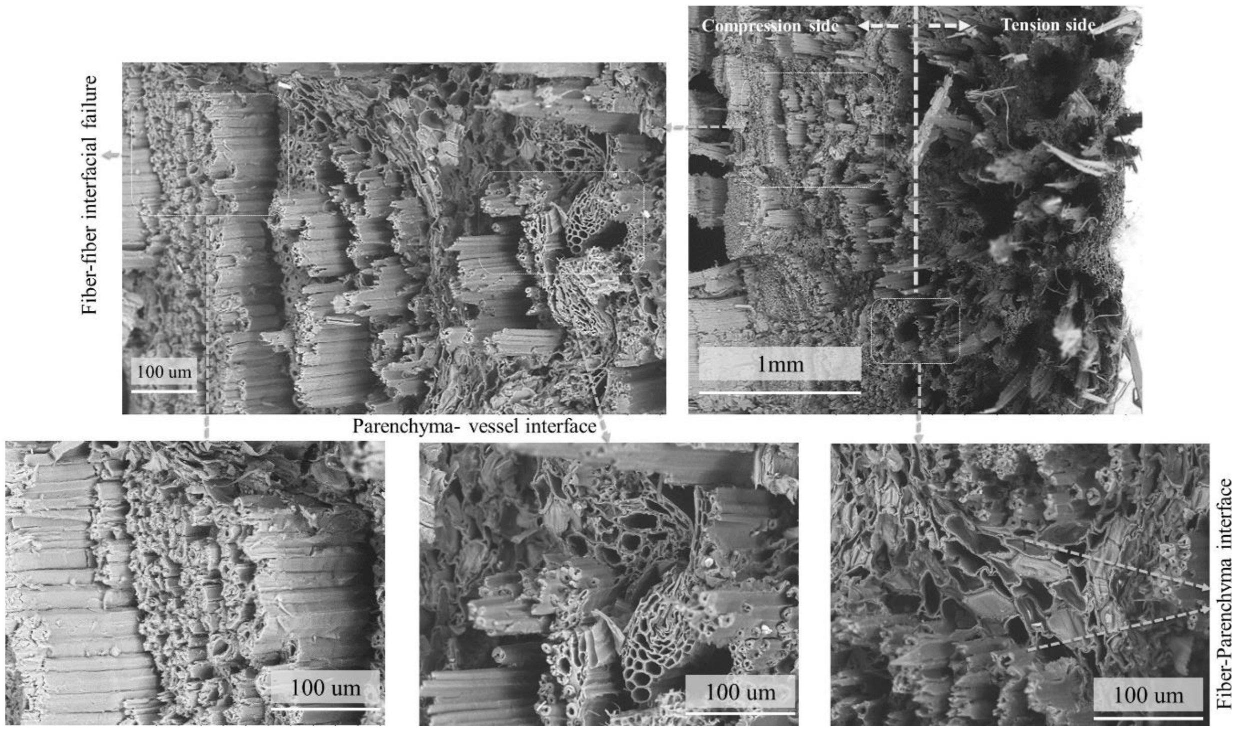
The bending-fractures investigated the fracture mechanism and determined the fracture sites, as well as other fractographic features such as pull out of the fibers. The bending fracture sections for un-densified and densified samples are shown in Figures 3.9 and 3.10 respectively. Samples densified with 10% MC has been analyzed in this part of the study since the best bending performance was achieved under this condition. Based on this observation, for both un-densified and densified bamboo samples, the fractured surface has been divided equally into two sections: compression and tension sides.

Figure 3.9 - Bending fracture section of un-densified bamboo showing the compression and tension zones.



Source: (KADIVAR et al., 2019a)

Figure 3.10 - Bending fracture section of 10% MC densified bamboo showing the compression and tension zones.



Source: (KADIVAR et al., 2019a)

At un-densified bamboo fracture section (Figure 3.9), four fracture behaviors are recognized: fiber-parenchyma interface failure, parenchyma failure, parenchyma-vessel interface failure, and fiber-fiber interfacial failure. During the bending test, at first, parenchyma fails and after it anchors by fibers. In the tension zone, the fibers are pulled out individually, while in the compression side, they are pulled out at first as fiber bundles and after individual fibers failed. The fracture in parenchyma shows a smooth slide between the cells characterizing an intercellular failure. Parenchyma sliding also happened in the parenchyma-vessel interface.

It can be reasonably assumed that the bending induced cracks for un-densified bamboo were mainly caused by the fiber-parenchyma interface fracture, which has been proved by Tang et al. (2019) to be very weak (CHEN et al., 2018b).

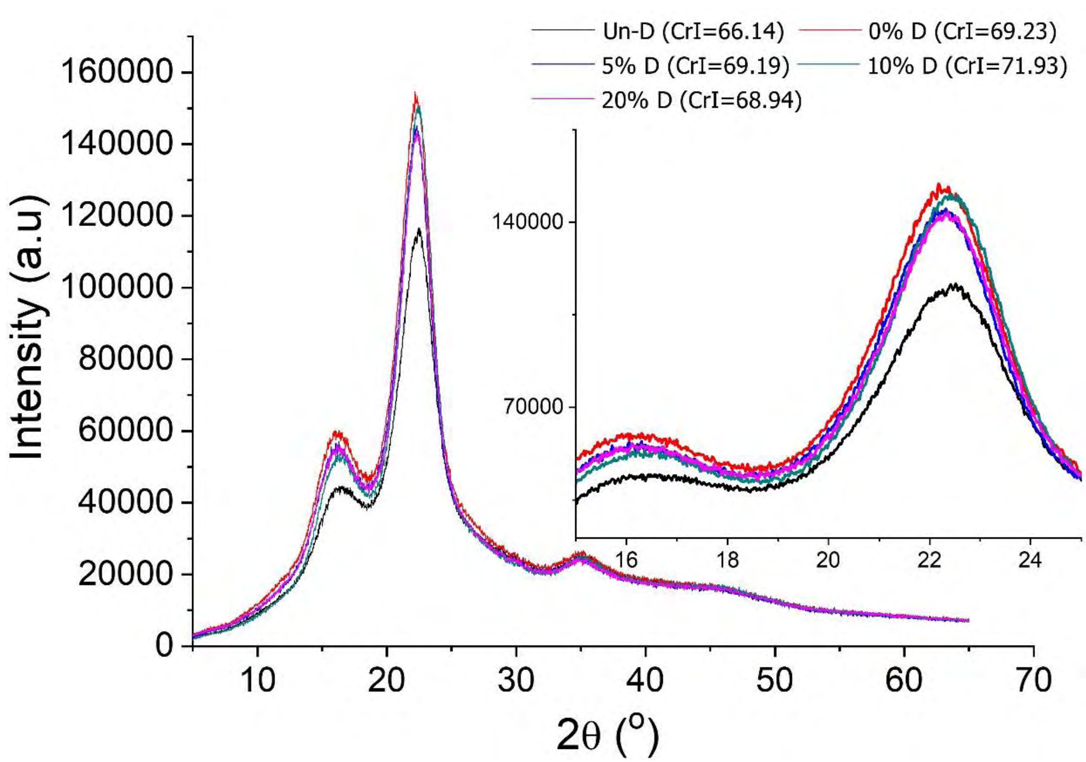
Bending fracture images of densified samples in the tension side show expansion of the section and a high amount of porosity in this part. Few individual fibers can be seen, which shows that in tension side fibers act as bundles. The interface of fibers-parenchyma shows good adhesion and samples failed mostly in fibers or parenchyma parts. However, in the compression side, the bending induced crack propagation through the compacted fibers, which caused fiber breakage. Although the parenchyma in both tension and compression side have been deformed and smashed because of densification, the failure mode in this case also is due to the sliding between the parenchyma cells (intercellular). This knowledge helps to understand how the TM process can improve the material quality during the bending behavior. The increase of fiber-parenchyma interface adhesion observed by the SEM analysis of the densified samples with 10% MC corroborates with the increase of LOP and MOR obtained through three-point bending tests.

3.3.5. XRD results

Cellulose is the main responsible for the strength of lignocellulose materials. It is well known that the relationship between the amorphous and ordered (crystalline region) parts of cellulose may influence the mechanical properties of wood or bamboo (JIANG et al., 2018; TANG et al., 2019). Since the TM process involves temperature, in order to track any change on the cellulose structure of the bamboo after densification, the crystallinity index (CrI) of the un-densified and densified samples was determined.

Figure 11 shows the XRD patterns of the bamboo densified and un-densified samples. All diffraction patterns exhibit the highest peak at 22.42° (002) and two weaker diffraction peaks at 16.34° (101) and 35.04° (040), which are assigned to cellulose I structure (NISHIYAMA et al., 2003; TANG et al., 2019). Densified bamboo samples with any MC display similar diffraction patterns as that of the un-densified counterpart. However, when the bamboo sample is densified, there is an increase in the (002) plane intensity. This difference is clear in the magnified embedded graph of Figure 11, which shows the distinct peaks in the 15-25° 2θ range. The crystallinity indexes calculated by the Seagal method for all the moisture conditions are shown in the top part of Figure 11. All densified bamboo samples showed higher CrI than un-densified samples. It can be inferred that the TM process had a small effect on the cellulose structure, probably because of the temperature used for the process. Tang also observed the increase of CrI in bamboo samples (*Phyllostachys heterocycle)* heat-treated in Tung oil, although different absolute CrI were achieved. In this case, using a treatment temperature of 140°C, the increase of cellulose crystallinity was followed by an increase in the MOR and MOE (TANG et al., 2019). Using a different method for the determination of CrI, Fatriasari also observed an increase of CrI in *D.* asper bamboo after a heat treatment at 330°C by a microwave oven (FATRIASARI et al., 2016). Among densified samples, the ones with 10% MC presented the highest CrI, which also presented the best bending mechanical properties. Although the increase of mechanical properties observed in the densified samples may be attributed to the increase of density, the change in the cellulose structure can also be affecting positively the mechanical behavior of the studied bamboo.

Figure 3.11 - XRD pattern of all the investigated conditions (un-densified and densified with 0%, 5%, 10% and 20% initial MC)

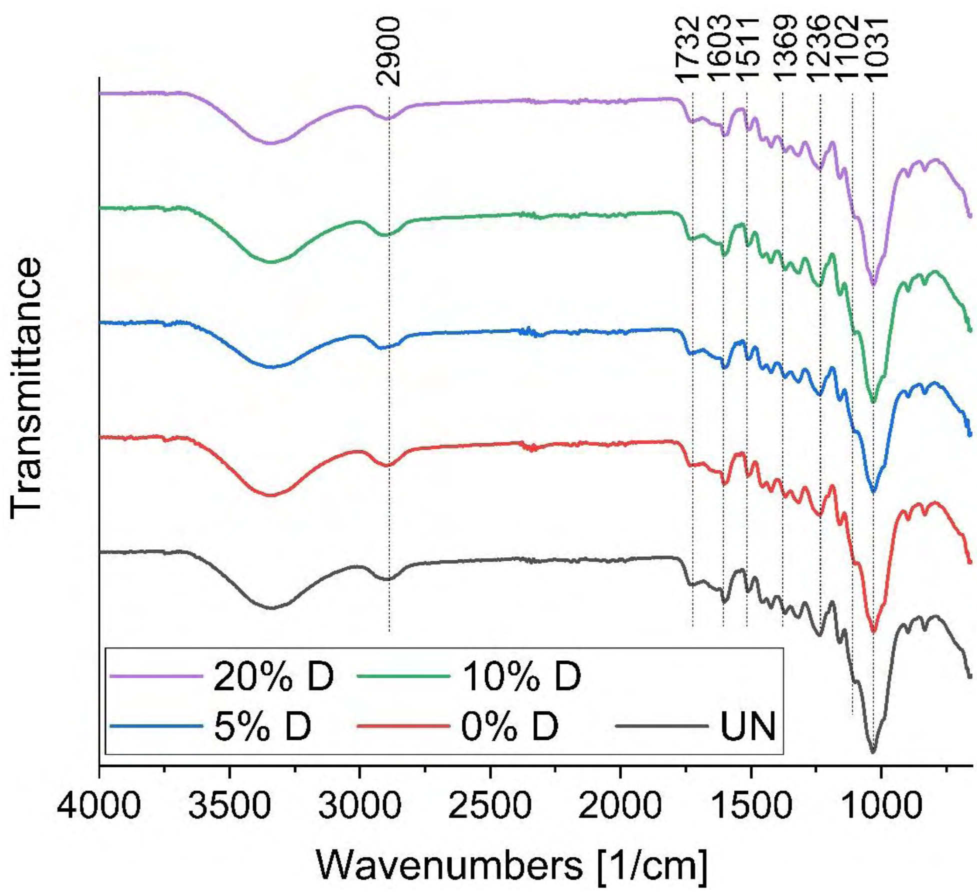


Source: (KADIVAR et al., 2019a)

3.3.6. FTIR analysis

FTIR spectroscopy combined with attenuated total reflection (ATR-FTIR) is a method to analyze bamboo component structures and chemical functional groups. Therefore, this method can show chemical changes induced by TM bamboo process. Figure 12 presents the FTIR spectra of un-densified and densified bamboo with different MC. According to the literature, the functional region in the 3800–2700 cm^{-1} range can be assigned to different CH stretching vibration groups and O–H stretching absorption bands (NIOKHOR et al., 2011). The FTIR spectra of all the samples present similar patterns. The fingerprint region, from 1800 to 600 cm^{-1}, which is assigned to different stretching or bending vibrations of the functional groups of wood components, is summarized in Table 2 (XU et al., 2013).

Figure 3.12 - FTIR spectra of densified and un-densified bamboo samples



Source: (KADIVAR et al., 2019a)

Table 3.2 - Summary of the main functional groups found in wood and bamboo (XU et al., 2013).

Wave number (cm^{-1})	Functional group	Assignment
1737	− COOH (C = O)	free carbonyl groups, Stretching of acetyl or carboxylic acid (hemicelluloses)
1650	C = O	quinines and quinine methides, adsorbed water
1601	C = C	Aromatic ring (lignin)
1511	C =C	Aromatic ring (lignin), stronger guaiacyl element than syringyl
1460	C‒H	Asymmetric bending in CH3 (lignin)
1425	CH2	Aromatic skeletal vibrations (lignin) and C H deformation in plane (cellulose)
1375	CH	C H deformation in cellulose and hemicellulose
1328	O‒H	phenol group (cellulose)
1248	CO	Guaiacyl ring breathing with CO-stretching (lignin and hemicelluloses), esters
1163	C − O − C	Carbohydrate
1120	C‒H	Guaiacyl and syringyl (lignin)
1039	C‒O, C‒H	Primary alcohol, guaiacyl(lignin)
896	C‒H	C H deformation in cellulose

Source: (XU et al., 2013).

FTIR fingerprint region contains many well-defined peaks that provide abundant information on various functional groups present in wood constituents. Table 3 presents a quantitative comparison of samples in terms of chemical components changes during TM process at various MC. Since C-H aliphatic stretching (around 2900 cm^{-1}) is more stable than C–OH, C–O–C, and R–COO–R bands and can be expected to stay intact during heat treatment (GAO; GUO; LUO, 2018) in this table the ratios between the intensities of peaks at 1732, 1603, 1511, 1369, 1236, 1102, and 1031 cm^{-1} over the peak at 2900 cm^{-1} were calculated. There is no significant difference in the relative intensities of these characteristic bands between un-densified and densified bamboo with different MC. These results are consistent with the results of Gao et al. (2018), who pressed Moso bamboo (*Phyllostachys pubescens*) at different temperatures, time and moisture content and found that polysaccharide (especially hemicelluloses) only underwent a progressive thermal degradation during TM process at a temperature higher than 180°C. The samples pressed at 120°C did not show any considerable difference compared to the reference (GAO; GUO; LUO, 2018). Tang et al. (2019) also did not observe considerable chemical changes of Moso bamboo treated up to 200°C in Tung oil. At 140°C, only a slight increase in the lignin content was detected, caused by the decrease of hemicellulose content. The amount of cellulose stayed constant for all the treatments (TANG et al., 2019).

Table 3.3 - Summary of the main functional groups found in wood and bamboo.

Samples	I_{1732}/I_{2900}	I_{1603}/I_{2900}	I_{1511}/I_{2900}	I_{1369}/I_{2900}	I_{1236}/I_{2900}	I_{1102}/I_{2900}	I_{1031}/I_{2900}
UN	0.99	0.97	0.98	0.95	0.92	0.86	0.77
0	1.00	0.98	0.99	0.96	0.93	0.87	0.77
5	0.99	0.97	0.98	0.96	0.94	0.89	0.80
10	0.99	0.97	0.99	0.95	0.93	0.87	0.77
20	1.00	0.98	0.99	0.96	0.93	0.87	0.78

I_{1732}/I_{2900}, I_{1603}/I_{2900}, I_{1511}/I_{2900}, I_{1369}/I_{2900}, I_{1236}/I_{2900}, I_{1102}/I_{2900}, I_{1031}/I_{2900}, refer to the ratio of the intensities of peaks at 1732, 1603, 1511, 1369, 1236, 1102 and 1031 cm^{-1} over the peak at 2900 cm^{-1} respectively.

Source: (KADIVAR et al., 2019a)

3.4. Conclusions

The influence of the starting MC on the densification process of D. asper bamboo was assessed. The densification degree was affected by the different testing conditions. The higher starting MC resulted in higher densification degree using the same pressing procedure, ranging from 23.1 to 31.2 %. Regarding the position of each layer along with the bamboo wall thickness, higher densification was achieved in the inner layer followed by middle and outer layers. However, the samples with 0 and 5% MC presented macro cracks after the TM process while the samples with 20% MC exhibited an irregular thickness throughout their length and width caused by blocked water steam within bamboo’s structure.

The spring-back effect after densification was almost negligible for all the analyzed conditions. However, water immersion negatively affected the dimensional stability (thickness swelling) of the densified samples in comparison with the reference. On the other hand, considering only the densified conditions, the higher the starting moisture content, the lower the thickness swelling and water absorption.

