Optimization of Thermo-mechanical Densification of Bamboo

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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 adopting 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

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 [1–5]. 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 [6–8]. 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 [9–14]. To solve these difficulties, bamboo needs to undergo special treatments and processes.

Densification processes can be applied to use bamboo more efficiently as an industrial material in modern constructions. By densifying a material and hence increasing its density, mechanical strength can be considerably improved. For instance, by 20-100 % increment in density, the mechanical strength in bending increased by 15-100% for the bamboo species *Phyllostachys edulis* and *Dendrocalamus asper* [15–19]. The applied process and utilized parameters such as temperature, pressure, densification degree, 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 [18,20,21], 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 [22]. 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 [22,23].

Takagi et al. (2008) thermo-mechanically densified bamboo *Phyllostachys bambusoides* at different temperatures ranging from room temperature to 220 °C [16]. Their results showed that the highest temperature results in the highest 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, 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 [22]. Kadivar et al. in 2019 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 [18].

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. It is suggested that densification degree (DD) is one of the most important parameters of the process [15,23] 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). It is defined as the maximum DD of the material without shear failure. In a previous study, Semple et al. (2013) [15] found that the compression of 50% was optimal for *P. edulis* bamboo, 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 [20]. The incomplete relaxation time for the permanent fixation of bamboo shape after densification causes strain recovery, called spring-back.

Although there are several studies to optimize some of these parameters, there is no consolidated information that considers 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) [24] applied the same TM densification method on two different bamboo species, Moso (*P. edulis*) and Madake (*P. bambusoides*) and achieved different results.

Almost all publications related to densified bamboo demonstrate that TM process increases mechanical performance. However, the biggest challenge of densified bamboo is about its dimensional stability, which is compromised by densification [18]. This study aimed to obtain the optimum densification degree of bamboo that can be achieved without shear failure under a specified temperature and compression rate. Moreover, an optimization approach was used to identify the most appropriate processing parameters to achieve the best dimensional stability (lowest spring back, water absorption, and swelling). Clarifying these matters enriches the knowledge of bamboo densification by TM processes and opens new opportunities to encourage the widespread adoption of structural densified bamboo products.

2. Materials and Methods

2.1. Samples

The tests were carried out on samples of bamboo *Dendrocalamus asper* species (3-5 years old) harvested from an experimental field at the University of São Paulo Campus (21°58′53.5″S 47°26′03.3″W). The samples have square dimensions of 30 mm in length and width, and thickness between 10-13 mm (Figure 1). For all the specimens, 100% of their wall thickness was used for the test. The whole thickness was used because this process is thought to be applied on 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) aqueous solution of 8% in a pilot-scale immersion tank, as described in Gauss et al. 2020 [25]. According to Gauss et al. 2019, this chemical treatment does not have a significant influence on the mechanical performance of bamboo [14]. The reason for the treatment is to replicate the normal practice to guarantee the durability of bamboo. The bamboo samples had to be stored before the samples preparation for the densification process and tests. Without any prophylactic treatment, bamboo is prone to insect attack and degradation in a few weeks in tropical countries like Brazil.

2.2. Design of experiments

A new test apparatus (Figure.1) was adapted using the combination of a small press that can be heated up to 200 ºC, an 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 hot pressing. Therefore, it is possible to understand the mechanical behavior of bamboo while increasing pressure at elevated temperatures.

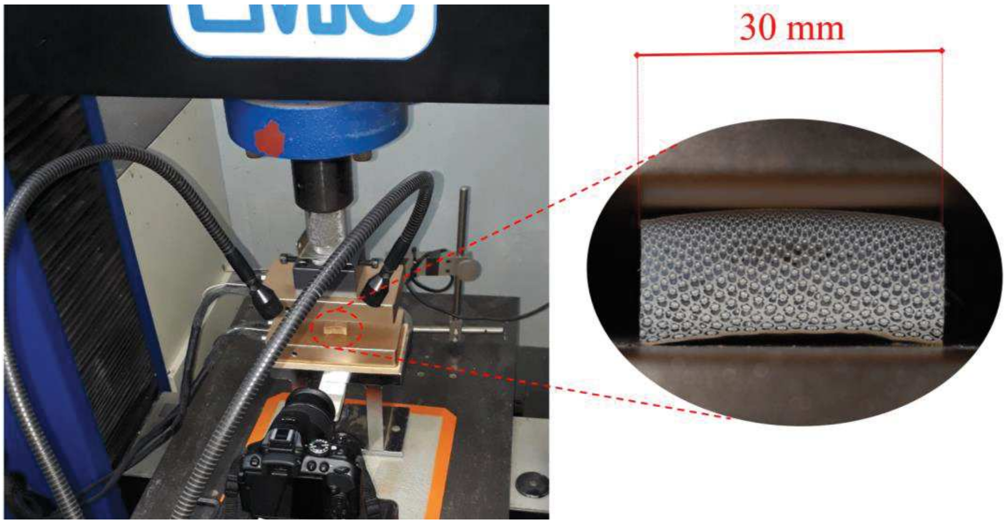


Figure 1- Experimental set-up for the densification process.

The experiments have been performed in two steps to deal with the problems mentioned in the introduction section; i.e. to specify the optimum densification degree of bamboo that can be achieved without shear failure, and the most appropriate process conditions to achieve the best dimensional stability of the samples.

2.2.1. Experimental design for step 1

In this step, the bamboo specimens were subjected to radial compression at different temperatures and compression rates until the samples collapsed (Table 1). There were 9 experimental steps with 5 replicates, requiring a total of 45 densified bamboo samples, under temperature variations of 30, 160, and 200ºC, and compression rates of 2, 4, and 8 mm/min. 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 the DD in which lateral deformation and shear failure start to occur. The temperature and pressure rate are the parameters that are deemed to be optimized for achieving the highest DDcr. The densification degree (DD) was calculated according to Equation 1, where t_0 and t_1 are the thicknesses before and after densification, respectively.

$$DD = (t_o - t_1)/t_o \times 100\% \qquad \text{Eq. 1}$$

2.2.2. Experimental design for step 2

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, 9 experiments with 5 replicates were performed under temperature variations of 30, 160, and 200ºC, and compression rates of 2, 4, and 8 mm/min. In addition to these samples, 16 random temperature treatments (100 and 140ºC) and pressure rates (2 and 4 mm/min) were included in the model for verification purposes. Therefore, a total number of 61 bamboo specimens were tested and densified, and 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 1.