The variation of the starting moisture content also affected the bending and dynamic modulus performance, whereas the optimum properties were obtained by the samples with 10% MC, increasing the MOR and MOE by 56 and 41% respectively, in comparison with un-densified samples. Additionally, fracture analysis revealed different failure modes, predominantly parenchyma failure, and fiber-fiber interfacial failure, after bending as a consequence of densification. The interface between parenchyma and fiber bundles was modified in a way that the linkage between both components increases after densification. Through SEM analysis, it was also observed microcracks mainly in 0 % and 5 % MC samples, which can justify the lower bending strength and the unstable behavior after immersion in water.

Thermo-mechanically bamboo densification process at 140°C caused minor changes in the chemical composition of bamboo as determined by FTIR spectroscopy. However, XRD analysis showed that the cellulose crystallinity indexes of the densified samples were higher than the un-densified ones. The highest value was observed for the 10% MC condition, with a CrI of 71.9%.

According to the overall results, the control of the initial moisture content of bamboo is essential to improve the efficiency of the TM process. In this study, the optimum condition in terms of mechanical and physical properties was found to be a starting moisture content of 10%, which is close to the moisture content of commercial bamboo and wood materials (around 12%) (KADIVAR et al., 2019a).

4. CHAPTER 4 – Optimization process of thermo-mechanical densification of bamboo

This chapter has been submitted to the construction and building materials journal, manuscript number: CONBUILDMAT-D-20-11235.

Abstract

Due to its reliability, strength, and ease of access, bamboo has become an attractive material for engineering applications. However, heterogeneous properties and durability issues still hinder the widespread use of bamboo as a building material. Thermo-mechanical treatment is a method to decrease the heterogeneity of bamboo culms and enhance mechanical properties and durability, but it may negatively impact dimensional stability. The objective of this study was to achieve the minimum spring back, water absorption, and thickness swelling for densified bamboo. Accordingly, the behavior of bamboo samples subjected to different thermo-mechanical (TM) treatments using a two-step analysis was investigated. In the first step, the optimum TM treatment for achieving the highest critical densification degree (DD) without shear failure was determined. In the second step, the three key elements of dimensional stability were studied for this optimum case. According to the first step results, the maximum achievable DD in which no shear failure happens and the texture is not disturbed is about 43.6%, and it can be obtained at 200°C with a compression rate of 2 mm/min. X-ray densitometry analysis confirmed that DD of around 50% achieved the highest value of density, 1.30 g.cm-3. The results of step 2 revealed that the lowest values of spring back, water absorption, and thickness swelling, 4.72%, 23.80%, and 17.70% respectively, for densified bamboo occur when the densification process is conducted at 200°C and adfopting a compression rate of 6.7 mm/min. In conclusion, by manipulating and optimizing process parameters, the dimensional stability and final quality of densified bamboo can be improved, opening new opportunities for this class of material.

Keywords: Bamboo Densification; Thermo-Mechanical treatment; Dimensional Stability

4.1. Introduction

Bamboo, as a renewable and environmentally friendly resource with a history of thousands of years, has been considered very popular around the world for its nutritious, pharmaceutical, textile, and construction applications (AWOYERA; UGWU, 2017; NIRMALA et al., 2018; DARIA; KRZYSZTOF; JAKUB, 2020; SILVA et al., 2020; SUN; HE; LI, 2020). In the construction business, especially in tropical and sub-tropical regions, bamboo culms play an important role in the industry. From scaffolding and water piping to shuttering and reinforcements for concrete, bamboo as a strong, light, and versatile material is always available (GHAVAMI, 2005; ATANDA, 2015; CHAOWANA; BARBU, 2017). However, there are certain difficulties and drawbacks for using bamboos that have concerned researchers and engineers, such as lack of standardization, flammability, jointing, and durability (JAYANETTI; FOLLETT, 2008; HARRIES; SHARMA; RICHARD, 2012; BRINDHA et al., 2017; PARASKEVA; GRIGOROPOULOS; DIMITRAKOPOULOS, 2017; GAUSS; KADIVAR; SAVASTANO, 2019; GUO et al., 2019). To solve these difficulties, bamboo undergoes special treatments and processes.

Densification processes can be applied to use bamboo more efficiently as an industrial material in modern constructions. Besides, by densifying the material, and hence increasing its density, mechanical strength can be considerably improved. For instance, by 20-100 % increment of the density, the mechanical strength in bending increased by 15-100% for the *Phyllostachys edulis* and *Dendrocalamus asper* species which was addressed in several types of research (TAKAGI et al., 2008; K.E. SEMPLE, F.A. KAMKE, A. KUTNAR, 2013; DIXON et al., 2016a; KADIVAR et al., 2019a, 2019b). The applied process and utilized parameters such as temperature, pressure, densification degree (defined in equation 3.1), and relaxation time determine the results obtained and the quality of the final product.

Thermo-Mechanical densification technique (TM) is one of the accepted environmentally friendly methods (KADIVAR et al., 2019a, 2020a; MANIA; MAJKA; ZBOROWSKA, 2019), in which bamboo is mechanically compressed in the radial direction at an elevated temperature with the aid of an initial moisture content (MC). Placing bamboo in such an environment makes it a viscoelastic material. At low temperatures, bamboo presents a high strength and modulus, which are gradually reduced by increasing the temperature. At temperatures above the glass transition temperature (Tg) of lignin, the mechanical behavior of bamboo changes to a rubbery state (MATAN; KYOKONG; PREECHATIWONG, 2007). Tg is reversibly affected by the moisture content, in which higher moisture leads to lower Tg. Therefore, temperature and moisture content are the main parameters for the plasticization of bamboo (MATAN; KYOKONG; PREECHATIWONG, 2007; KADIVAR et al., 2020b).

Takagi et al. (2008) (TAKAGI et al., 2008) TM densified bamboo *Phyllostachys bambusoides* at different temperatures ranging from room temperature to 220 °C. Their results showed that the highest the temperature, the highest the density, and flexural modulus. However, the highest flexural strength was achieved at 160°C. Although it is called Thermo-Mechanical process, chemical mechanisms will also be involved at temperatures above 160 °C. According to Matan 2007 (MATAN; KYOKONG; PREECHATIWONG, 2007), due to the Tg dependency on the MC applying a temperature in the range of 100-170 °C requires an approximate MC between 2-8 % to plasticize *D. asper* bamboo. Kadivar et al. in 2019 (KADIVAR et al., 2019a) stated that a low moisture content may cause cracking, while MCs above 15%, trap the water inside the bamboo cells and result in heterogeneous densification.

The mentioned parameters and several more factors such as the compression rate, which is rarely mentioned in the literature, must be controlled during the process to achieve the desired quality. Reviewing the literature of bamboo densification suggests that densification degree (DD) is one of the most important parameters of the process (K.E. SEMPLE, F.A. KAMKE, A. KUTNAR, 2013; KADIVAR et al., 2020b), since it is directly correlated to the mechanical properties. However, as expected, there is a limitation to this enhancement, which in this paper is called the critical densification degree (DDcr), which is defined as the maximum DD of the material without shear failure. In a previous study, Semple et al. (2013) (K.E. SEMPLE, F.A. KAMKE, A. KUTNAR, 2013) found that the compression of 50% was optimal for bamboo Moso species whereas compression up to 33% of the thickness, caused excessive lateral displacement and shear damages to the tissue. Their results reflected the effect of fixed parameters, i.e.,processing at 170 °C, and applied steam pressure of 775 kPa for 13.3 min. By changing each of these parameters, the optimum DD might change. Theoretically all of these effective parameters of the densification process are involved and there is a knowledge gap about the correlation of DDcr and other parameters such as temperature and rate of pressure.

Relaxation time is another important parameter, which depends on DD, temperature, and compression rate. In the case of wood, the stress relaxation curves above 100 °C are quite different in shape from those below 100 °C, showing a rapid decrease in stress with increasing temperature . The incomplete relaxation time for the permanent fixation of bamboo densification causes strain recovery called spring back.

Although there are several studies to optimize some of these parameters, there is no consolidated information that takes into account all of the effective parameters and investigates their correlation during processing. It should be noticed that optimized parameters vary with species. Tanaka et al. (2006) (TANAKA et al., 2006) applied the same TM densification method on two different bamboo species, Moso (*P. edulis*) and Madake (*P. bambusoides*) and achieved dissimilar results.

Almost all publications related to densified bamboo demonstrate that TM process increases mechanical performance, while the biggest challenge of densified bamboo is about its dimensional stability, which is sacrificed by densification (KADIVAR et al., 2019a). This study aims to answer two main questions. First: using a determined temperature and compression rate, what is the optimum densification degree of bamboo so that no shear failure happens, in other words, to what extent can bamboo be compressed without structural failure? Second: what is the more appropriate practice to achieve the best dimensional stability (lowest spring back, water absorption, and swelling) ? Those two approaches are the key aspects of better understanding the phenomena and clarifying them enriches the knowledge of the bamboo densification TM process and could support and encourage the widespread adoption of structural densified bamboo products.

4.2. Materials and Methods

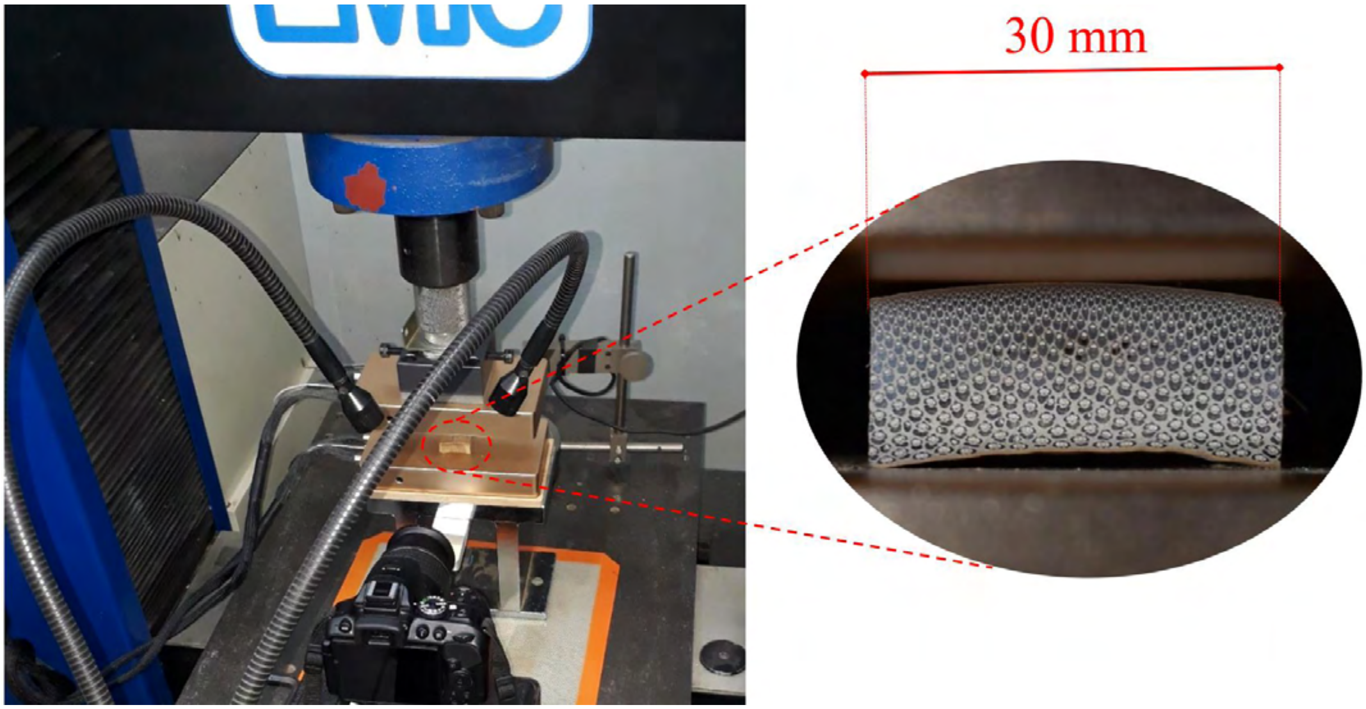
4.2.1. Samples

The tests were carried out on samples of bamboo *Dendrocalamus asper* species harvested from an experimental field at the University of São Paulo Campus (21°58′53.5″S 47°26′03.3″W). The samples possess the square dimensions of 30 mm in length and width, and the thickness varies between 10-13 mm (Figure 4.1). For all the specimens, 100% of their wall thickness was used for the test. The reason for using the whole thickness is that this process is thought to be applied in an industrial or pilot scale, so it is crucial to reduce the time and steps of the process. If the samples pass through the thicknesser before densification, some parts of the material will be lost, reducing the material usage efficiency, and there will be two processes to adjust the dimensions. Before the extraction of the samples, the bamboo poles were treated using a disodium octaborate tetrahydrate (DOT) solution of 8% in a pilot-scale immersion tank, as described in Gauss et al 2020. According to Gauss 2019 (GAUSS; KADIVAR; SAVASTANO, 2019), this chemical treatment does not have a significant influence on the mechanical performance of bamboo.

4.2.2. Design of experiments

A new test apparatus (Figure.1) was adapted using the combination of small press plates that can be heated up to 200 ^oC, a EMIC universal testing machine to apply the load and control the compression rate and a camera for observing vertical and lateral deformation during the densification process. This set up simulates an open thermo-mechanical system and enabled the collection of load-deformation data during a hot press. Therefore, it is possible to understand the mechanical behavior of bamboo while increasing pressure at elevated temperatures.

Figure 4.1 - Experimental set-up for the densification process.



Source: Author's authorship.

The experiments have been performed in two steps to answer questions one and two mentioned in the introduction section.

4.2.2.1. Step 1 of experimental design

In this step, the bamboo specimens were subjected to radial compression at different temperatures and compression rates until the samples collapsed (Table 4.1). The force-deformation graphs were obtained for all cases. In addition, for each running test, the camera recorded deformation in the radial and lateral directions versus the time. The active parameters in this step are temperature and compression rate with DDcr as the response. DDcr is defined as DD that corresponds to the lateral deformation initiation, which occurs when the material fails in shear. The temperature and pressure rate are the parameters that are deemed to be optimized for achieving the highest DDcr.

4.2.2.2. Step 2 of experimental design

In step 2, DD has been fixed according to the obtained results of step 1. After achieving the desired DD (at a specific displacement), this position was maintained for 1 hour to analyze the bamboo relaxation, which is related to the decrease of load with time. In this step, the responses are spring back (SB), water absorption (WA), and thickness swelling (TS), which need to be minimized. The variables' ranges and responses for each step are shown in table 4.1.

Table 4.1 - TM parameter levels and responses for each step

Step	Variable							response		
	Temperature (°C)			Compression rate (mm/min)			DD (mm/mm %)			
Step 1	30	160	200	2	4	8	–	DD (mm/mm)		
Step 2							According to the results of step 1	WA	TS	SB

DD: Densification Degree, WA: Water absorption, TS: Thickness Swelling, SB: Spring Back

4.2.3. The density analysis by X-ray densitometry

Two bamboo culm samples from each of the four treatments were selected: (Treat. 1) control and (Treat. 2) 30, (Treat. 3) 50, and (Treat. 4) 70% of DD, densified at 160 ºC and compression rate of 4 mm/min. To prepare the bamboo culm cores for densitometry analyses, the bamboo culm samples previously glued in wooden support were transversely cut (1.5 mm thickness) with a parallel double circular saw and conditioned in a climatic chamber at 20°C and 60% relative humidity until reaching a stable moisture content of 12%. The thin bamboo culm samples were then scanned with a calibration scale of cellulose acetate using an X-ray densitometry equipment (Faxitron MX20-DC12, Faxitron X-Ray, Illinois, USA). The bamboo culms digital X-ray images were analyzed in 3 different positions by WinDendro® software (Regent Instruments Inc.) producing micro density profiles and the mean, maximum and minimum density values of each treatment. Additionally, the bamboo digital X-ray radiography was used to obtain the bamboo anatomical microstructure image. The software MultiSpec allowed an accurate quantitative determination of the bamboo culms tissues, as well as the anatomical modifications induced by the treatments and. The analyzes were performed according to the procedures at the Laboratory of Wood Anatomy, Identification and X-Ray Densitometry, Department of Forest Sciences at the ESALQ/University of São Paulo, Piracicaba, SP. (TOMAZELLO et al., 2008).

4.2.4. Dimensional Stability

For the spring-back test, the densified samples were placed in an environmental chamber at (25 ± 5) °C and (60 ± 3) % RH for four weeks. Then the spring-back factor was calculated from equation 3.4, in which t_1 (the thickness immediately after densification), and t_2 (the thickness after conditioning the samples at the chamber) were required.

The water absorption (WA) and thickness swelling (TS) tests were carried out based on ASTM D1037-12 for the bamboo specimens before and after densification [25]. First, the specimens were dried out at (105 ± 2) ^{o}C before measuring the weight and volume. Afterwards, they were fully immersed in water at (22 ± 1) ^{o}C, and the weight and thickness changes were measured after 1 h and 24 h. Subsequently, TS and WA were calculated from Equations (3.5) and (3.6), respectively. For these equations, m_0 and m_2 that are the masses before and after the immersion of the samples in water after 1 h and 24 h respectively, and t_2 and t_3 that are the thicknesses before and after the test, respectively, are required.

4.2.5. Statistical analysis and optimization process

MINITAB® Release 14 Statistical Software was used to perform the statistical analysis of the results. Moreover, Analysis of variance (ANOVA) ($p < 0.05$) and Tukey's test were applied to verify the effect of the treatment conditions.

Response Surface Methodology (RSM), which is a collection of mathematical and statistical techniques, was used in the two steps to explore the interactions of significant conditions and optimize the bamboo densification parameters. This technique is one of the main functions for design of experiments in Minitab software to analyze problems in which a response of interest is influenced by several variables (CASTILLO, 2007). There were 9 experimental steps with 5 replicates, requiring a total of 45 densified bamboo samples, under temperature variations of 30, 160, and 200 ^{o}C, and compression rates of 2, 4, and 8 mm/min. In addition to these samples, several random temperature treatments (100 and 140 ^{o}C) and pressure rates (2 and 4 mm/min) were included in the model for verification purposes. The relationship among the responses and variables, and also the optimum range for each variable, were demonstrated by the response surface and contour plots. In addition, a regression model has been used to calculate the results. For step 2, since there is more than one response involved, the Multi Response Optimization Method (MROM) was applied.