Table 1 - TM parameter levels and responses for each step

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

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

2.3. The density analysis by X-ray densitometry

To verify the step 1, and to see the influence of densification degree, bamboo samples densified to defferent densification degree and analysed by X-ray densitometry test. The control sample or un-densified (T1), densified to 30% (T2), densified to 50% (T3), and densified to 70% of DD (T4). A middle level of temperature, 160 ºC, and a middle level of compression rate, 4 mm/min was choosed to densify the samples. The samples for densitometry analyses were prepared by gluing bamboo culm samples onto wooden supports and cutting them transversely (1.5 mm thickness) with a parallel double circular saw. The samples were then conditioned in a climatic chamber at (20 ± 5) °C and (60 ± 3) % 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 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. 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, Brazil. [26].

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:

$$SB = (t_2-t_1)/t_1 \times 100\% \qquad \text{Eq.2}$$

Where t_1 is the thickness immediately after densification and t_2 is the thickness after conditioning the samples at the chamber.

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 at (105 ± 2) ºC before measuring the weight and volume. Afterwards, they were fully immersed in water at (22 ± 1) ºC, and the weight and thickness changes were measured after 1 h and 24 h. Subsequently, TS and WA were calculated from Equations (3) and (4), respectively. Where m_0 and m_2 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 are the thicknesses before and after the test, respectively.

$$WA = (m_2-m_0)/m_o \times 100\% \qquad \text{Eq.3}$$

$$TS = (t_3-t_2)/t_2 \times 100\% \qquad \text{Eq.4}$$

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 the design of experiments in Minitab software to analyze problems in which a response of interest is influenced by several variables [27]. The relationship among the responses and variables and 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.

3. Results and discussion

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 30ºC (Video 1), and the other from the sample compressed at 200ºC with a compression rate of 8 mm/min (Video 2). Figure 2 also presents the step by step changes of the sample during the densification process at 160ºC 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. Also because of an existing small curvature in the samples, at the beginning of the loading process, the bottom part is under tension while the upper part is under compression. Therefore small flattening occurs. In addition, it was seen that at room temperature, compaction at any compression rate leads to a deep crack in 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 prevents the cracks, as shown in video 2.

Figure 2b shows a sample densified at an elevated temperature; after pure flattening, the bottom part of the sample completely touches the press plate. However, the top of the specimen is still slightly curved and requires more pressure to fully contact the top press plate. Although, increasing the load after pure flattening decreases the thickness, yet the sample is not completely flat (Figure 2c). 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 2d). After the DD_{cr}, increasing load induces lateral deformation, which is the result of fiber detachment and eventually, the collapse of the bamboo structure. Moreover, it is observed that the beginning 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 2e).