4.3. Results and discussion

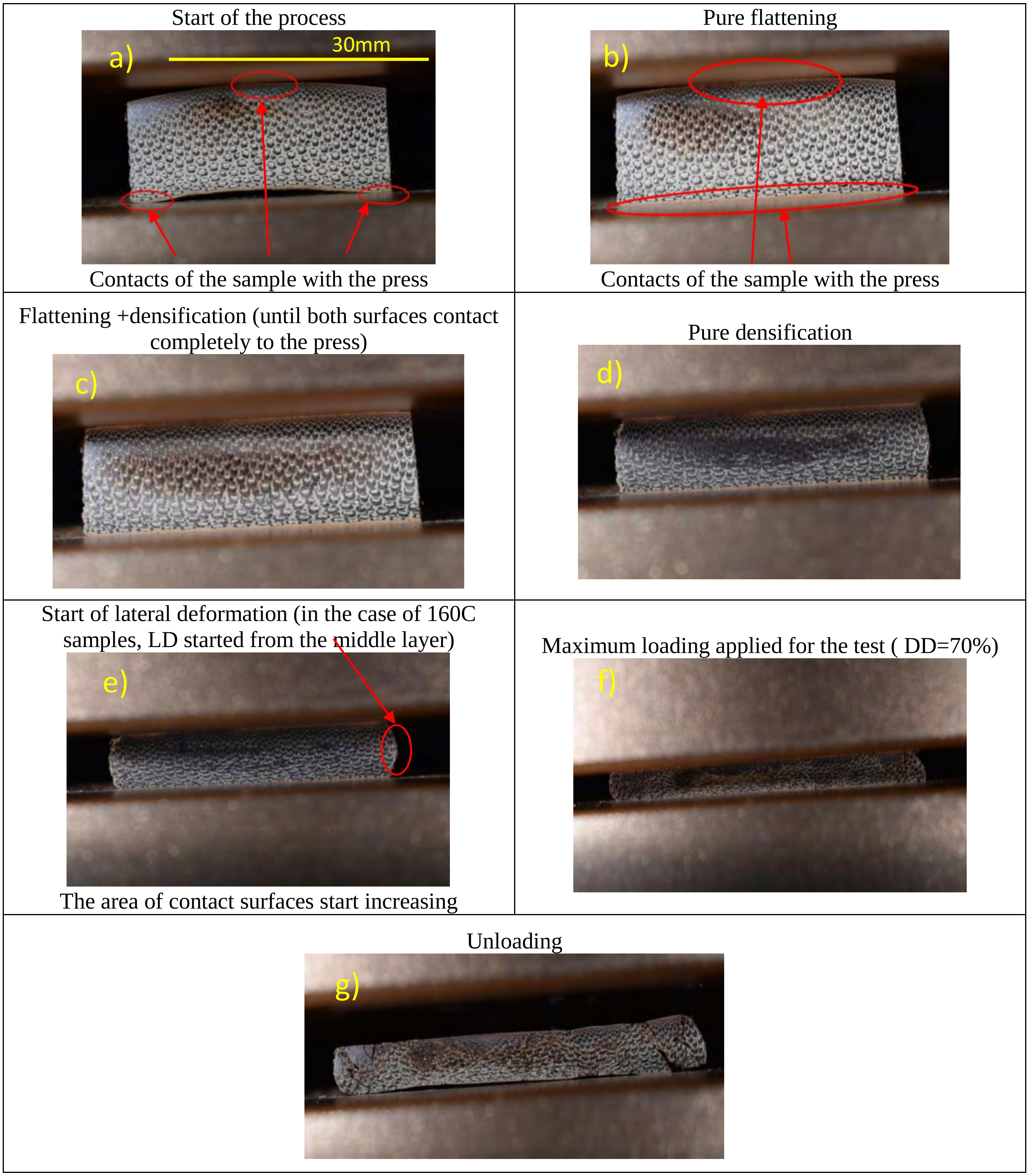
4.3.1. The behavior of bamboo under hot compression

A camera recorded the 2D geometrical change of the samples during the densification process. Since the length did not change during compression, only the width and thickness of the samples were extracted from the videos. Two videos are presented as supplementary material, one from the sample compressed at 30 ^{o}C (Video 1), and the other from the sample compressed at 200 ^{o}C with a compression rate of 8 mm/min (Video 2). Figure 4.2 also presents the step by step changes of the sample during the densification process at 160 ^{o}C with a rate of 8 mm/min. The observation of the videos indicated that:

- The behavior of bamboo under transverse compression, at different temperatures, was completely different. However, by changing the compression rate, no significant difference can be seen in the videos.
- Because of an existing small curvature in the samples, at the beginning of the load application, the bottom part is under tension while the upper part is under compression. Therefore small flattening occurs.
- At room temperature compaction at any compression rate leads to a deep crack at the middle of the sample. It is possible to see this phenomenon in video 1. However, increasing the compression temperature leads to a softer and smoother material flattening, which prevent the cracks, as can be seen in video 2.

- As figure 4.2, b shows for sample densified at an elevated temperature, after pure flattening, the bottom part of samples completely contact to the press plate. However, the top of the specimen is still slightly curved and requires more pressure to fully contact the top press plate.
- Increasing the load after pure flattening lead decreasing the thickness, yet continue the remain flattening (figure 4.2, c). In this step, flattening combines with densification.
- When the two surfaces are completely in contact with the press plates, the applied load causes pure densification and thickness decrease until achieving the critical densification degree, DD_{cr} (figure 4.2, d).
- After the DD_{cr}, increasing load induces lateral deformation, which is the result of fiber detachment and the collapse of the bamboo structure.
- The start of lateral deformation in the case of samples densified at room temperature occurs in the inner layer (bottom of the samples), which is in tension. While at elevated temperatures, samples start to expand in the middle section (see Figure 4.2 e).

Figure 4.2 - Step by step changes of the sample during the densification process at 160°C



Source: Author's authorship.

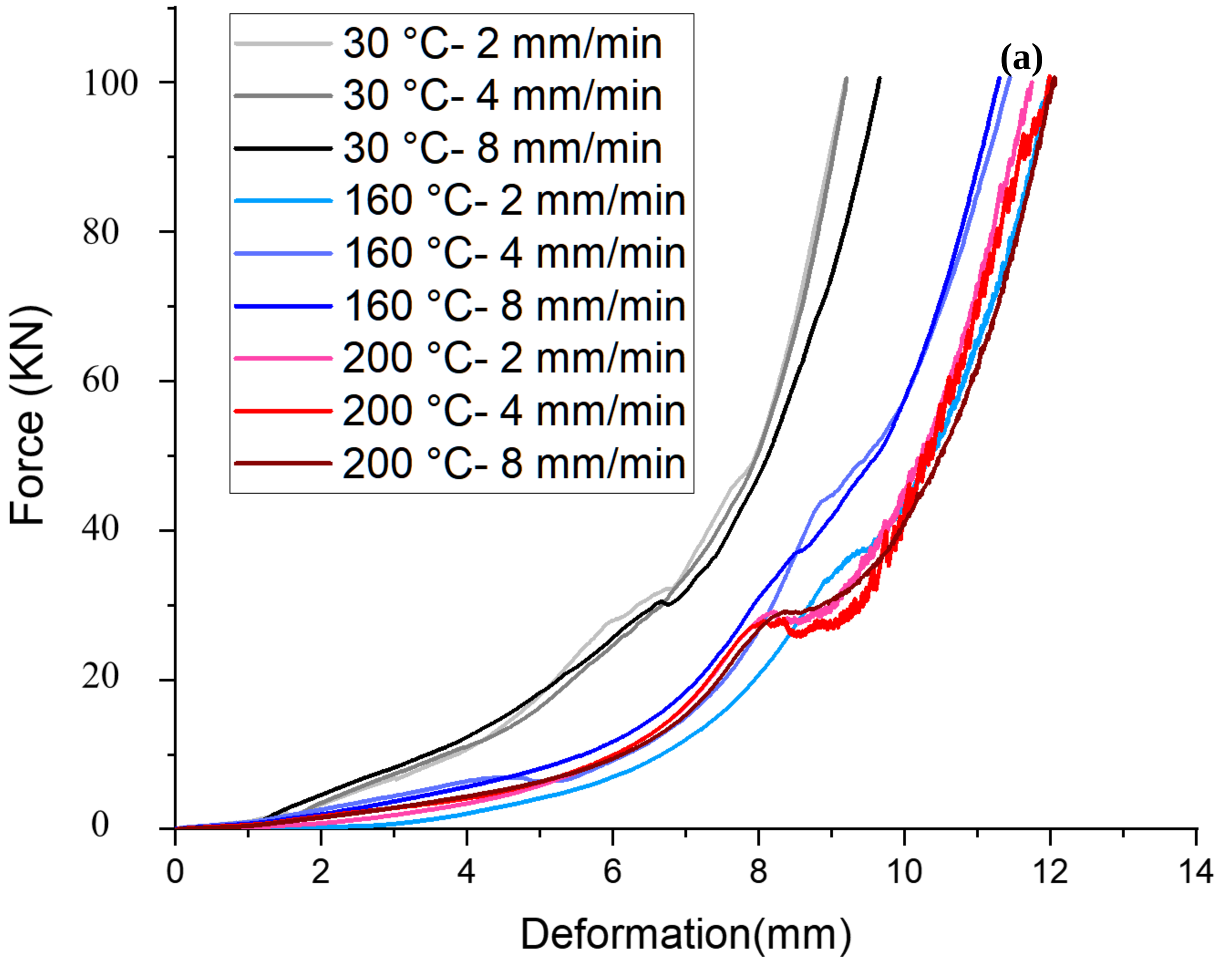
For each test of the first step, the applied force was plotted against the deformation of the sample. Figure 4.3-a demonstrates the load- displacement graph for 3 compression rates (2, 4, and 8 mm/min), and 3 different temperatures (30, 160, and 200ºC). According to the obtained results, compression at room temperature requires higher loads compared to the other two temperatures. This phenomenon is distinguishable in the graphs. However, the compression rate has little effect on the load-deformation behavior, and it is almost negligible in all cases. The initial sample curvatures have little influence on the curves since the maximum curvature of the middle of the samples does not exceed 2 mm.

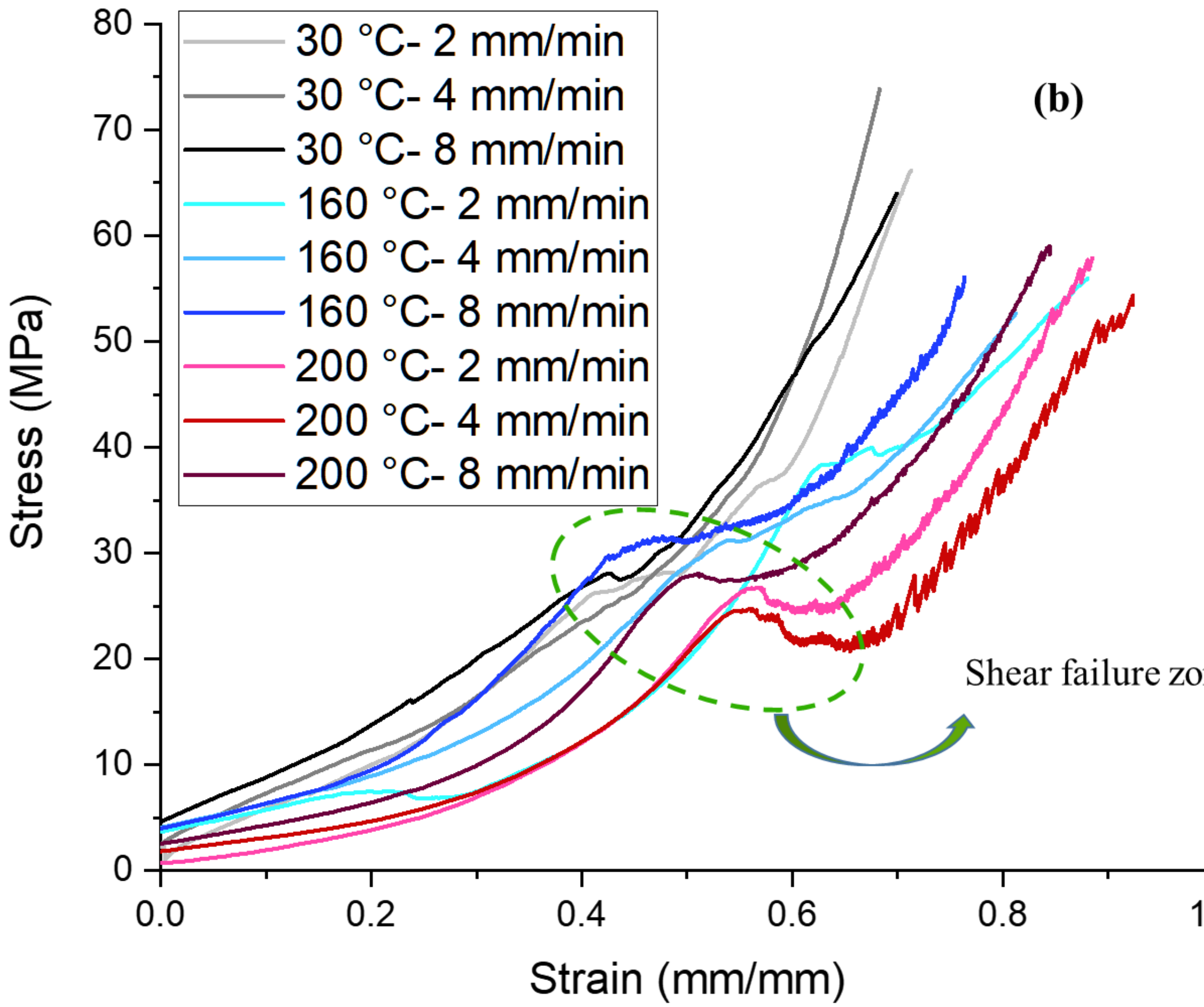
To obtain the stress-strain graphs, the effective area during the compression was calculated using a video tracker software. By using the point mass tracking technique, the expansion width was correlated with time. Since the length difference was insignificant before and after the process, to calculate the area changes during compression, the length variations were neglected. Dividing the force by area, stress/strain graphs were obtained, which are presented in Figure 4.3-b. In these graphs, the flattening part, which happens at the beginning of loading, was excluded because it presents relatively high stress in the results due to the small contact face.

As can be seen in the stress/strain graphs, the slope of the graphs, which represents the modulus of elasticity, decreases with increasing the temperature. On the other hand, the compression rate brings about two different and opposing phenomena regarding the modulus of elasticity of the bamboo samples. First of all, when the compaction rate is higher, the samples do not have enough time to warm up, and the initial temperature profile is valid throughout the samples' thicknesses, which would cause a higher rate of stress to strain ratio. However, as the compaction speed reduces, not only the samples can have more time to warm up and show more elastic behavior, but also it may continuously dry the sample, which is considered as a rising factor for the elasticity modulus.

It can also be observed that shear failure starts in the strain range of 0.4-0.5, which is equivalent to a densification degree of 40% to 50%. The higher the temperature and the lower the compression rate, the higher the starting point of the shear failure.

Figure 4.3 - Load-deformation (a) and stress-strain (b) graphs of the bamboo samples densified at different temperatures and compression rates.



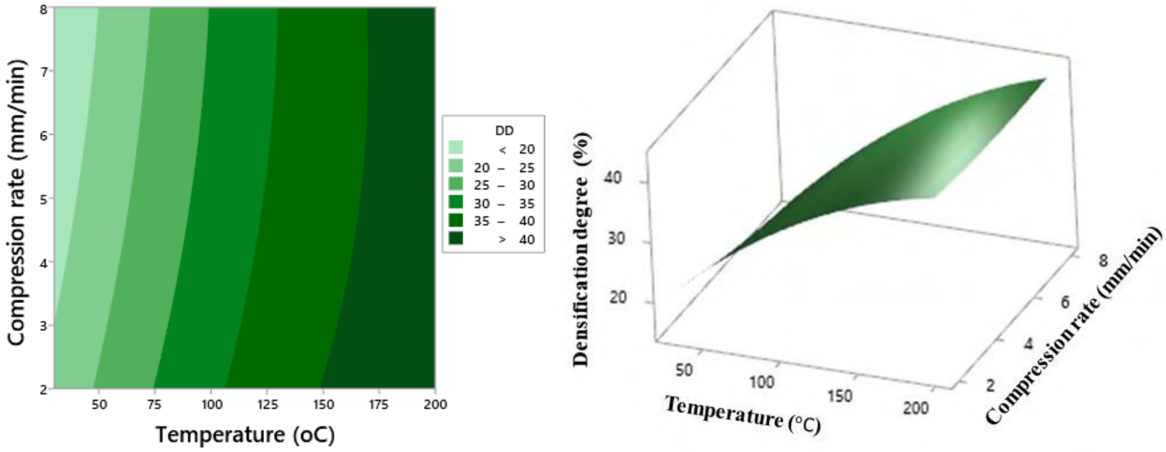


Source: Author's authorship.

4.3.2. Optimization results of step 1

Figure 4.4 demonstrates the results of DDcr versus temperature and pressure rate. The contour plot (figure 4.4a) shows the region with the highest DD in a darker color. This graph shows the best range of temperature and pressure rate where the material can reach the highest DD without shear failure occurrence. It can be seen that with an increment of temperature densification degree increases remarkably. On the other hand, by increasing the compression rate, a mild reduction on DD can be observed. The former phenomenon is related to the increase of bamboo plasticity with temperature, while the latter is assumed to occur because of the lack of time for relaxing the material during the compression.