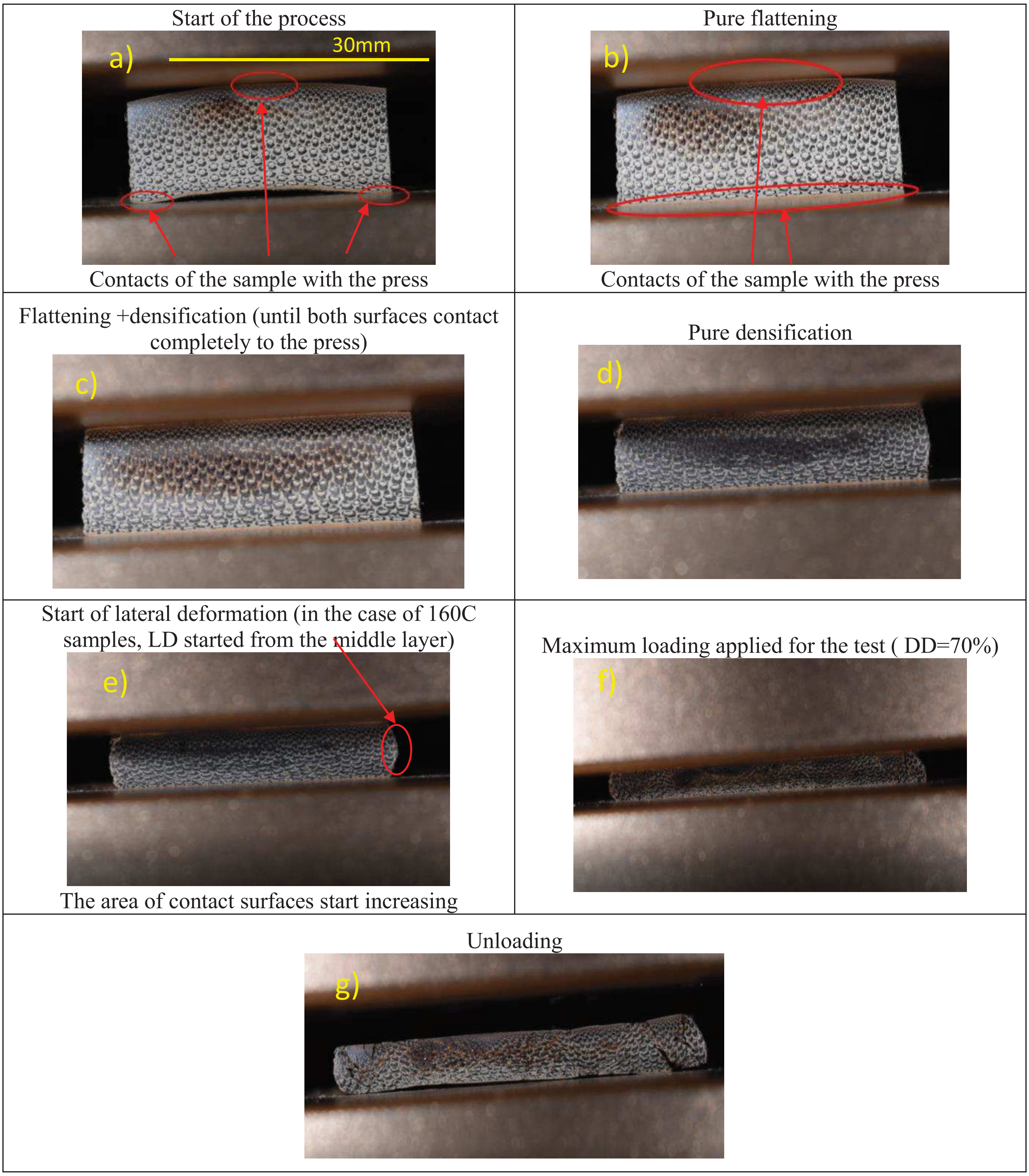


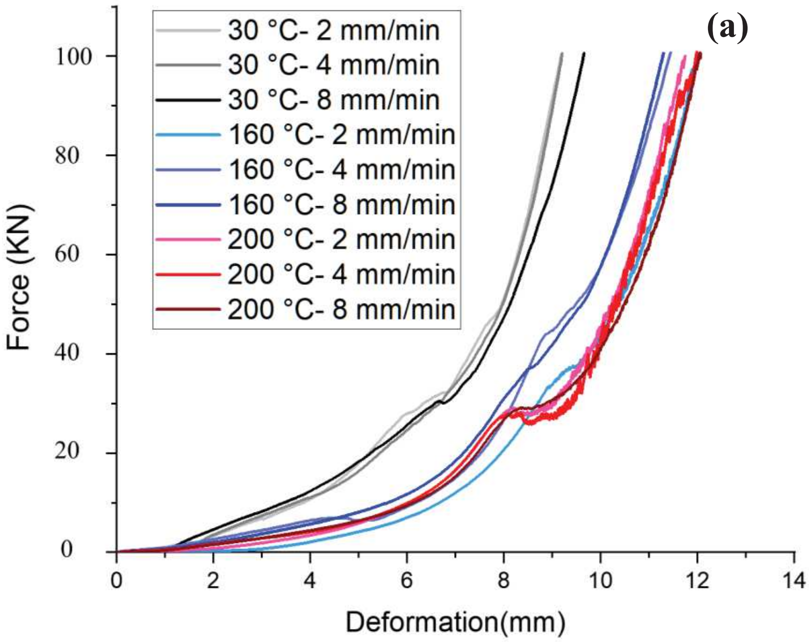
Figure 2 - Step by step changes of the sample during the densification process at 160°C

For each test of the first step, the applied force was plotted against the deformation of the sample. Figure 3a 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.

The effective area during the compression was calculated using a video tracker software to obtain the stress-strain graphs. 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, the length variations were neglected to calculate the area changes during compression. Dividing the force by area, stress/strain graphs were obtained, which are presented in Figure 3b. 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, when the compression 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 compression rate reduces, the samples can have more time to warm up and show more elastic behavior, but it may also continuously dry the sample, which is considered 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 shear failure starting point.



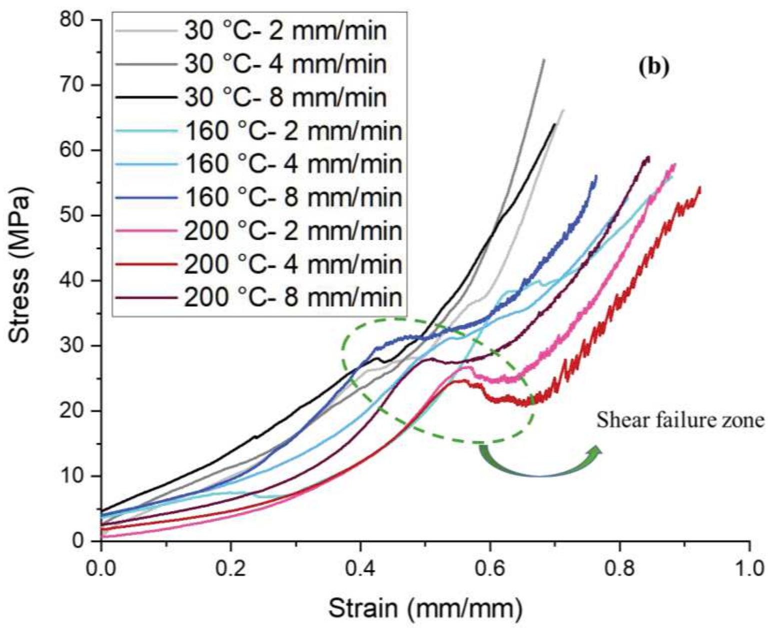


Figure 3 - Load-deformation (a) and stress-strain (b) graphs of the bamboo samples densified at different temperatures and compression rates.

3.2. Optimization results of step 1

Figure 4 demonstrates the results of DDcr versus temperature and pressure rate. The contour plot (Figure 4a) shows the region with the highest DD in a darker color. This graph shows the best range of temperature and compression rate where the material can reach the highest DD without shear failure. 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 in 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 lack of time for relaxing the material during compression.

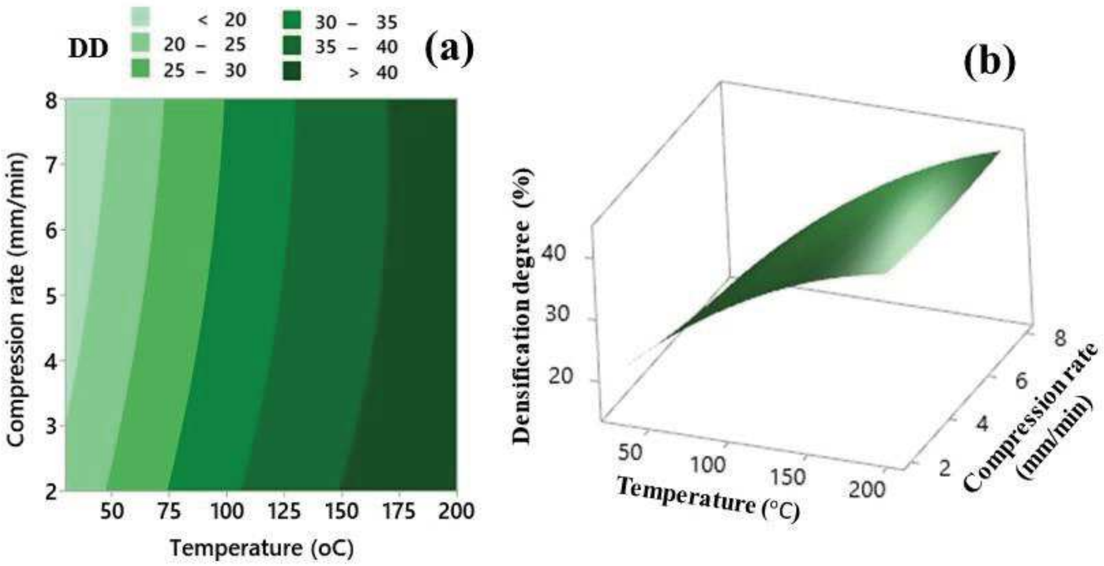


Figure 4 - a) Contour plot and b) Surface plot of DD versus pressure rate and temperature,

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 good compliance with the model.