Figure 4.4 - a) Contour plot and b) Surface plot of DD versus pressure rate and temperature,

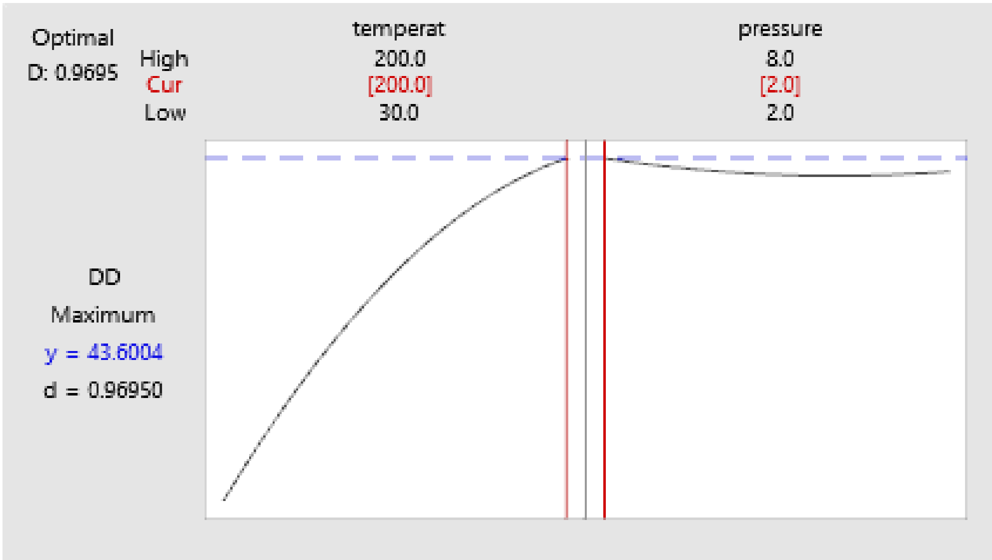


Source: Author's authorship.

Variance analysis indicates that according to F-value, the temperature is more significant than the compression rate. The mutual interaction of the temperature and compression rate is not also comparable to the temperature. The R-sq value of this analysis was 96.65, which shows the model explain quite well.

Figure 4.5 presents the optimization plot, stating that the maximum DD can be achieved at 200 ^{o}C with a compression rate of 2 mm/min. The relationship between these two variables are presented in Figure 4.5, where the optimum condition is indicated by the red lines. According to this statistical analysis, the maximum achievable DD is about 43.6 using the investigated conditions.

Figure 4.5 - The optimization plot obtained in step 1.



Source: Author's authorship.

Although the DDcr increases with temperature, at high compression rate explosion happened to some of the samples. Figure 4.6 shows the detachment of fibers out of the matrix at the temperature of 200 ^{o}C and a compression rate of 8 mm/min. The reason for this phenomenon is that when high compression rates are used, the water inside the material does not have enough time to get out of the pores. With water trapped inside the vessels, high steam pressure is generated, causing an explosion.

Figure 4.6 - bamboo defibrillation caused by steam pressure

Before densification After densification

Source: Author’s authorship.

4.3.3. Density profile and microstructure by X-ray densitometry of the samples of the step 1

Figure 4.7 demonstrates the values of average, maximum and minimum densities of bamboo samples; T1 is the control sample, and T2, T3, and T4 are the samples densified to nominal densification degrees of 30, 50, and 70%. The bamboo culm average density values obtained by X-ray densitometry indicate no statistically significant difference between T1-T4, with 0.74 and 0.92 g/cm^3, respectively. The same for treatments T2-T3, with 1.22 and 1.30 g/cm^3, respectively. However, T1-T2 and T2-T3 were statistically different. The increases in the bamboo mean density of the T2, T3, and T4 treatments were 65, 76, and 24%, respectively, in relation to T1.

For the treatments T1, T2, T3 and T4 the ***minimum mean*** density value of the bamboo culms were 0.30, 0.69, 0.83 and 0.35 g/cm^3, respectively; the ***maximum mean*** density value of the bamboo culms were 1.17, 1.45, 1.60 and 1.31 g/cm^3, respectively. There is a tendency to decrease the bamboo culm mean and minimum density up to the T3, followed by a drop in density at T4 . However, for the maximum mean density of bamboo culms , there are no marked differences in relation to the treatments.

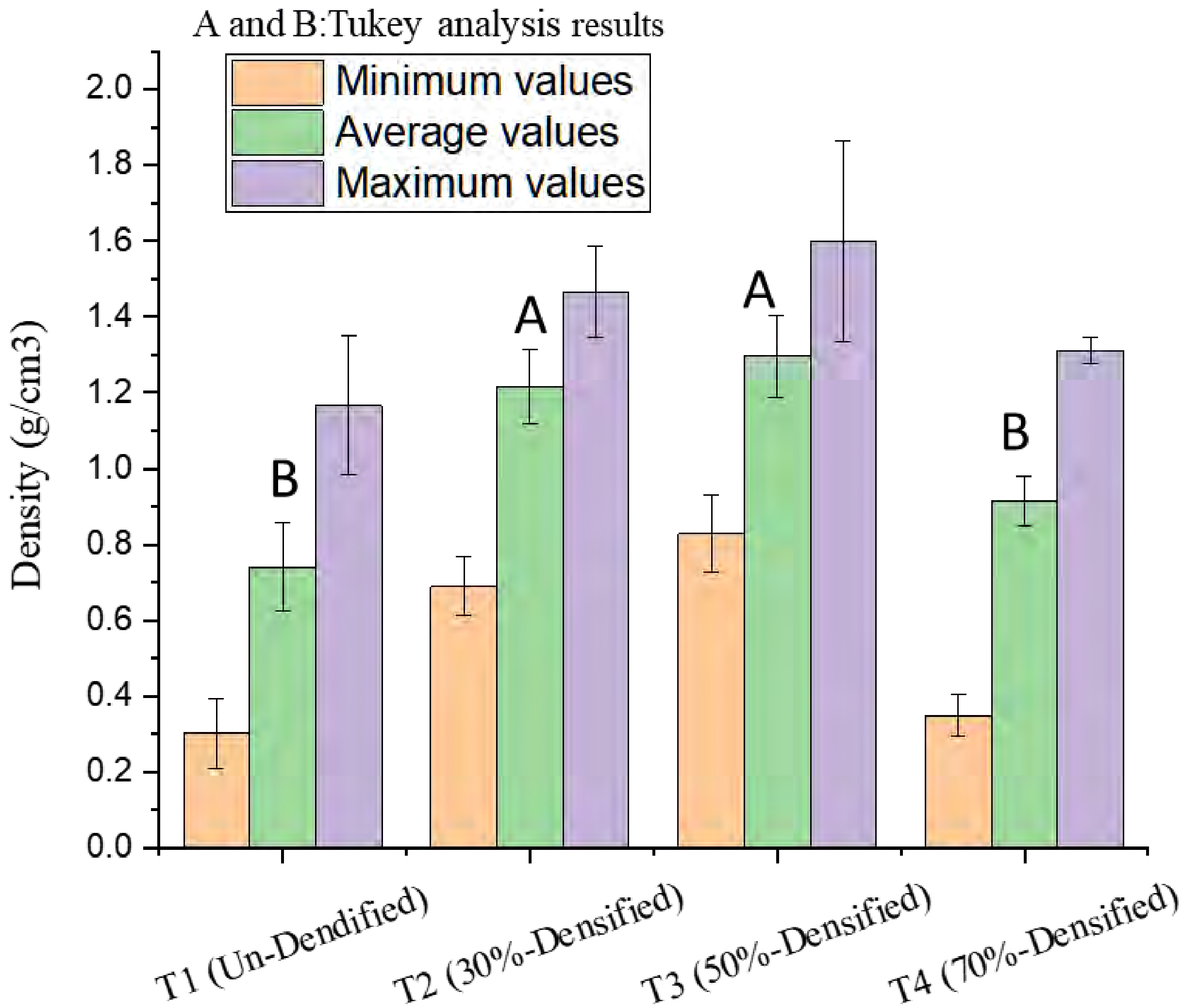
The microdensity profiles along the thickness of the samples make it possible to understand the effect of the treatments, and the corresponding thickness reduction, on the modifications of the anatomical microstructure. The T1 (control) shows a significant variation in microdensity from the outer to the inner layers due to the cellular tissue composition, whether fiber sheath (support, denser tissue), vascular (sap flow, lighter tissue), or parenchyma (storage, medium tissue). It should also be noted that in the culm inner to the outer layer, there is a gradual decrease of mean microdensity due to the dimensions and percentage of cellular tissue.

With the T2 treatment (30% DD) the variation in microdensity along the bamboo culm thickness is maintained, although presenting a subtle alteration in the bamboo tissue microstructure: reduction in the space of the parenchyma (5%) and vascular tissues (3%), resulting in greater percentage (8%) and proximity of the fiber bundles. The bamboo mean density increase was 65%.

The T3 treatment (50% DD) showed significant changes in the microdensity profile: lower density values in the outer and inner layers. This difference is more significant and accentuated in the inner layer due to the greater predominance of soft tissues (vascular and parenchyma) in relation to hard tissues (fiber sheath). It is also observed a reduction between the minimum and maximum densities and an increase in the mean density, which implies improved homogeneity and uniformity along the bamboo thickness. Quantitative analyzes for this treatment indicate an increase of 13 % of the hard tissues (fiber bundles), and a reduction of 13% of the soft tissues (vascular and parenchyma), with a 76% increase in density and 50% thickness reduction. The results showed that T3 seems to be most appropriate treatment to improve the bamboo culm properties, which can be confirmed by the correlation with other physical and mechanical properties.

The T4 was of more severe conditions inducing significant changes in the microdensity profile of bamboo culms compared to T1: an abrupt drop in density in the innermost layers (soft tissues) and an increase in the outermost layers (hard tissues). There was also an increase in the difference in minimum and maximum densities. The analyzes indicate a mean density increase of only 24%. Additionally, a collapse of the bamboo anatomical microstructure was observed, resulting in an undifferentiated and compact mass due to the pressing of the bamboo tissues and the formation of micro-cracks.

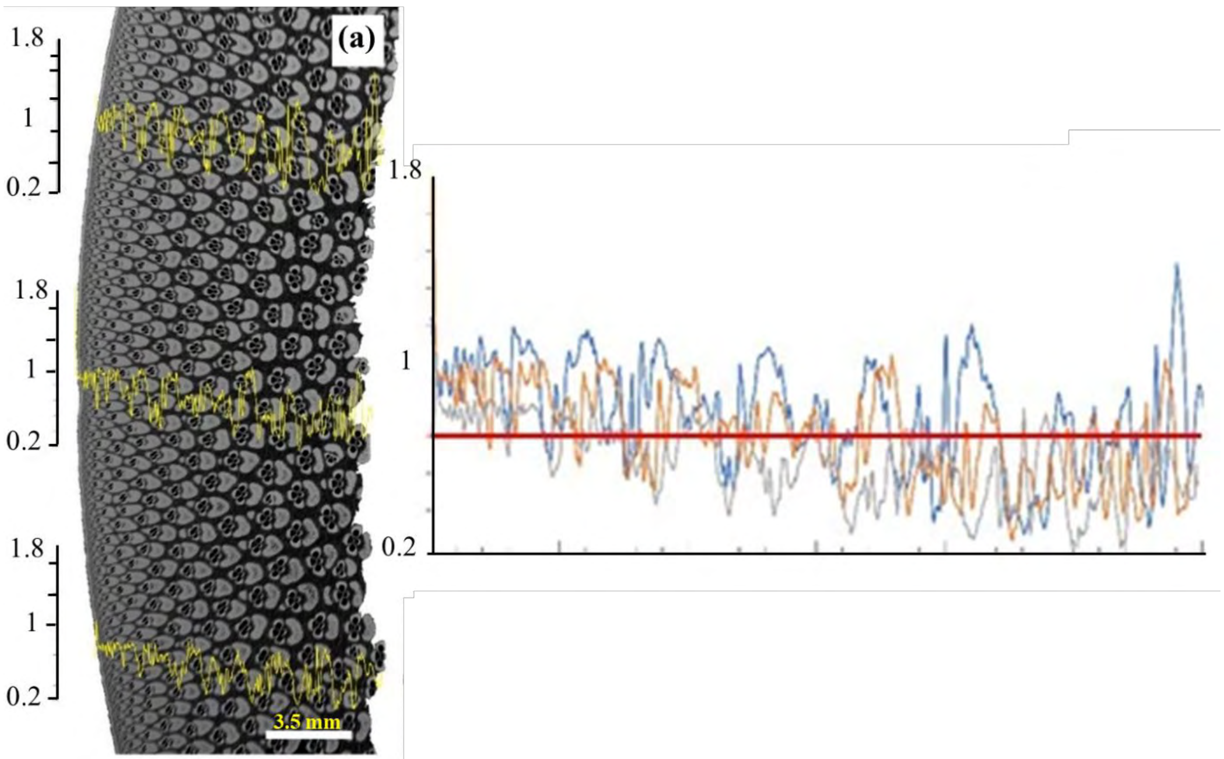
Figure 4.7 - The values of average, maximum and minimum density of bamboo (Same letters (A or B) mean there is no statistical difference among treatment conditions).

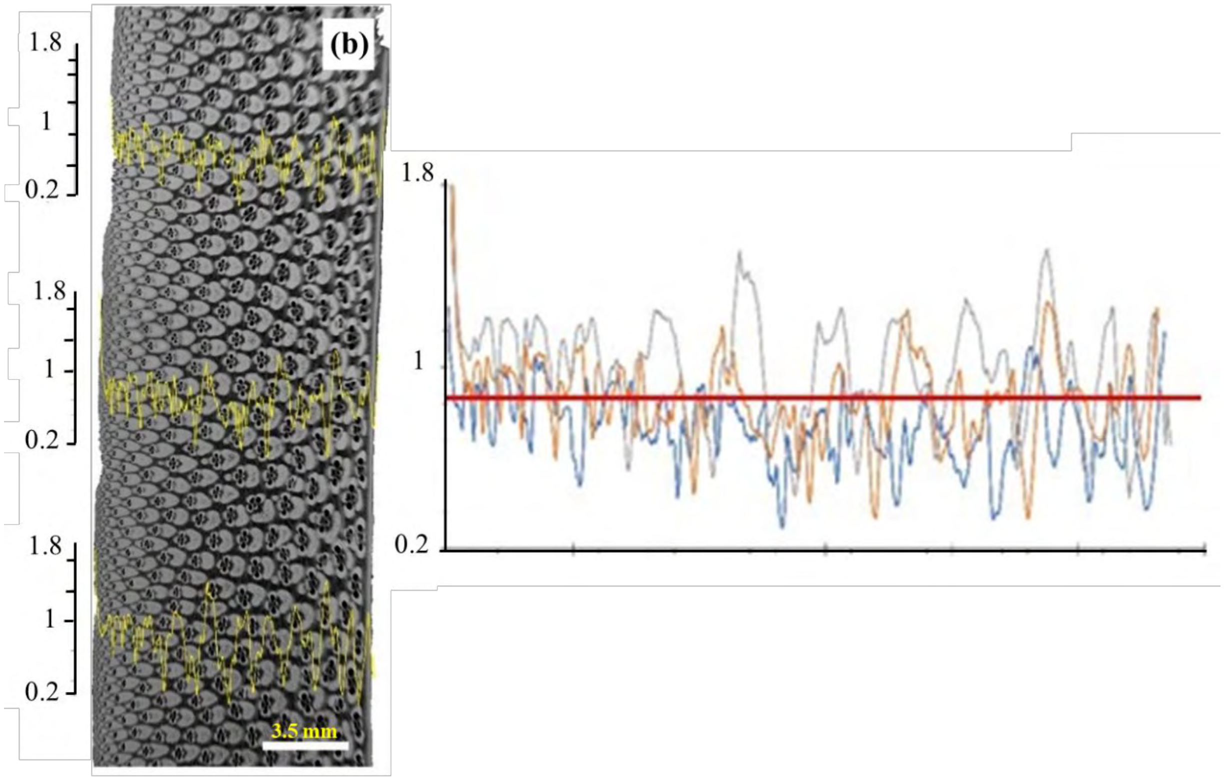


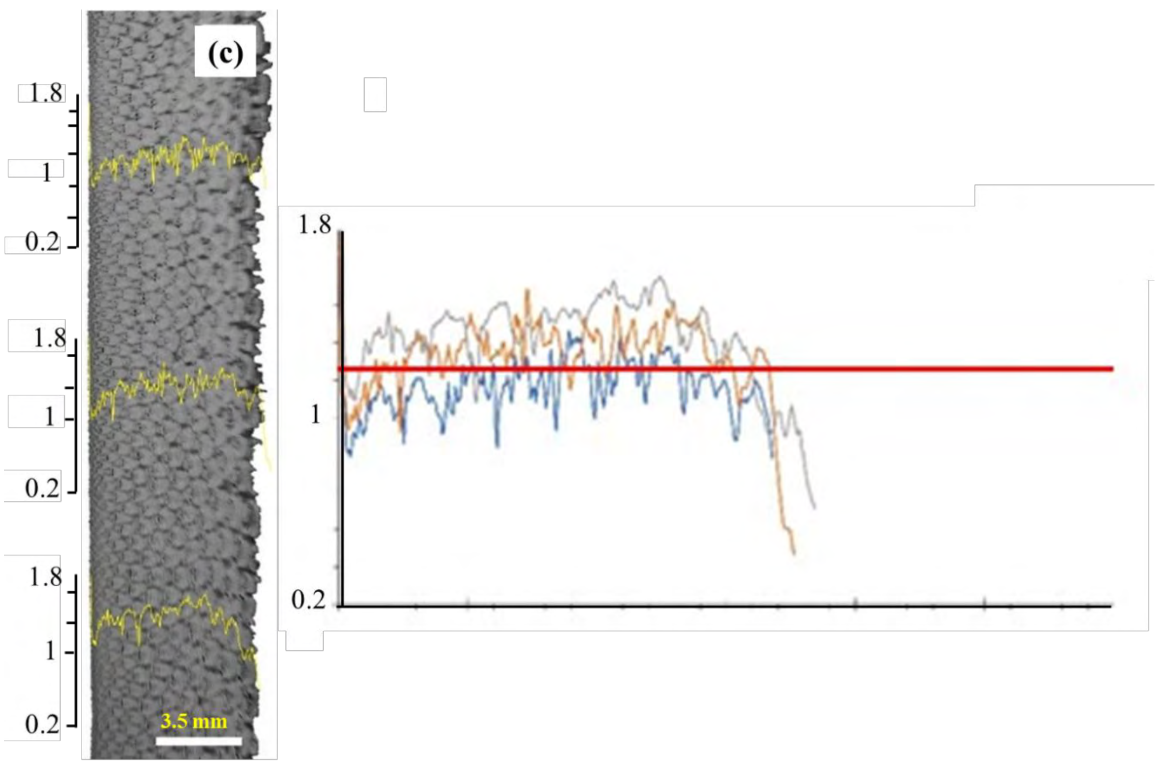
Source: Author's authorship.