Figure 5 presents the optimization plot, stating that the maximum DD can be achieved at 200°C with a compression rate of 2 mm/min. The relationship between these two variables is presented in Figure 5, where the red lines indicate the optimum condition. According to this statistical analysis, the maximum achievable DD is about 43.6 using the investigated conditions.

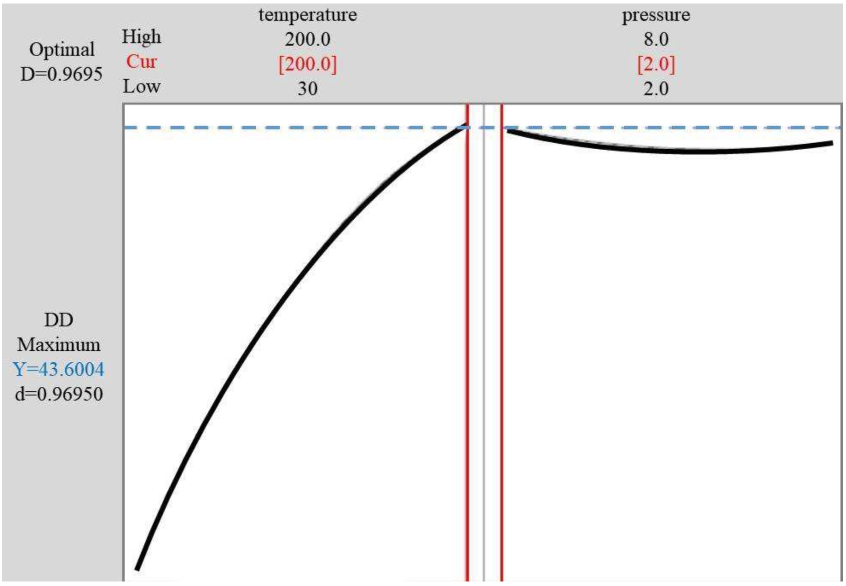


Figure 5 - The optimization plot obtained in step 1.

Although the DDcr increases with temperature, at a high compression rate, some of the samples exploded. Figure 6 shows the detachment of fibers out of the matrix at 200ºC and a compression rate of 8 mm/min. When high compression rates are used, the water inside the material does not have enough time to exit the pores. With water trapped inside the vessels, high steam pressure is generated, causing an explosion.

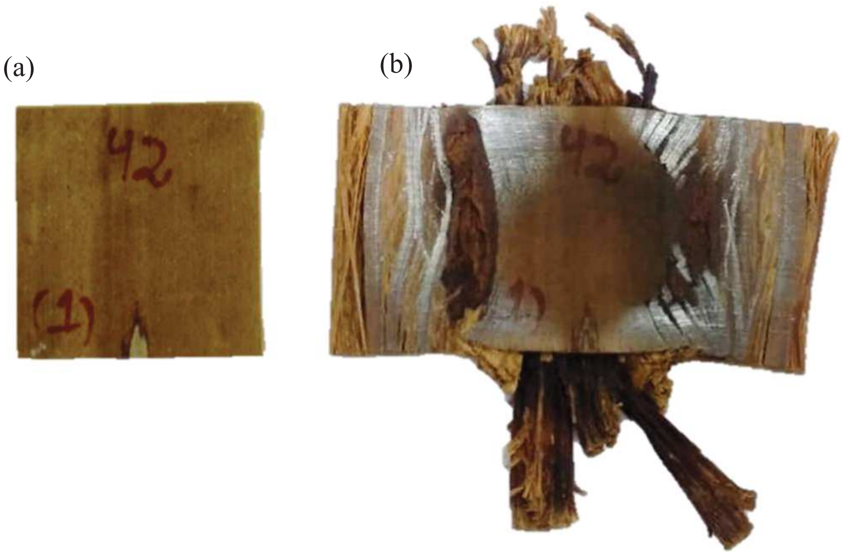


Figure 6 - Bamboo defibrillation caused by steam pressure, a) before densification, b) After densification

3.3. Density profile and microstructure by X-ray densitometry

Figure 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 samples were 0.30, 0.69, 0.83, and 0.35 g/cm^3, while the maximum mean density values were 1.17, 1.45, 1.60, and 1.31 g/cm^3, respectively. The mean and minimum densities tend to increase up to the treatment T3, followed by a drop in density for the T4 sample. Nevertheless, there are no marked differences concerning the treatments for the maximum mean density of the samples.

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 average 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 with 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 the 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 subject to more severe conditions, inducing significant changes in the sample's microdensity profile 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 between the 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.

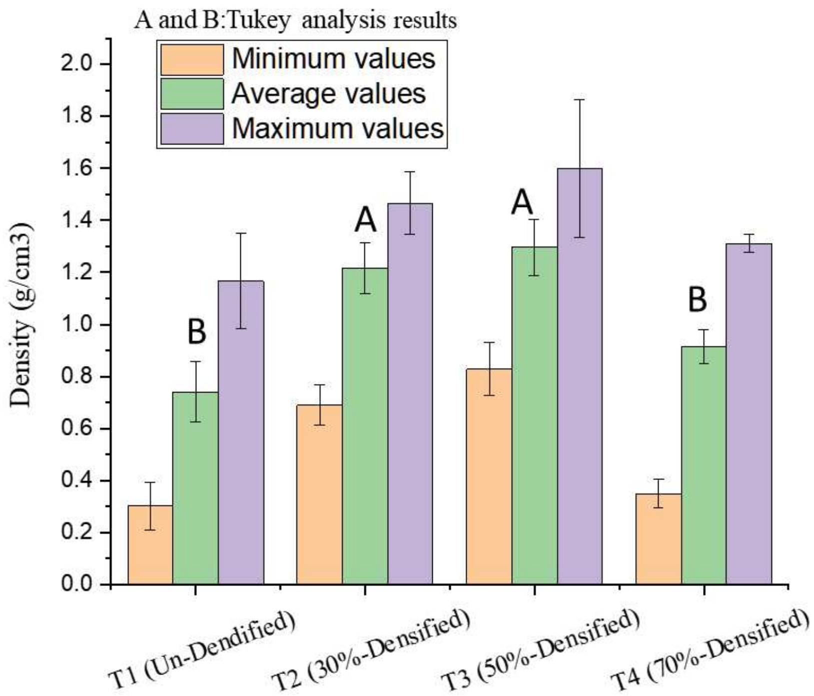


Figure 7 - The values of average, maximum, and minimum densities of bamboo (Same letters (A or B) mean there is no statistical difference among treatment conditions).

In Figure 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.

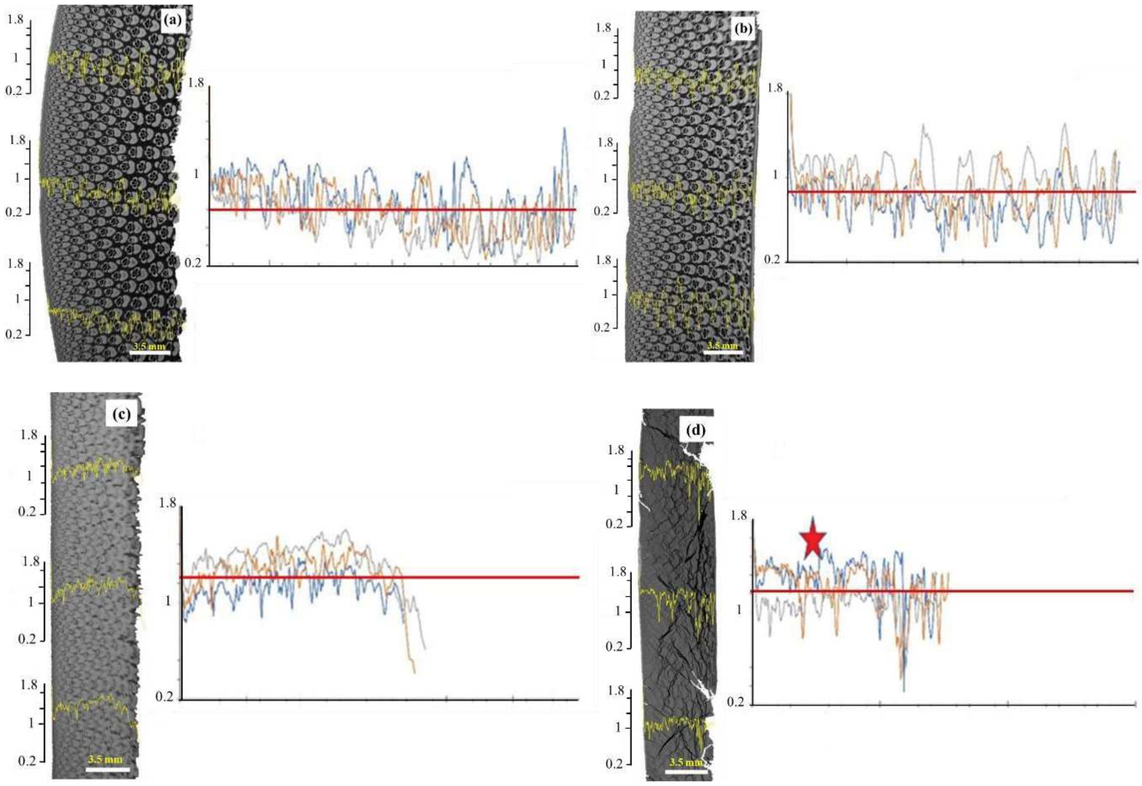


Figure 8 - Bamboo culm microstructure and microdensity profiles (yellow lines show the position): tratamento T1 controle (a); T2 30% DD (b); T3 50% (c); T4 70% DD (d). Scale: 3.5 mm

In Figure 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 8).

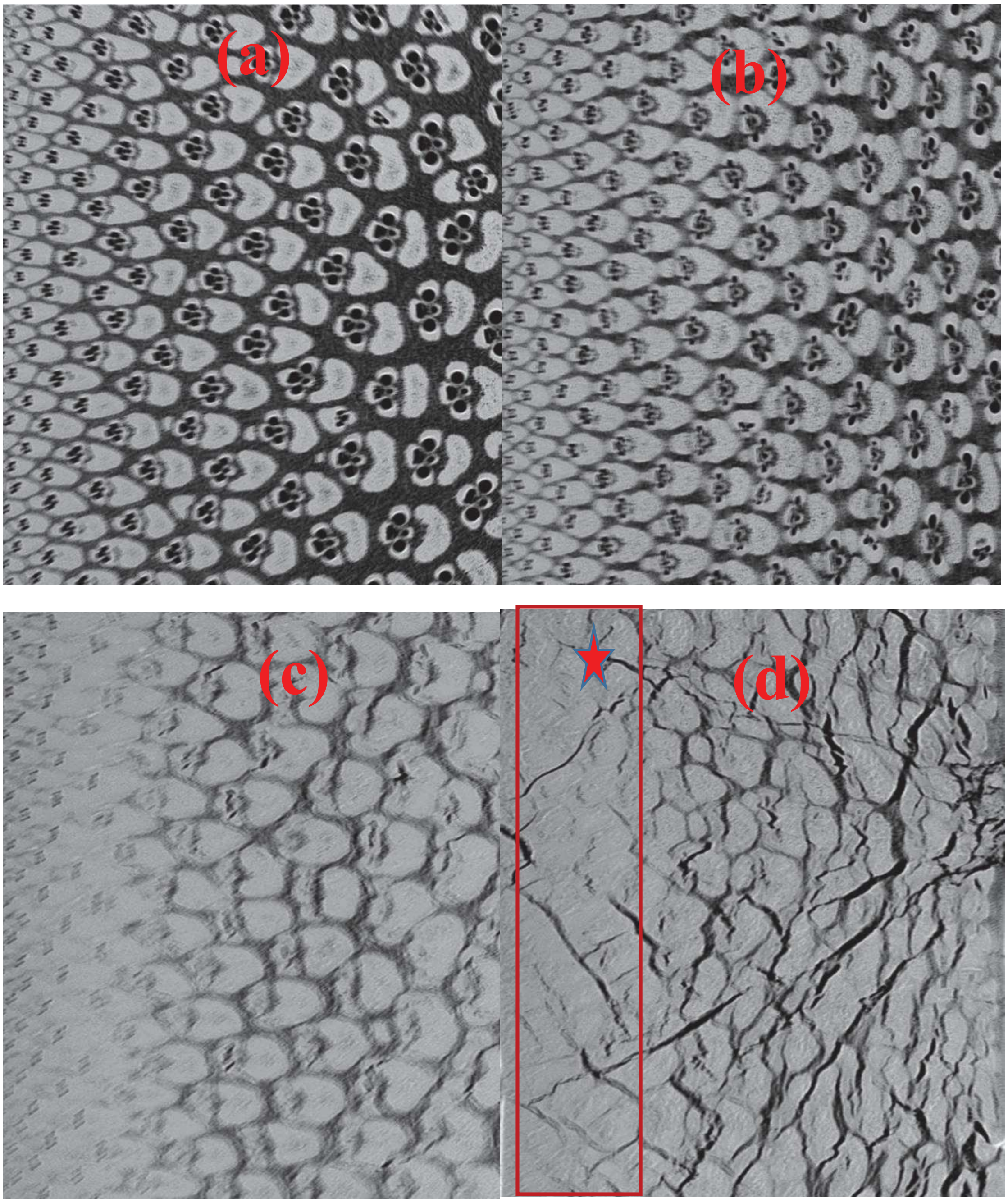


Figure 9 - Bamboo culm microstructure: T1 (control) (a); T2 30% DD (b); T3 50% (c); T4 70% DD (d),