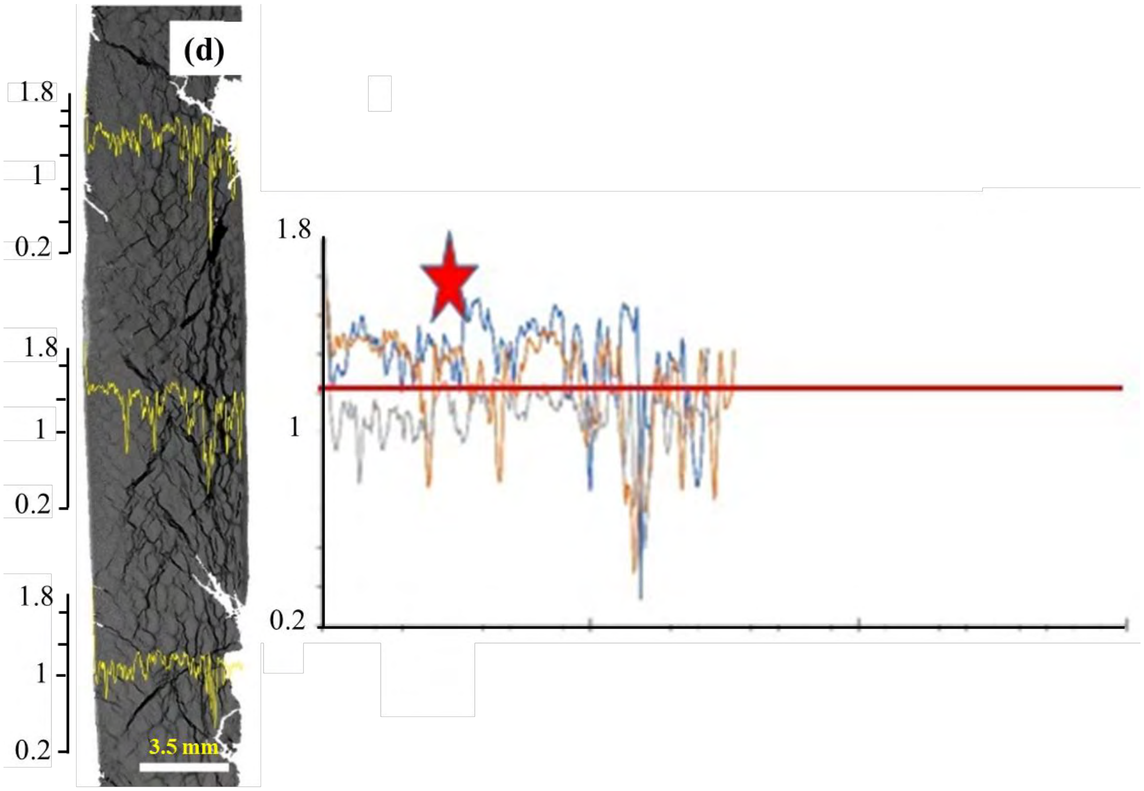
In figure 4.8, the microdensity profiles are represented. The yellow lines show the 3 positions of the bamboo samples that were analyzed. It is possible to observe the variations of the anatomical microstructure of the samples in the different treatments. The model of variation in the microdensity of bamboo samples demonstrates the effect of treatments. In this Figure, the outer and inner layers indicate the origin and the end of the microdensity profile. The red horizontal lines indicate the corresponding average densities of each condition. As a reference, the length of these lines represents the thickness (12.9 mm) of the T1 sample. The red star included in Figure 9d corresponds to the culm region with increased density and the corresponding one in Figure 9, of the anatomical microstructure of the T4 culms.

Figure 4.8 - Bamboo culm microstructure and microdensity profiles (yellow lines show the position): tratamento T1 controle (a); T2 30% DD (b); T3 50% (c); T4 70% DD (d).




(b)
1.8
1
0.2
1.8
1
0.2
1.8
1
0.2
3.5 mm
1.8
1
0.2

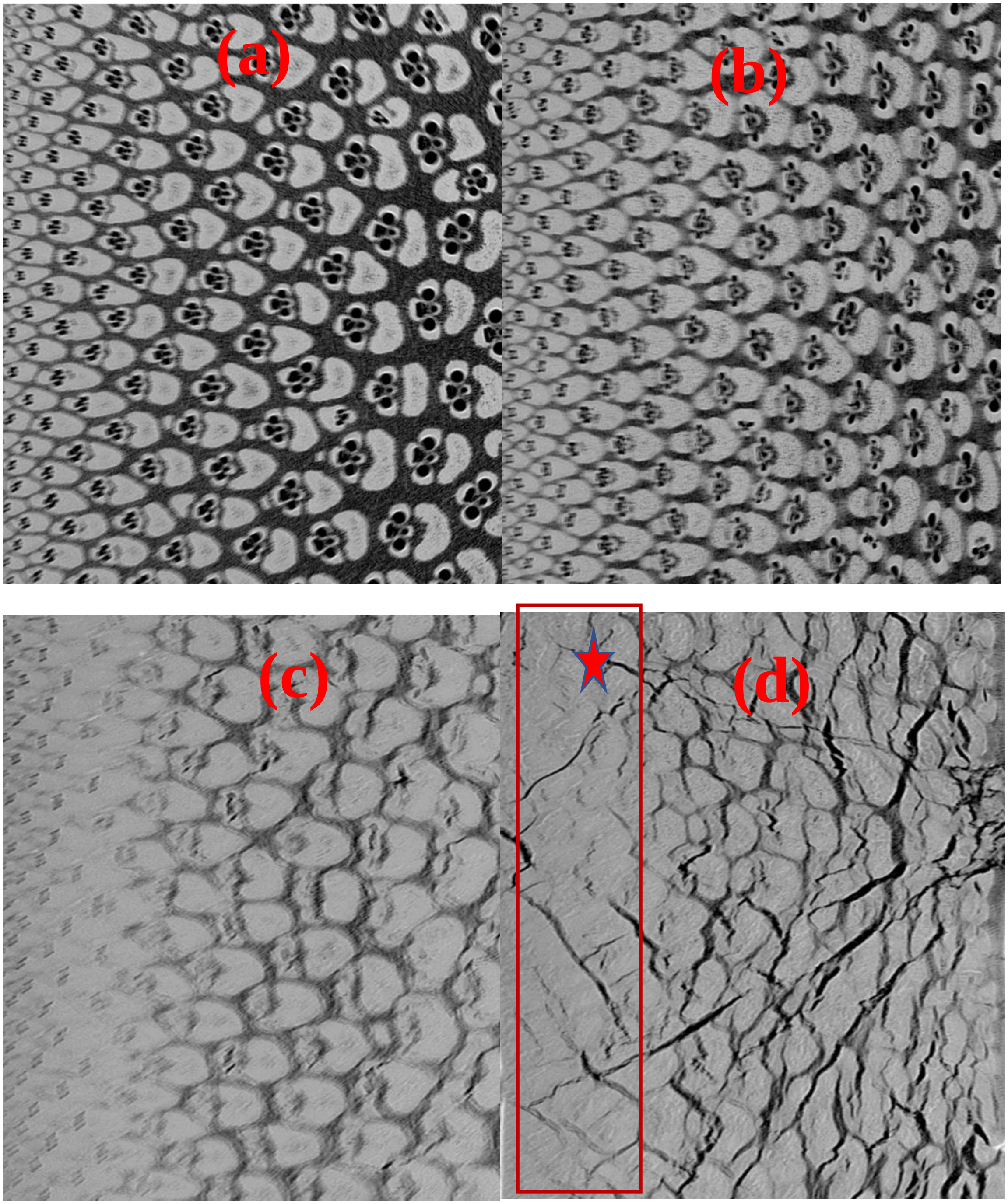

(c)
1.8
1
0.2
1.8
1
0.2
1.8
1
0.2
3.5 mm
1.8
1
0.2



Source: Author's authorship.

In Figure 4.9, bamboo culm microstructure for different treatments: T1 control treatment (a); T2 30% DD (b); T3 50% (c); T4 70% DD (d), indicating soft tissue (vascular and parenchyma) and hard tissue (fiber sheath). It is possible to observe changes in the anatomical microstructure of stalks in different treatments in relation to hard and soft tissues. The region marked in red corresponds to the star indication in the microdensity profiles of treatments T4 (Figure 4.8).

Figure 4.9 - Bamboo culm microstructure: tratamento T1 controle (a); T2 30% DD (b); T3 50% (c); T4 70% DD (d),



Source: Author’s authorship.

4.3.4. Dimensional stability analysis (step 2)

According to the results of step 1, DD between 30% to 50% results in better quality in terms of density and at the same time fiber cohesion. Therefore, DD=50% and the other parameters mentioned in Table 4.1, was used for the dimensional stability analysis in this step. The tests were repeated five times for each condition densified at 30, 160, and 200 ^{o}C at 2,4, and 8 mm/min of compression rate. The initial MC of the samples were (7±0.5) %, and after the densification process and 60 min of relaxing time, the samples were almost dry. Table 4.2 shows the value of weight loss for different conditions. The higher the temperature, the higher the weight loss; however, there is no regular pattern in terms of using different compression rates.

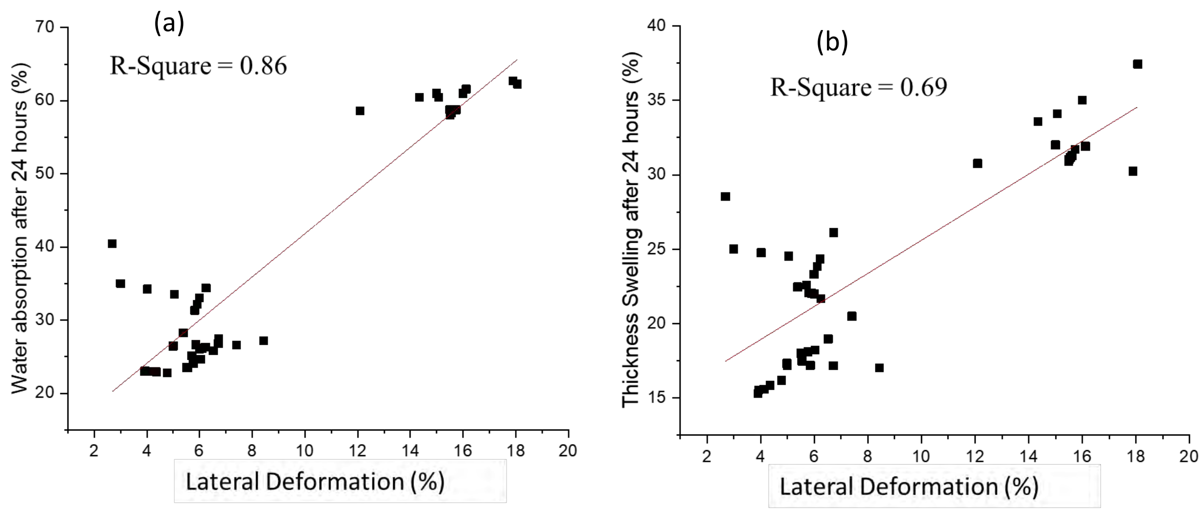
Table 4.2 - Weight losses of samples densified at different temperatures and compression rates.

Pressure rate (mm/min) / Temperature (^{o}C)	30	160	200
2	0	10.37 (0.80)	14.18 (0.79)
4	0	10.64 (1.07)	14.6 (6.08)
8	0	10.79 (2.17)	14.27 (6.5)

Source: Author's authorship.

Considering all the data, Figure 4.10 illustrates the water absorption and thickness swelling factors against the lateral deformation of the samples. The first graph shows that the slope of the regression line is significantly different from zero (R-Square = 0.86); therefore, it can be concluded that there is a trend between water absorption and lateral deformation. Although the same discussion can be given for swelling and lateral deformation (R-Square = 0.69), it is obvious that the slope of the WA graph is much steeper than the TS graph. Therefore, it can be deduced that LD is a suitable criterion to be checked during the densification process, considering that the higher the LD, the higher the WA and TS.

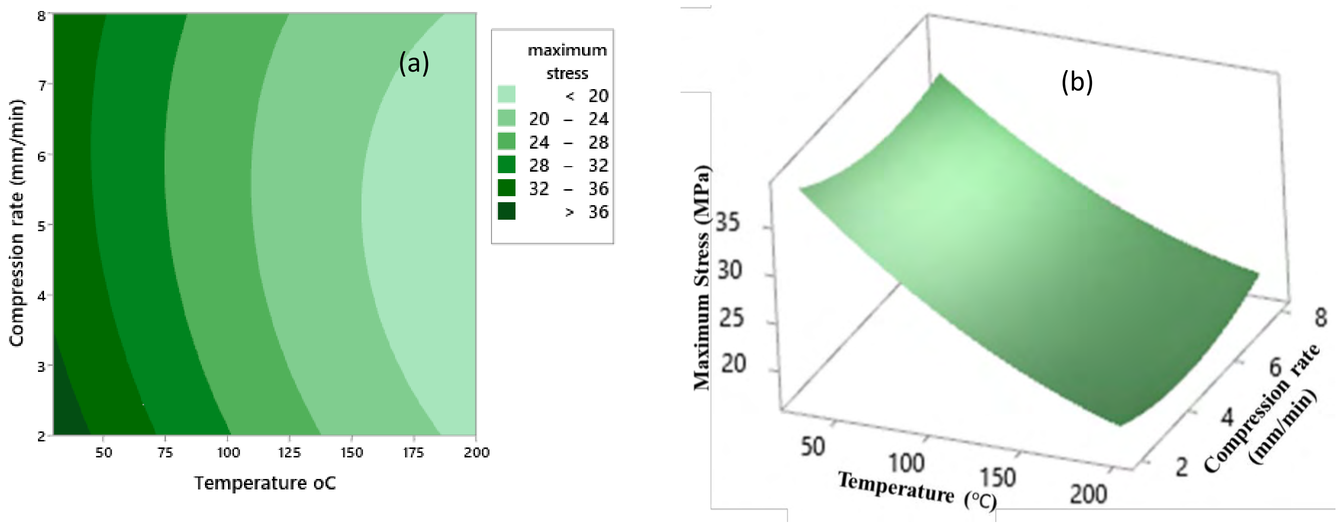
Figure 4.10 - The correlation of a) water absorption and b) thickness swelling with lateral deformation



Source: Author's authorship.

Figure 4.11 presents the contour and surface plot of the maximum stress, which is required to densify bamboo to 50% of its original thickness, with variations of the temperature and compression rate values. According to this figure, the maximum stress rate occurs at the lowest temperatures. In other words, to densify bamboo to 50% DD, more force is needed at a low temperature. Higher temperatures, on the other hand, facilitate relaxation of the internal stresses (TENORIO; MOYA; FILHO, 2020). Besides, the influence of the compression rate on the maximum stress rate is almost negligible for various temperatures.

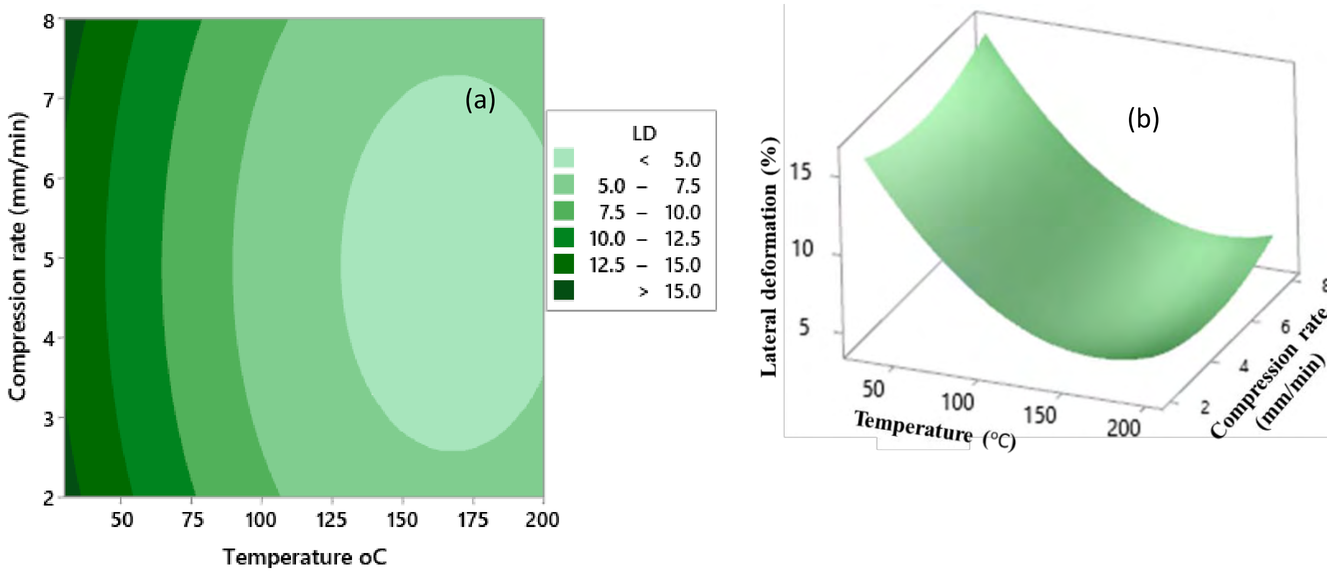
Figure 4.11 - Counter plot (a), and surface plot (b) of maximum stress, which is required to densify bamboo to DD of 50%, versus pressure rate and temperature.



Source: Author’s authorship.

Variations of lateral deformation due to bamboo compaction to 50% DD by varying temperature and compression rate are presented in Figure 4.12. The result of the analysis indicates that LD is reduced by increasing the temperature with a minimum between 160-200 ^{o}C. Compression rate showed a small effect on LD. The reduction in LD with temperature is related to the increase in bamboo plasticity. The variation of LD at the temperature range of 160-200 ^{o}C by changing the compression rate is attributed to the water loss during the process and deviation of the test results.

Figure 4.12 - Counter plot (a), and surface plot (b) of lateral deformation (LD) induced by densifying bamboo to DD of 50%, at different treatment conditions.