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 fiber cohesion. Therefore, DD=50% and the other parameters mentioned in Table 1, were used for the dimensional stability analysis in this step. The tests were repeated five times for each condition densified at 30, 160, and 200ºC at 2,4, and 8 mm/min of compression rate. The initial MC of the samples was (7±0.5) %, and after the densification process and 60 min of relaxing time, the samples were almost dry. Table 2 shows the value of weight loss for different conditions. Since weight losses at 160 ºC and 200 ºC are higher than initial moisture content, degradation might occur. It has been proved before that bamboo degrade at a temperature higher than 160 ºC [23]. The higher the temperature, the higher the weight loss; however, there is no regular pattern in terms of using different compression rates.

Table 2- Weight losses percentage of samples densified at different temperatures and compression rates (COV in parentheses).

Pressure rate (mm/min) / Temperature (°C)	30	160	200
2	0	10.4 %(0.80)	14.2 % (0.79)
4	0	10.6 % (1.07)	14.6 % (6.08)
8	0	10.8 % (2.17)	14.3 % (6.5)

Considering all the data, Figure 7 illustrates the water absorption and thickness swelling results 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), a better correlation is observed in the WA 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.

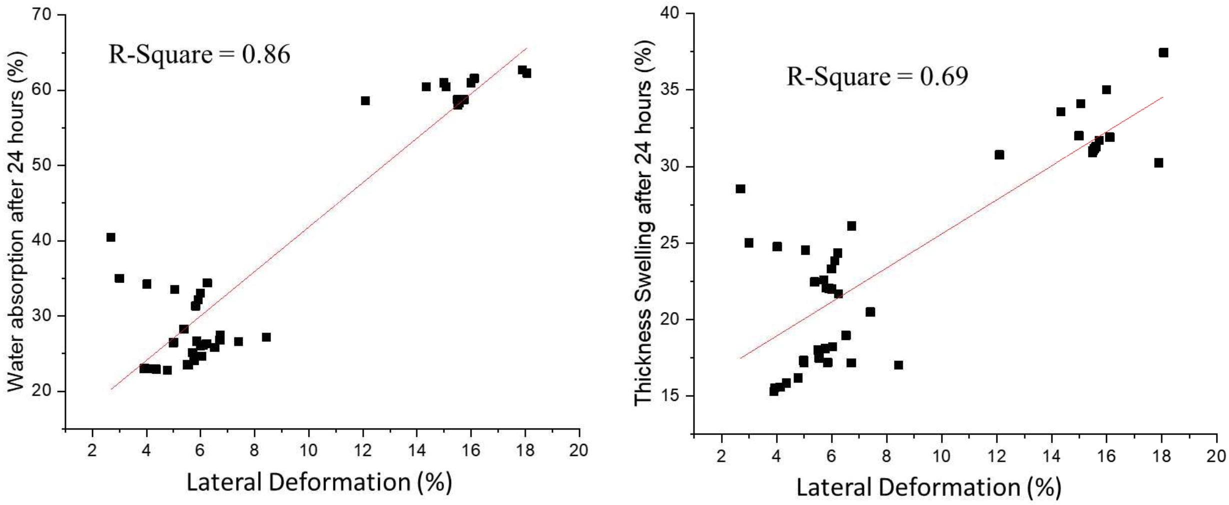


Figure 10 - The correlation of a) water absorption and b) thickness swelling with lateral deformation

Figure 8 presents the maximum stress contour and surface plot, 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. On the other hand, higher temperatures facilitate the relaxation of the internal stresses and increase the plasticity of lignin [28]. Besides, the influence of the compression rate on the maximum stress rate is almost negligible for various temperatures.

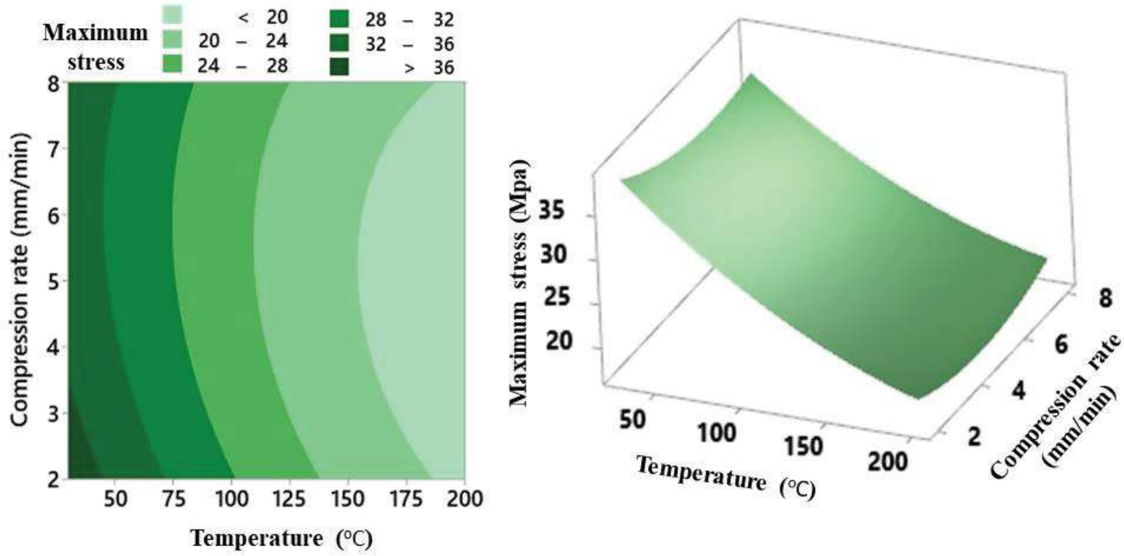


Figure 11 - Counter plot (a), and surface plot (b) of maximum stress, which is required to densify bamboo to DD of 50%, versus pressure rate and temperature.