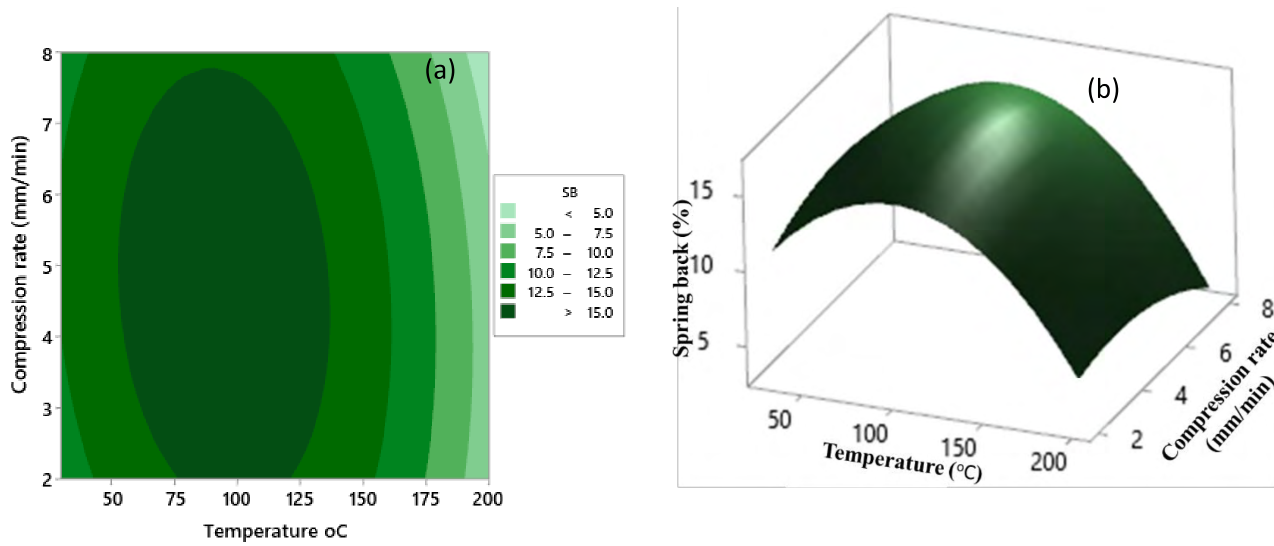
Source: Author's authorship.

Figure 4.13 demonstrates the spring back (SB) result analysis for the discussed parameters. According to the figure, the spring back factor rises with the temperature increment up to around 100 ºC. At this point, the graph reaches its maximum value and tends to decrease again for higher temperatures. It can be because of increasing elasticity as a result of the increase in temperature. However, at a temperature higher than 100 ºC thermal degradation of the cell wall components occurs, which can cause a more stable state after densification, similar to wood. According to wood literature, at a temperature higher than 150 ºC, hemicellulose degrades, and the higher the temperature and the time of treatment, the higher the amount of hemicellulose degradation and consequently, the lower the spring back. Similar to wood densification, at lower temperatures, there is very little stress relaxation, and thus, the deformation is expected to be mainly elastic.

It is also noticeable that the maximum SB happens at moderate pressure rates. Lower pressure rate means more time of pressing process and therefore, more degradation of bamboo.

Lower spring back at high-pressure rates can also be a result of higher lateral deformation and more cracks.

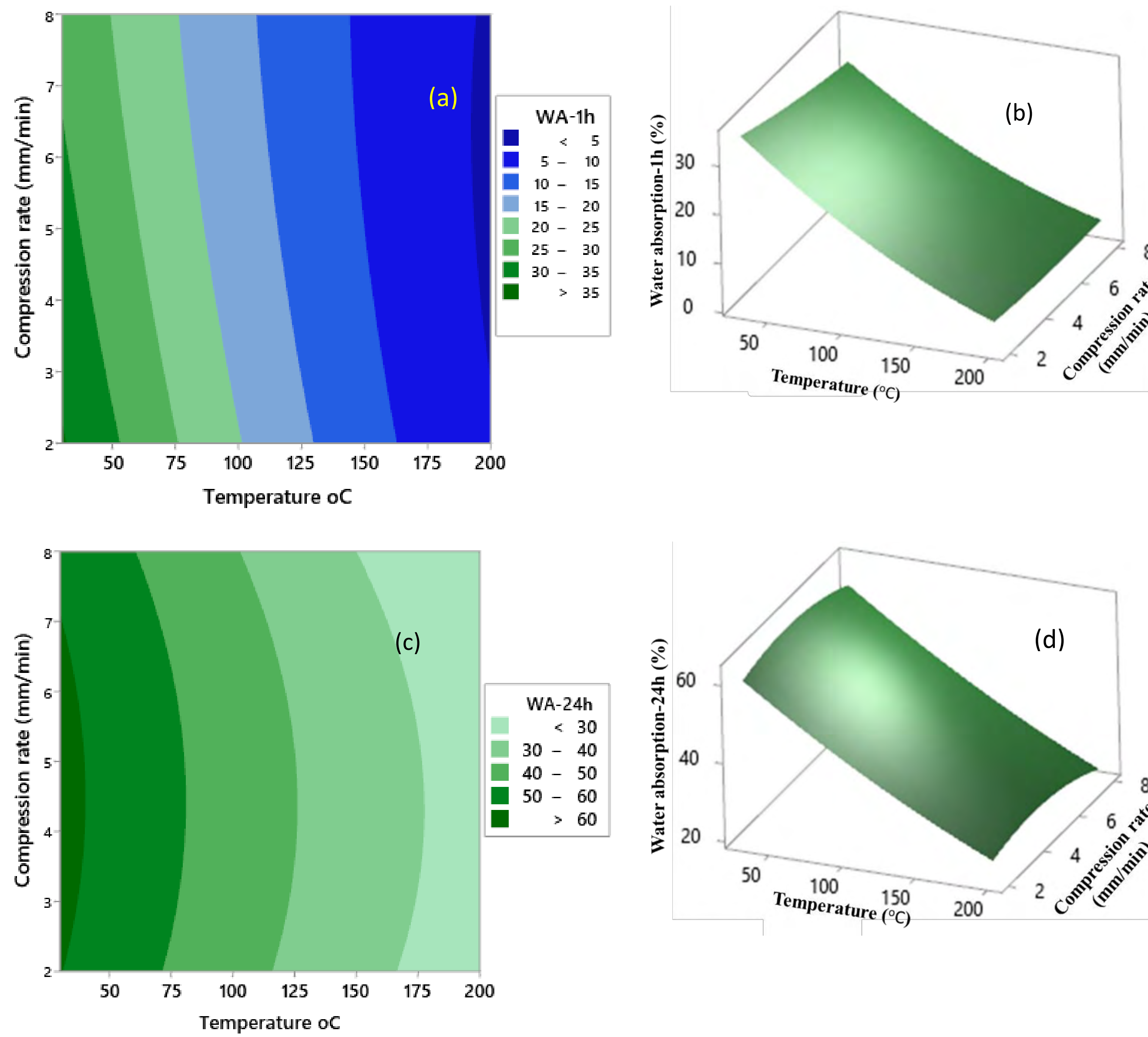
Figure 4.13 - Counter plot (a), and surface plot (b) of spring back (SB) induced by densifying bamboo to DD of 50%, at different treatment conditions.



Source: Author's authorship.

The contour and surface plots of water absorption results after 1h and 24h are illustrated in figure 4.14a-d. WA had a decreasing trend with the rise in temperature and compression rate after 1 hour. However, after 24 hours, the optimum compression rate factor for WA is around 5 mm/min. Two possibilities can explain this different behavior. First, the immersion of the samples in water during 1h is not enough to achieve equilibrium of the bamboo samples in the presence of water. Therefore, higher deviations are expected. Second, the influence of smaller cracks, if present, is expected to be more pronounced after longer times of immersion since the water absorption is related to open defects. Also, it is worth noticing that the rate of water absorption has increased drastically from range of 4.5-36.3 to the range of 22.9-60.44 after 1h to 24h, which was well predicted.

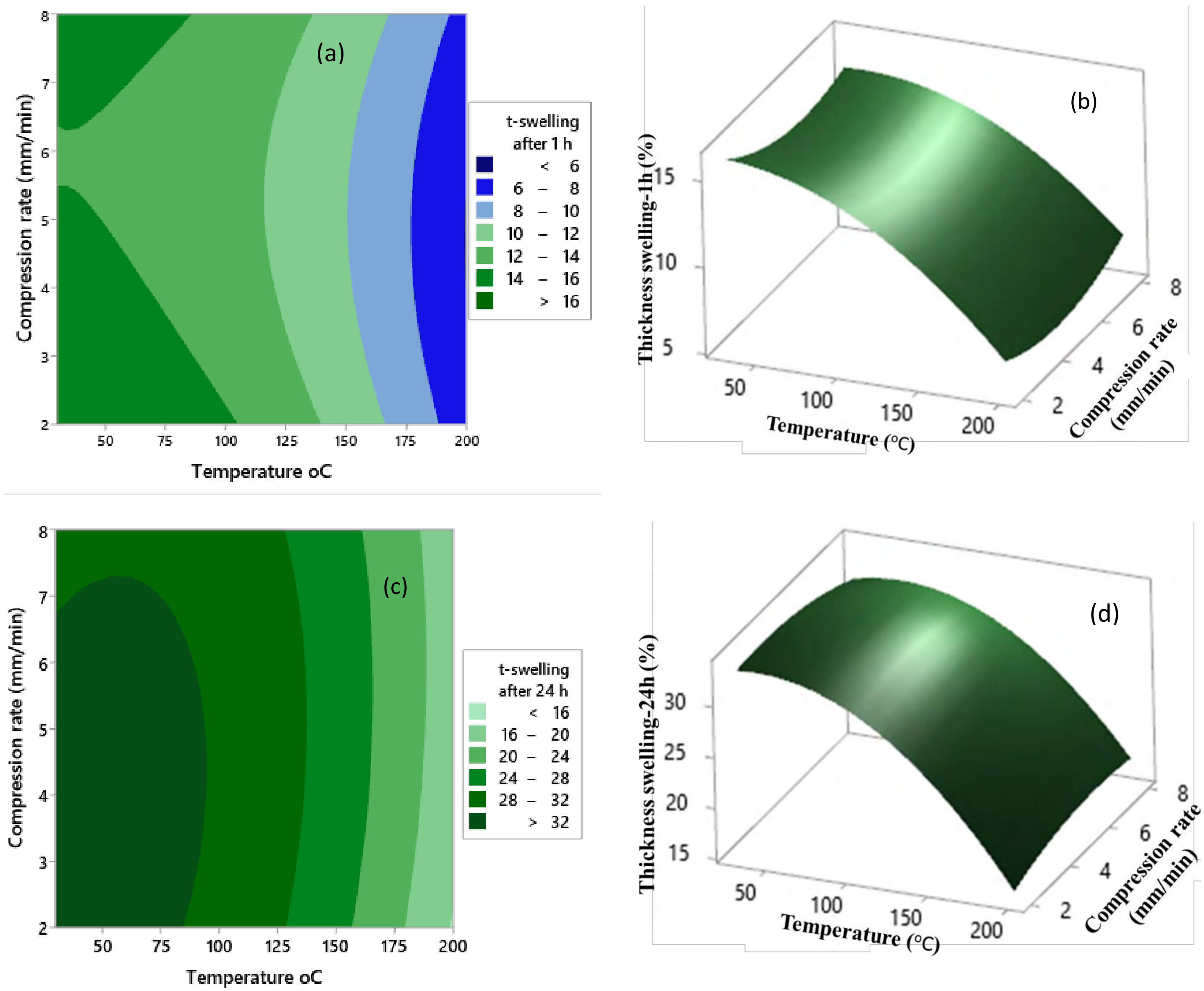
Figure 4.14 - Optimization analyzes of water absorption (WA) of densified bamboo with DD of 50%, at different treatment conditions (Counter plot (a), and surface plot (b) of the results after 1h, and Counter plot (c), and surface plot (d) of the results after 24h).



Source: Author's authorship.

Figure 4.15 presents the thickness swelling of the samples after 1h and 24h. As can be seen, the influence of the compression rate is more pronounced on this parameter. The highest TS occurs at the lowest temperature and compression rates. Regarding the temperature influence on the dimensional stability of densified bamboo, the results are similar to that of wood because of the same composition of these two materials. According to the wood literature, (Dwianto et al 1999; Navi and Heger 2004) higher temperatures may break existing covalent and hydrogen bonds and form new cross-links and hydrogen bonds between cellulose and hemicellulose. These modifications in chemical bonds of bamboo can also happen during densification, helping the stabilization after deformation.

Figure 4.15 - Optimization analyzes of thickness swelling (TS) induced by densifying bamboo to DD of 50%, at different treatment conditions (Counter plot (a), and surface plot (b) of the results after 1h, and Counter plot (c), and surface plot (d) of the results after 24h).



Source: Author's authorship.

4.3.5. Optimization of the Dimensional stability results

MROM analyzed the relationships between the investigated responses (WA, TS, SB, and maximum stress) and variables (temperature and compression rate) to provide the optimal solution. The target here is to minimize all the mentioned responses. The results show that the optimum temperature and pressure rate to achieve the minimum responses of dimensional stability are 200 ^{o}C and 6.73 mm/min, respectively. The predicted values of responses as the optimum results are shown in Table 4.3.

Table 4.3 - Multiple Response Prediction of bamboo densified at 200^{o}C and 6.73 mm/min.

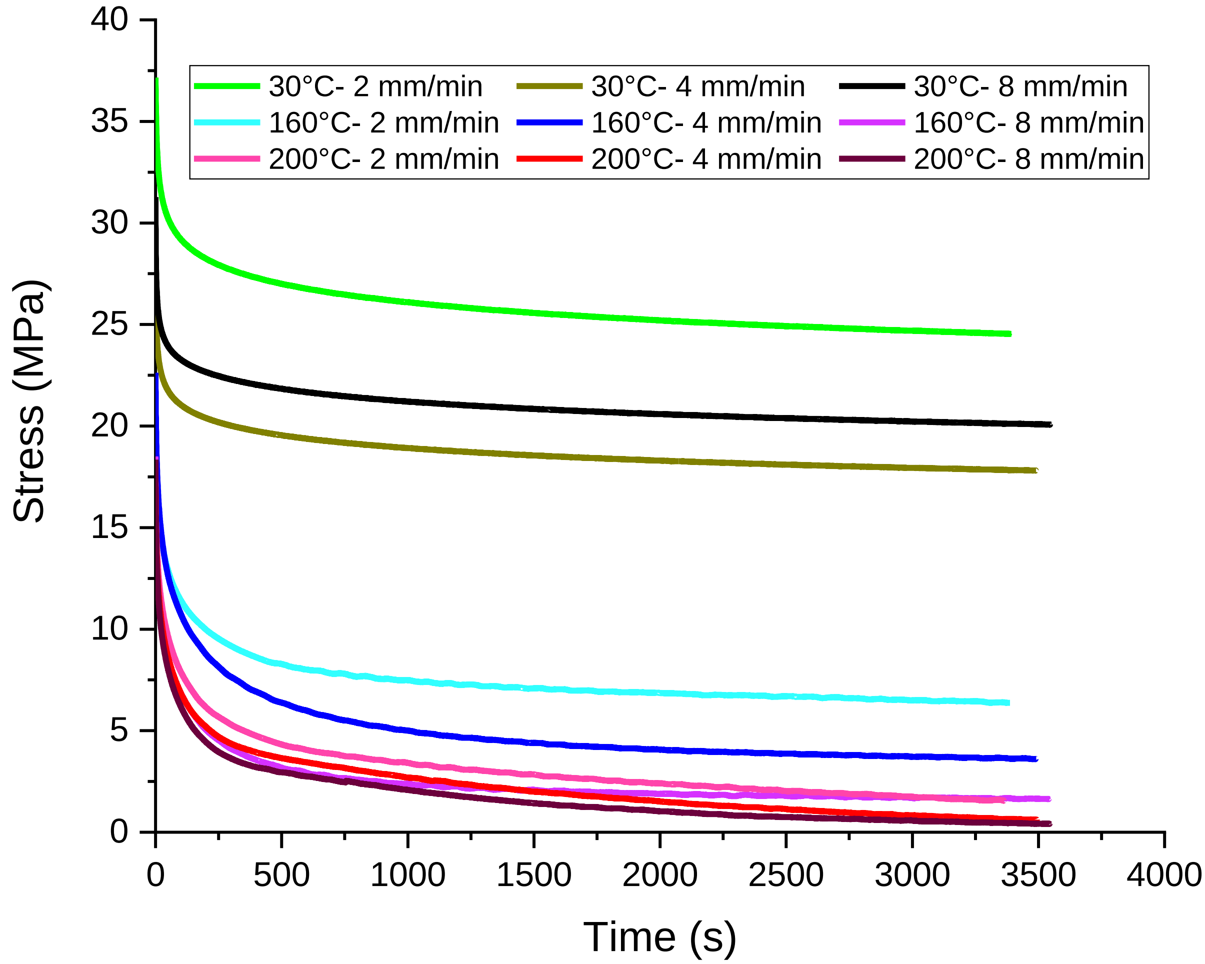
Densification Process Response	**Optimum value**
Lateral Deformation (%)	5.2
Maximum Stress (Mpa)	18
Thickness Swelling after 1 h (%)	6.5
Thickness Swelling after 24 h (%)	17.7
Water Absorption after 1h (%)	4.4
Water Absorption after 24h (%)	23.8
Spring Back (%)	4.7

Source: Author's authorship.

4.3.6. Influence of parameters on bamboo relaxation time

To measure relaxation time, bamboo specimens were compacted in the radial direction to about 50 % of their original thickness (DD=50%) over a temperature range of 30 to 200 °C using rate of 2, 4, and 8 mm/min for 60 minutes. Then, by keeping the displacement constant, the force begins to decrease with time until a constant value. Figure 4.17 shows the force relaxation curves for different treatments. Most of the force decay befell within the first 100 seconds at room temperature and within the first 250 seconds at elevated temperatures. According to this plot, a relaxation time higher than 600 seconds at both room and elevated temperatures can be recommended because, after this time, the force becomes more constant. At an identical point of time, by raising the temperature, force decreases, which means temperature promotes relaxation of the material. At room temperature and 160°C, the force tends to a positive constant value, while at 200 °C the force tends to zero. These results corroborate with the reduced spring back effect, and hence higher stability, of the samples densified at 200 ºC.

Figure 4.17 - Force relaxation curves of bamboo samples



Source: Author's authorship.

4.4. Conclusions

The present study deals with obtaining an optimum thermo-mechanical treatment to achieve at first the highest critical densification degree, and second, best dimensional stability for bamboo strips. The dimensional stability analysis for the test cases subjected to the optimum densification degree was carried out to investigate spring back, water absorption, and thickness swelling factors. In order to present accurate and reliable results, 27 different tests for step 1, and 45 different tests for step 2 were opted and analyzed experimentally. The following outcomes were found:

- By manipulating the densification process parameters, the desired specifications in terms of physical properties can be achieved.
- The highest temperature and the lowest compression rate are the optimum parameters to achieve the maximum DD; the densification degree was found to be more dependent upon temperature than upon pressure rate.
- Higher temperature resulted in better flattening, greater weight loss, and better dimensional stability of TM densified bamboo.
- It was found that the optimal densification parameters are: DD between 30-50%, a temperature of 200 ^{o}C, compression rate of around 6mm/min, and a relaxation time higher than 600 s.
- Applying TM densification using the mentioned parameters, the highest obtainable DD was 43.6% with a density of 1.3 g/cm^3.

The minimum pressure required to densify bamboo in the optimum situation is 18 MPa, and the optimum response of SB, WA after 1 hour and 24 hours soaking, thickness swelling after 1 hour and 24 hours soaking was predicted to be 4.72%, 4.35%, 23.80%, 6.46%, and 17.70% respectively.

5. CHAPTER 5 – Physical properties of thermo-hydro-mechanically (THM) flattened and densified bamboo (moso and *D. asper*)

This chapter was presented in the meeting of Brazilian Materials Research Society (XVIII B-MRS Meeting, Balneário Camboriú, SC, Brazil, September 22 – 26, 2019), and received Bernhard Gross Award and the "ACS Publications Best Oral Presentation Prize".

Abstract:

The combination of flattening and densification process of bamboo has still not been understood and explored. In this study, the flattening phase is followed by densification using thermo-hydromechanically (THM) process to increase the density and improve the physical properties. Different pre-treatments with water were performed before the process. All groups of samples were subjected to the THM modification (flattening and densification) and a comparison in terms of microstructure, densification degree, number of cracks, spring back effect, water absorption, and swelling was performed. Results show that applied process improves some physical properties of bamboo materials; e.g. increases the density and makes the structure uniform. However, there are exceptions for swelling and water absorption. The pre-treatments did not have statistical influence on the densification degree.

Keywords: Thermo-hydro-mechanically (THM) process, Bamboo, Flattening, Densification.

5.1. Introduction

Engineered bamboo products, such as plybamboo, scrimber and glued laminated bamboo play an important role in the future bamboo use in the constructed environment (PLANT, ; SHARMA et al., 2015a). Bamboo flattening is a promising process in which very high material usage efficiency is achieved, on the contrary of other well-known bamboo products (FANG et al., 2018b). High temperature softening is the main feature of flattened bamboo manufacturing process and although cracks can appear, gluing several elements together makes a high quality and efficient final product. However, the combination of flattening and densification process has still not been fully understood and explored. In this study, the flattening phase is followed by densification using thermo-hydro-mechanically (THM) process to increase the density and improve the physical properties. According to the literature, THM treatment of bamboo, in small samples, can produce material with higher mechanical properties than natural bamboo (ARCHILA-SANTOS; ANSELL; WALKER, 2014; DIXON et al., 2016a).

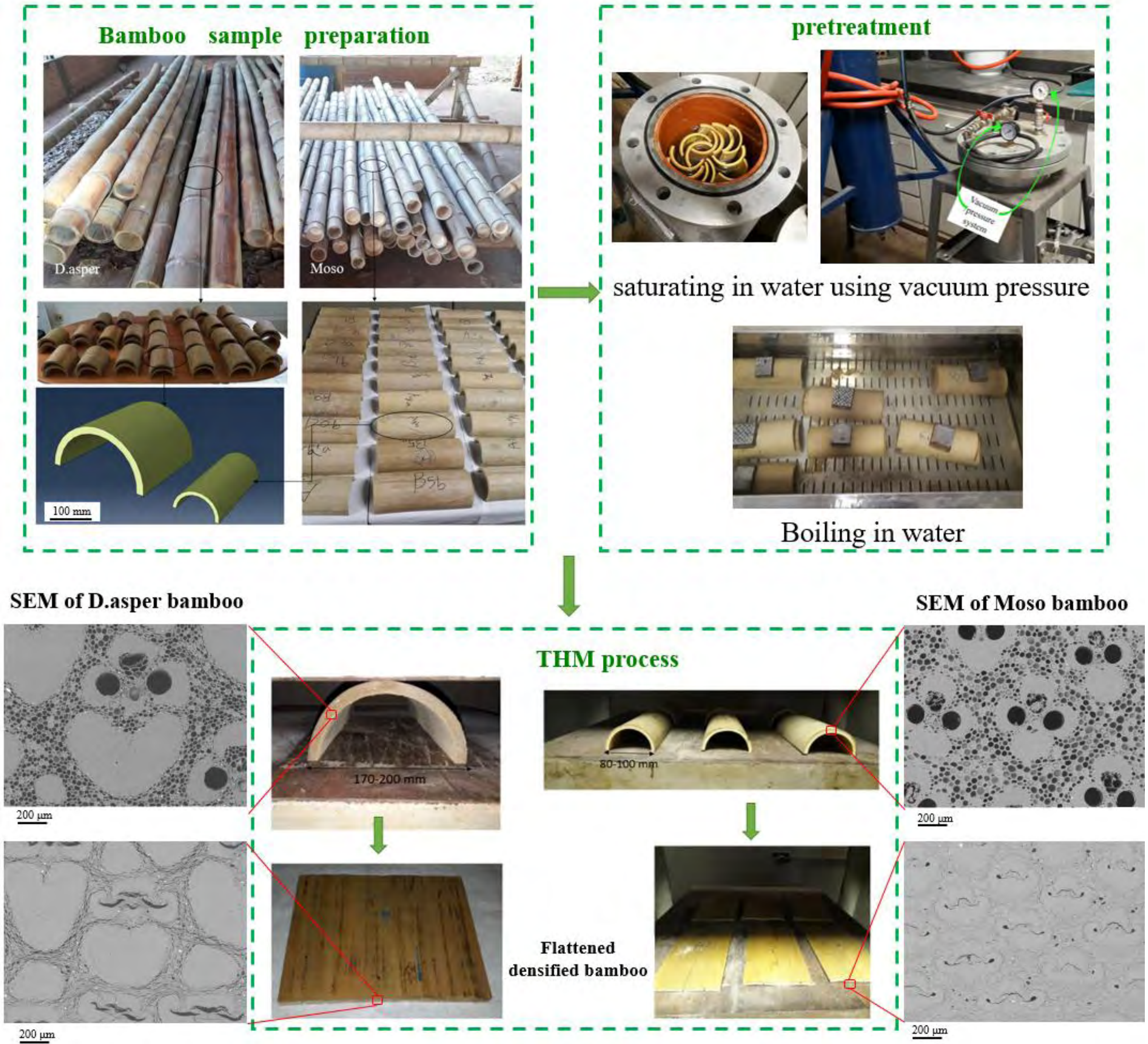
In order to develop bamboo materials that can be used as reinforcement and at the same time have high material use efficiency, we studied the effects of flattening and densification of two bamboo species Dendrocalamus asper and Phyllostachys pubescens (Moso), aiming in this first study, the investigation of the involved process and physical properties. Since increasing the moisture content in wood leads to the reduction of the glass transition temperature (Tg) and consequently facilitates the flow (plasticization) of lignin (ARCHILA-SANTOS; ANSELL; WALKER, 2014), in this project different pre-treatments with water were performed before flattening the samples. All groups of samples were subjected to the THM modification (flattening and densification) and a comparison in terms of microstructure, densification degree, number of cracks, spring back effect, water absorption and swelling was performed.

5.2. Experimental Procedure

A total number of 72 internodes of Moso bamboo with the dimension about diameter (D): 80-100 mm, length (L): 200 mm, wall thickness (t): 5-7mm, and D. asper bamboo; D: 170-200 mm, L:200 mm, t:10-14 mm from mature culms were used. After crosscutting and splitting (Fig.1), bamboo samples were first flattened, conditioned and then densified. Three pretreatment condition was evaluated, one group of samples was saturated in water using vacuum/pressure (D.S), a second group was boiled in water for 1 hour at 100 °C (D.B), and a third group was used as reference with an equilibrium moisture content of approximately 15% (D.Eq). For the production of THM compressed specimens, as demonstrated in Fig.1, an open thermo- hydraulic press system was utilized. The parameters for the first phase, flattening, and the second phase, densification, are shown in table 5.1. After flattening the samples were conditioned up to a moisture of 7% and then densified in the same thermo-hydraulic press.

The densification degree (DD) was estimated using Eq. 3.1. Oven dried density, spring back, thickness swelling (TS), and water absorption (WA) of the two bamboo species were also investigated. Samples with dimensions of 20 x 20 mm x wall thickness were obtained, and after drying in the oven at 103 ± 2 °C, the density was calculated using the water immersion method. Furthermore, the spring-back (SB) of the densified samples was determined after conditioning at 20±2°C and at the relative humidity (RH) of 65±3% for four weeks and calculated using Eq.3.4. The thickness of the samples after densification and also the thickness after conditioning of samples at 20± 2 °C and 65±3% RH were considered. The water absorption and thickness swelling tests after 24 h were carried out based on ASTM D1037 − 12. Moreover, the microstructures before and after densification were analyzed by scanning electron microscopy (SEM).

Figure 5.1- Flattening and densification process.



Source: (KADIVAR et al., 2019b)

Table 5.1- Flattening and densification process parameters.

phase	***Flattening***	***Densification***
Pressure (MPa)	2	10
Temperature (°C)	140	140
Time (min)	5	20

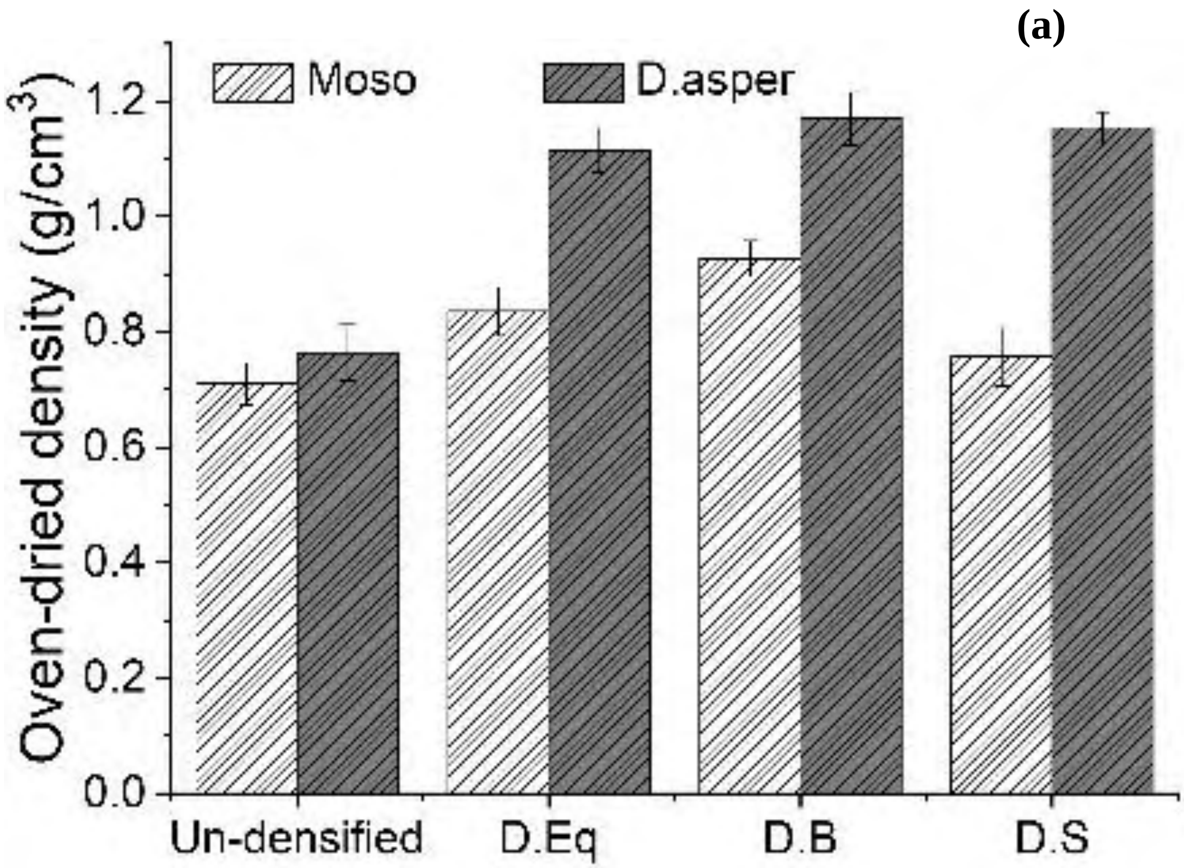
Source: (KADIVAR et al., 2019b)

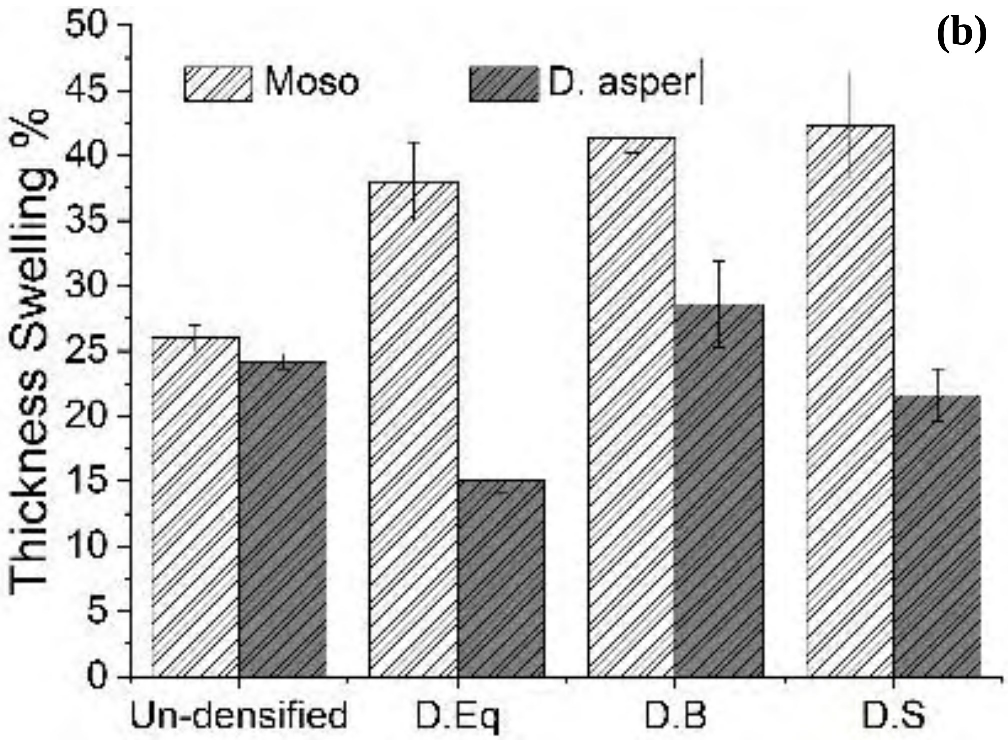
5.3. Results and Discussion

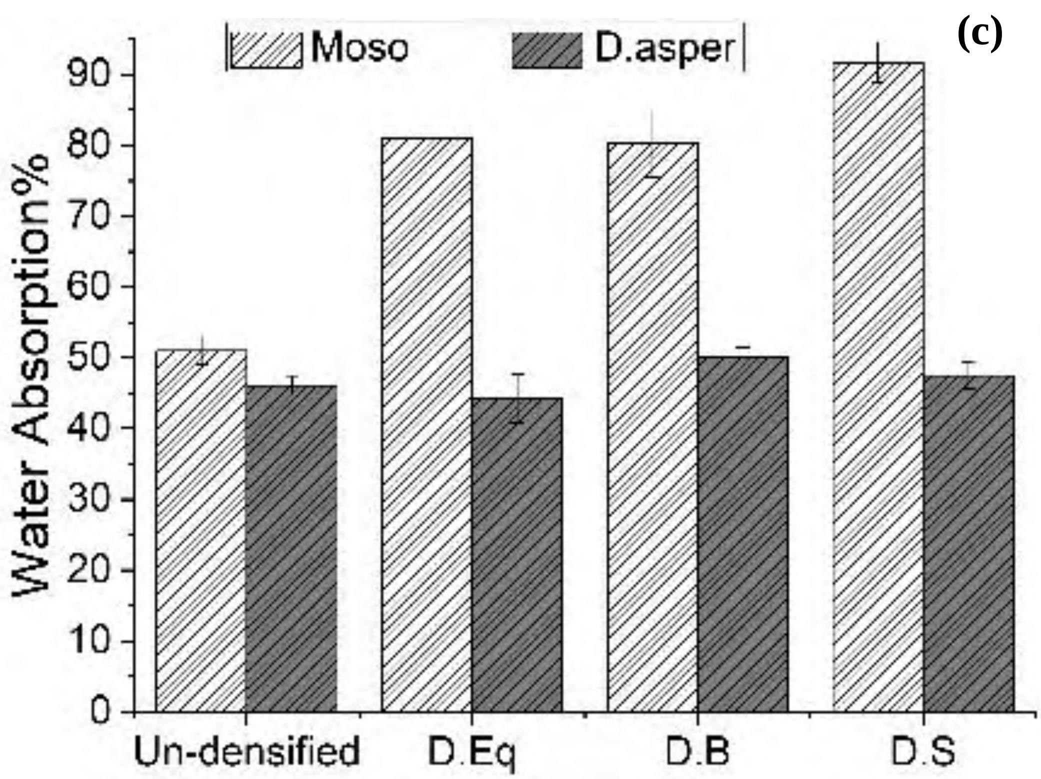
Using the same flattening process (see Table 5.1) it was possible to observe that the D.B condition presented the lowest average amount of cracks per specimen in both species, 5 and 7 cracks for Moso and D. asper respectively. However, the pre-treatments did not have statistical influence on the densification degree. For Moso bamboo it was achieved an average degree of densification of 24.6%, whilst for D. asper 32.5% was observed. In Figure 5.1, microscopic images reveal the collapse of vessels and parenchyma without the deformation of the fiber bundles after the densification process. This effect caused an increase of the density, more pronounced in D. asper bamboo, as shown in Figure 5.2. Spring-back results also showed no difference between the pre-treatment conditions, with averages around 1% for both species, even after four weeks after densification.