Variations of lateral deformation of bamboo samples with 50% DD by varying temperature and compression rate are presented in Figure 12 . The analysis indicates that LD is reduced by increasing the temperature with a minimum between 160-200 ºC. 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 ºC by changing the compression rate is attributed to the water loss during the process and the deviation of the test results.

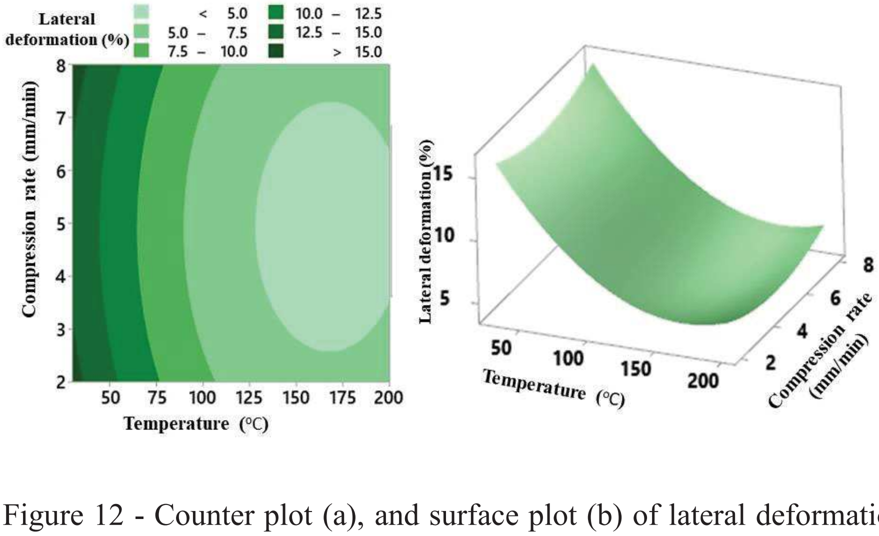


Figure 12 - Counter plot (a), and surface plot (b) of lateral deformation (LD) induced by densifying bamboo to DD of 50%, at different treatment conditions.

Figure 13 demonstrates the spring back (SB) result analysis for the discussed parameters. According to the Figure, the spring-back factor rises with temperature up to around 100 ºC. At this point, the graph reaches its maximum value and tends to decrease again for higher temperatures, which is explained by the increasing elasticity due to 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. Like wood densification, there is very little stress relaxation at lower temperatures, and thus, the deformation is expected to be mainly elastic.

It is also noticeable that the maximum SB happens at moderate pressure rates. A 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.

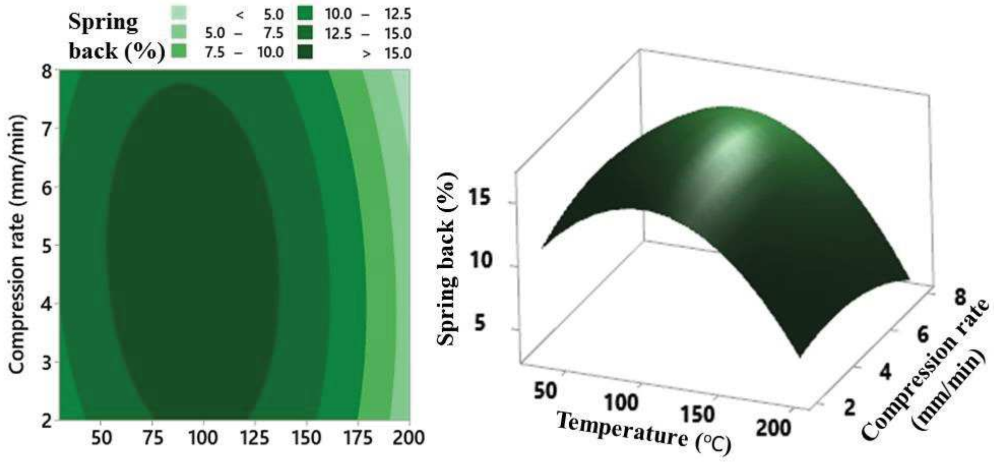


Figure 13 - Counter plot (a), and surface plot (b) of spring back (SB) induced by densifying bamboo to DD of 50%, at different treatment conditions.

The contour and surface plots of water absorption results after 1h and 24h are illustrated in Figure 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 for 1 hr 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 immersion times since the water absorption is related to open defects. It is also worth noticing that the rate of water absorption has increased drastically from a range of 4.5-36.3 to 22.9-60.44 after 1h to 24h, respectively, which was well predicted.