It is also found that similar to some densified wood, the densification process had an increasing effect on the swelling and water absorption of bamboo. For Moso bamboo, the WA (84%) and TS (41%) of the densified samples, in comparison with un-densified ones, was approximately 60% higher. On the other hand, D. asper samples had the same behavior of the un-densified samples, with 47% and 23% of WA and TS, respectively. The main reason for this rather weak effect of densification could be the existence of cracks, occurred after flattening. For this reason, this study recommends investigating the flattening before the densification process. The verdict about the pretreatment of densified bamboo depends on the outcome of further experiments with controlling the load rate.

Figure 5-2- Physical properties results (a) oven-dried density; (b) swelling; (c) water absorption.







Source:(KADIVAR et al., 2019b)

5.4. Conclusions

Flattening and densification treatment improves some physical properties of bamboo materials; e.g. increases the density and makes the structure more uniform. However, there are exceptions for swelling and water absorption. Boiling of the bamboo as a pretreatment method, before densification, reduced the number of cracks in comparison with the other pre-treatments. Densification of bamboo is quite different from that of the wood, because of the cylindrical shape of bamboo and it is required to study densification in association with the flattening process for bamboo materials. Although more studies have to be conducted, the overall results obtained for D.asper showed better performance than of those from Moso bamboo.

6. CHAPTER 6 – Evaluation of an optimized bamboo sandwich panel using layers of two different bamboo functional elements

This chapter was presented in the meeting of Brazilian Materials Research Society (XVIII B-MRS Meeting, Balneário Camboriú, SC, Brazil, September 22 – 26, 2019),

Abstract:

To obtain high efficiency and in the same time, high mechanical properties, it is combined two bamboo functional elements to make bamboo sandwich panels (BSP). The skin is made of flattened-densified bamboo, which is the strongest bamboo element, but has the lowest utilization ratio, and the core of particleboard, which is made of bamboo residues with high utilization ratio. According to the achieved results, using densified bamboo on face sheets of BSPs, the MOR and MOE results in longitudinal direction increased by 917% and 833.3%, respectively, in comparison with the bamboo particle board core with the absolute values of 32.11 MPa (COV: 0.14) for MOR and 1.36 GPa (COV: 0.40) for MOE. Low thermal conductivity results, with values of 0.203 W/m-K indicate that BSP can be considered as an insulation material.

Keywords: flattened-densified bamboo, particleboard, thermal conductivity, three-point flexure

6.1. Introduction

Among the investigated bamboo panels that had been reported by several researchers (CHAOWANA; BARBU, 2017; HUANG; SUN; MUSSO, 2017a), bamboo particleboards (BPB) have the lowest range of module of rupture and module of elasticity, while plybamboo and glued laminated bamboo have the highest. Flattened bamboo is the biggest bamboo industrial element produced during the last decades using many novel technologies on bamboo culm flattening (FANG et al., 2018b). However, it is not possible to obtain big size elements from all parts of a bamboo culm because of the dimensional and macroscopic properties variation along its length. Since the middle part of a bamboo culm, which is more homogeneous than the upper and bottom part, is the most appropriate part for flattened bamboo production, the work efficiency and utilization ratio of this method are low. To improve utilization ratio and, at the same time, overall product performance, the authors came up with a solution to combine the elements with different size functions and make bamboo sandwich panels (BSP). Flattened-densified bamboo, which is the strongest element, but has the lowest utilization ratio, was used for face sheet and bamboo particleboard, which is made of bamboo residues, was applied as the core. This solution becomes a compromise among these conflicting responses; mechanical properties and resource utilization.

6.2. Experimental Procedure

Mature bamboo culms, *Dendrocalamus asper* species were obtained from an experimental field in Brazil (21°58'53.5"S 47°26'03.3"W). The bamboo culms were divided into three parts (figure 6.1). The middle part was converted into a flattened and densified bamboo (DB) using a thermo-hydro mechanical process (THM). The upper and bottom parts were crushed into particles (between 1 to 6 mm), incorporated with castor oil polyurethane adhesive, and used to produce the core layer by thermo pressing process. A polyvinyl acetate based commercial wood glue was used for bonding the flattened bamboo face layers (thickness of 5 mm) with the particle board core layer (thickness of 15 mm), obtaining symmetric bamboo sandwich panels with the same grain direction in both sides and with dimensions of (550 × 550 × 25) mm3. The physical, mechanical, and thermal properties of the densified bamboo, BPB, and BSP specimens were measured separately according to the relevant standards. The physical characterization was based on the evaluation of the density and moisture content (MC). The material behavior subjected to three-point flexure test was carried out according to the C393/C393M, 2016 Standards using (200 × 50 × thickness) mm3 samples. E1530.2019 Standards were used to evaluate the Resistance to Thermal Transmission.

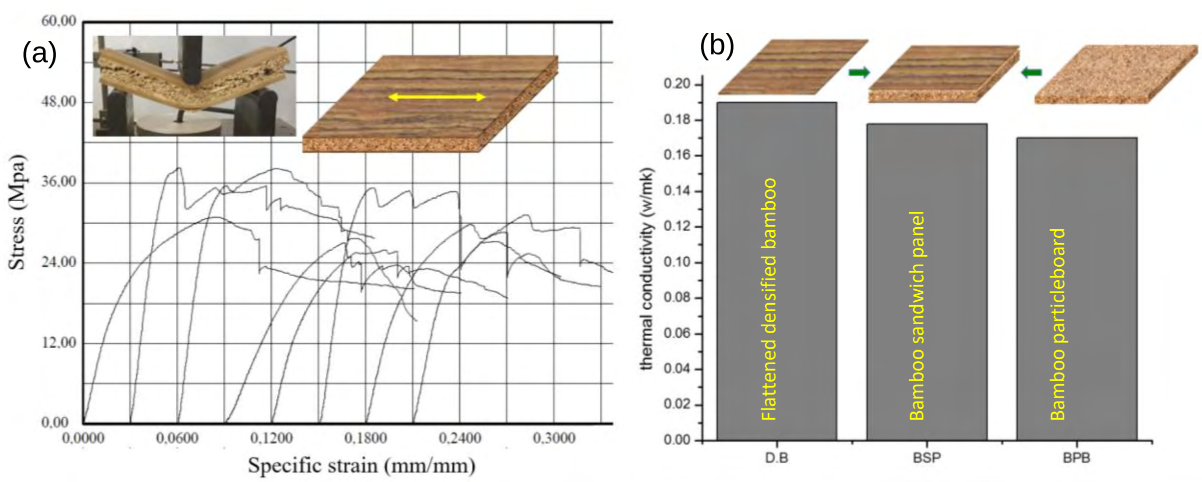
Figure 6.1- The experimental process.

Source: (KADIVAR et al., 2019c)

6.3. Results and Discussion

According to the achieved results, the density of the surface, core, and assembled sandwich panels were in average 993.85 (COV: 0.013), 455.99 (COV: 0.080), 675.52 (COV: 0.036), kg/m3 respectively. Therefore, the BPB panels can be classified as a lightweight material, while BSP has medium density. The individual layers of densified bamboo showed average MOR and MOE values of 349.20 MPa (COV: 0.083) and 30.41 GPa (COV: 0.051) respectively. Using densified bamboo on face sheets of BSPs, the MOR and MOE results in longitudinal direction increased by 917% and 833.3%, respectively, in comparison with the BPB core with the absolute values of 32.11 MPa (COV: 0.14) for MOR and 1.36 GPa (COV: 0.40) for MOE (figure 6.2 a). However, in the transversal direction just a few increments were observed. Low thermal conductivity results, with values of 0.203 W/m-K indicate that BSP can be considered as an insulation material. However, for all the samples, face sheet, core, and assembled sandwich panels, it has been shown a negligible difference on the specific thermal conductivity (figure 6.2 b).

Figure 6.2- Characterization results of BSP. (a) Bending plot; (b) thermal conductivity results.



Source: (KADIVAR et al., 2019c)

6.4. Conclusions

Reinforcing bamboo particleboards by the utilization of flattened densified bamboo, produce a material with 100% utilization ratio, a remarkable increase in flexural strength (compared to the particleboards), and a very good thermal performance which classifies the product as an insulation material.

7. CHAPTER 7 – Conclusions and final remarks

In conclusion, and based on all the literature review and experiments have been done during this doctorate project, densified bamboo not only has the potential to be a high performance product itself but also can be combined with resin and other functional bamboo elements for developing different types of boards. The thermo-mechanical densification process improves the mechanical and physical performance of bamboo, but caution must be taken in selecting the appropriate parameters. Table 7.1 shows some of the improvements in bamboo performance as the result of densification.

Table 7.1- The main improvements in bamboo performance as the result of densification.

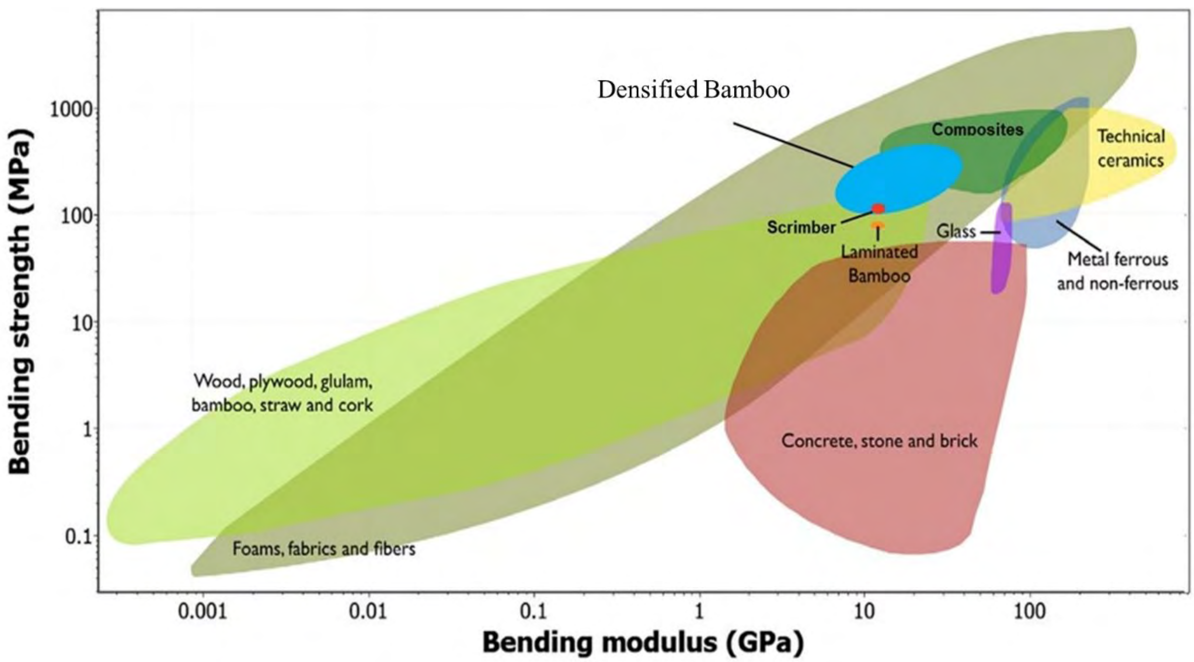
Parameter	**Water Absorption**	**Thickness Swelling**	**MOR (Mpa)**	**MOE (Gpa)**
Natural bamboo	45%	24%	230	19.62
Densified bamboo	23.80%	17.70%	318	27.75
improvements	47%	26%	38 %	41%

Source: Author's authorship.

Densification degree, temperature, initial moisture content, and compression rate are among the main effective parameters that have impacts on densified bamboo. Applying inappropriate values of these parameters can weaken natural bamboo, especially in terms of dimensional stability, which is a feature of physical properties.

The initial moisture content of around 10%, a densification degree between 30-50%, a temperature of 160^{o}C to 200^{o}C, and a compression rate of around 6mm/min are the suggested parameters that can result in achieving the best physical and mechanical performance for bamboo *Dendrocalamus Asper* (Under the conditions of this piece of work), and a time higher than 10 min is required for inner stress relaxation. The water absorption and thickness swelling of densified bamboo after 24 h of immersion in water are 23.80% and 17.70%, respectively, which are considerably lower than that of natural bamboo. The TM process decreases around 50% of water absorption and 26% of thickness swelling. Regarding the bending properties, MOR of densified bamboo is 318 MPa, and MOE is 27.75 GPa, which are higher than un-densified bamboo 38 and 41%, respectively. In comparison to some other engineered bamboo and also various building materials, the bending properties of densified bamboo is shown in Figure 7.1. This figure reveals the bending strength envelope of densified bamboo, and also includes results of the scrimber made of Moso bamboo studied by Sharma 2015 (SHARMA et al., 2015a). The material is within the range of foams, fabrics, and fibers materials, and on average, it is stronger than natural materials such as wood, plywood, glulam, raw bamboo, straw, and cork, and also stronger than concrete, stone, and brick.

Figure 7.1- The Bending modulus vs. bending strength for various construction materials as well as densified bamboo.



Source: Adapted from Sharma 2015(SHARMA et al., 2015a), The flextural properties of densified bamboo were included by the author.

Since the basic characteristics of different bamboo species are not the same, the other bamboo species require their specific prescription for the densification process. There are still questions about the impact of sample dimensions and the type and size of equipment used, chemical pretreatments as well as several other issues that need to be solved to fill the knowledge gap.

A summary of conclusions and recommendations related to each chapter is presented below.

Chapter 2 – Literature review (Densification of Bamboo: State of the Art)

- The bending modulus of rupture (MOR) and modulus of elasticity (MOE) for both natural and densified bamboo are linearly correlated to the density.
- The densification process can be performed either in an open system, the thermo-mechanical (TM) method, or in a closed system by the viscoelastic-thermal-compression (VTC) process approach, using heat and pressure.
- The efficiency of densified bamboo can vary depending on the process parameters.

- Existing studies have evaluated the process mostly in flexural strength and some physical properties. Information regarding the other properties, such as tensile and compression strength of densified bamboo is scarce.

Chapter 3 – The influence of the initial moisture content on densification process of D. Asper bamboo: physical-chemical and bending characterization

- The achieved densification degree, physical, and bending performance were affected by the different starting moisture content (MC).
- Using the same pressing procedure, 140°C temperature, and 4.4 MPa pressure, the higher starting MC resulted in higher densification degree, up to 31.2 %.
- Densifying bamboo with moisture content less than 5% presents macro cracks after the TM process while using an MC higher than 15% exhibits an irregular thickness throughout their length and width caused by blocked water steam within bamboo's structure.

- Applying TM densification process using a temperature less than 160°C negatively affected the dimensional stability (water absorption and thickness swelling) of the densified samples in comparison with the reference.
- The optimum bending properties were obtained by the samples with 10% MC, increasing the MOR and MOE by 56 and 41% respectively, in comparison with un-densified samples.
- TM densification process modifies the interface between parenchyma and fiber bundles, and increases the linkage between both components.
- Through SEM analysis, it was also observed microcracks mainly in 0 % and 5 % MC samples, which can justify the lower bending strength and the unstable behavior after immersion in water.
- Thermo-mechanically bamboo densification process at a temperature lower than 160°C doesn’t have a significant influence in the chemical composition of bamboo.
- TM densified bamboo has a higher cellulose crystallinity index compare to the un-densified ones.

Chapter 4 – Optimization process of thermo-mechanical densification of bamboo

- Temperature is one of the main factors in softening bamboo during the compaction process, which makes the material easier to deform, decreases the modulus of elasticity, and improves the dimensional stability. However, the use of high temperatures can burn bamboo (depends on the time of contact with the plates), and also, if it is associated with high compression rates, it can cause defibrillation.
- Compression at room temperature requires higher loads compared to the process at elevated temperatures.
- Shear failure starts after a densification degree of around 50%, the higher the temperature, the higher the start point of shear failure.
- According to X-ray densitometry analysis, densifying bamboo to 50% DD and using the temperature of 160°C and compression rate of 4 mm/min increases the density of natural

bamboo from 0.74 to 1.30 g/cm3. The densification degree of 70% caused the formation of micro-cracks.

- Water absorption and thickness swelling are linearly correlated with the lateral deformation of the densified bamboo.
- Applying pressure around 18 MPa, DD between 30-50%, a temperature of 200°C, the compression rate of around 6mm/min, and a relaxation time higher than 600 s results in achieving improvement of 4.72%, 23.80%, and 17.70% for spring back, water absorption, and thickness swelling respectively.

Chapter 5 – Physical properties of thermo-hydro-mechanically (THM) flattened and densified bamboo (moso and Dendrocalamus asper)

- Flattened-densified bamboo is one of the application examples of densified bamboo. The applied process had a negative effect on the swelling and water absorption of bamboo. For Moso bamboo, the water absorption (WA) (84%) and thickness swelling (TS) (41%) of the densified samples, in comparison with un-densified ones, was approximately 60% higher. On the other hand, *D. asper* samples had the same behavior of the un-densified samples, with 47% and 23% of WA and TS, respectively.

chapter 6 – Evaluation of an optimized bamboo sandwich panel using layers of two different bamboo functional elements

- Bamboo sandwich panel using densified bamboo on face sheets and bamboo particleboard as the core is another application examples of densified bamboo, with the thermal conductivity of 0.203 W/m-K, MOR of 32.11 MPa, and MOE of 1.36 GPa.

7.1. Sugestions for future research

Many aspects of densified bamboo still need to be investigated. The suggested studies are as follows:

- *Characterisation of densified bamboo after field degradation tests*

- *Fungi decay investigation of densified bamboo*
- *Study of bamboo densification using a closed system*
- *The effect of citric acid modification on the densification process of bamboo:*
- *The effect of pretreatment using chemical materials and oil on densified bamboo*
- *life cycle assessment of the densified bamboo and bamboo panels*

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