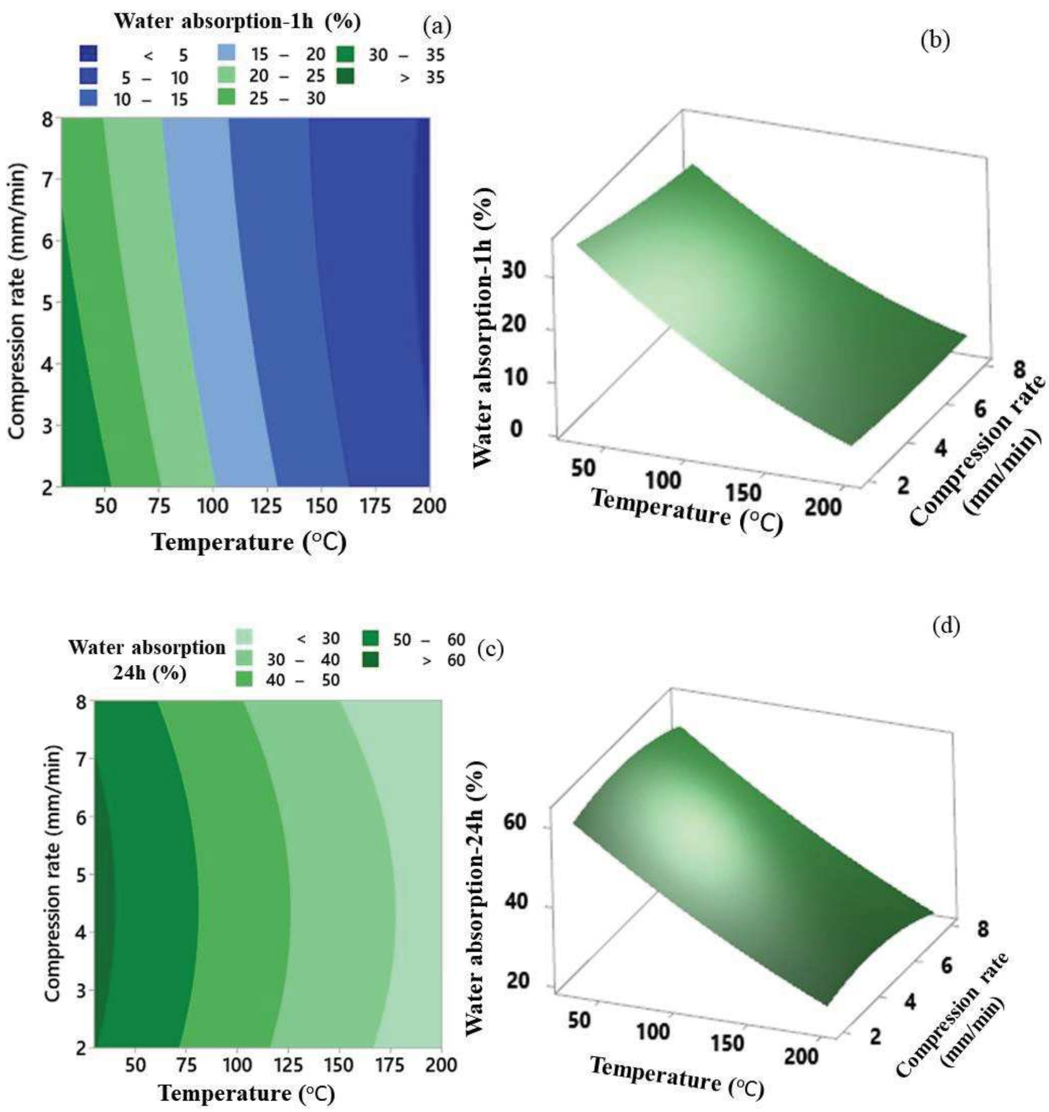


Figure 14 - Optimization analyzes of water absorption (WA) of densified bamboo with DD of 50% at different treatment conditions.

Figure 15 depicts the thickness swelling of the samples after 1 h and 24 h. The influence of the compression rate is more pronounced than on this property. The highest TS occurs at the lowest temperature and compression rates. The influence of temperature on the dimensional stability of densified bamboo is similar to that of wood, considering the similar composition of these two materials. According to the wood literature, 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.

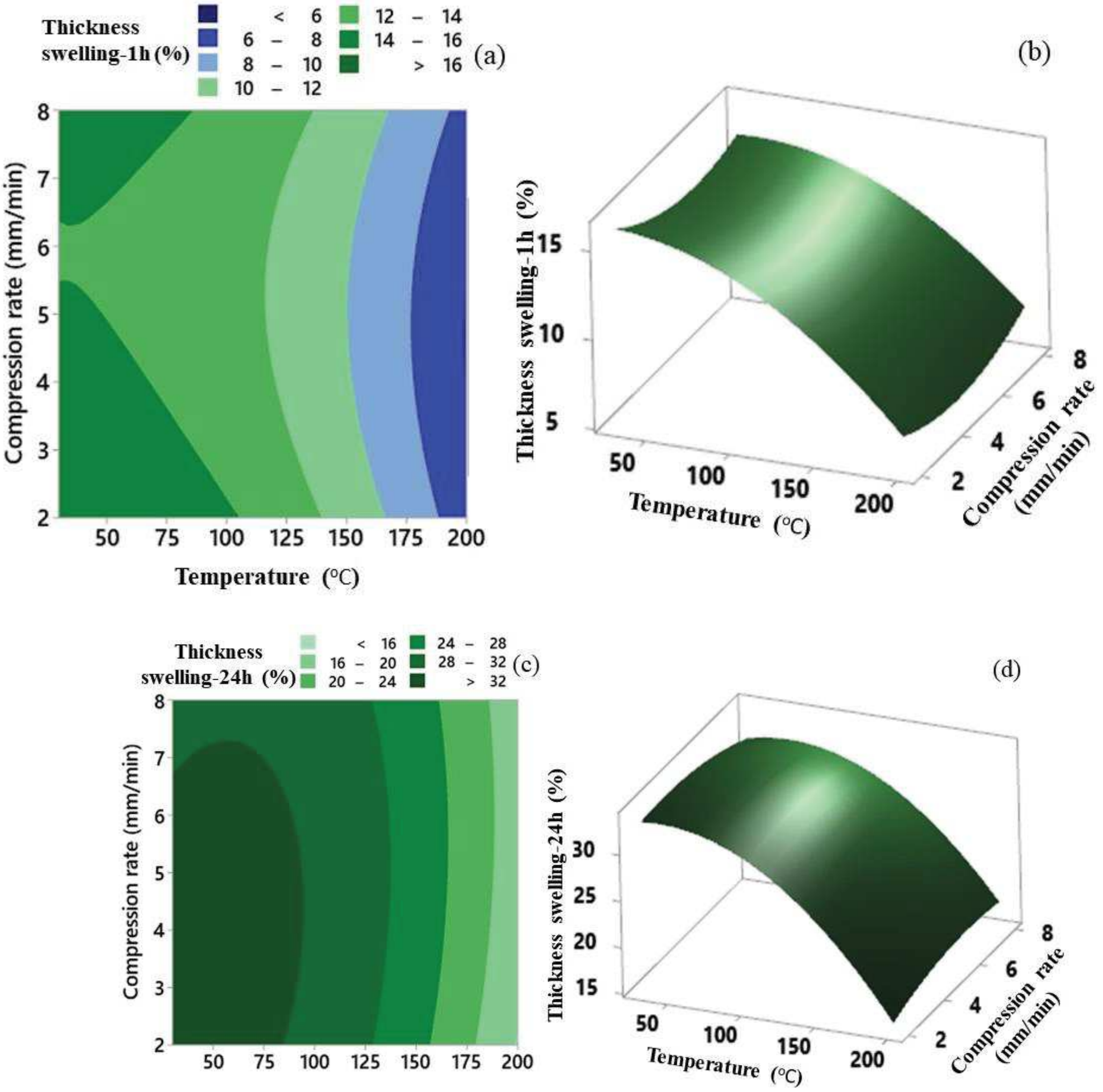


Figure 15 - Optimization analyzes of thickness swelling (TS) induced by densifying bamboo to a DD of 50% at different treatment conditions.

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 200ºC and 6.73 mm/min respectively. The predicted values of responses as the optimum results are shown in Table 3.

Table 3 - Multiple Response Prediction of bamboo densified at 200ºC 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

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 compression rates of 2, 4, and 8 mm/min for 60 minutes. Then, by keeping the displacement constant, the force decreases with time until a constant value. Figure 16 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.

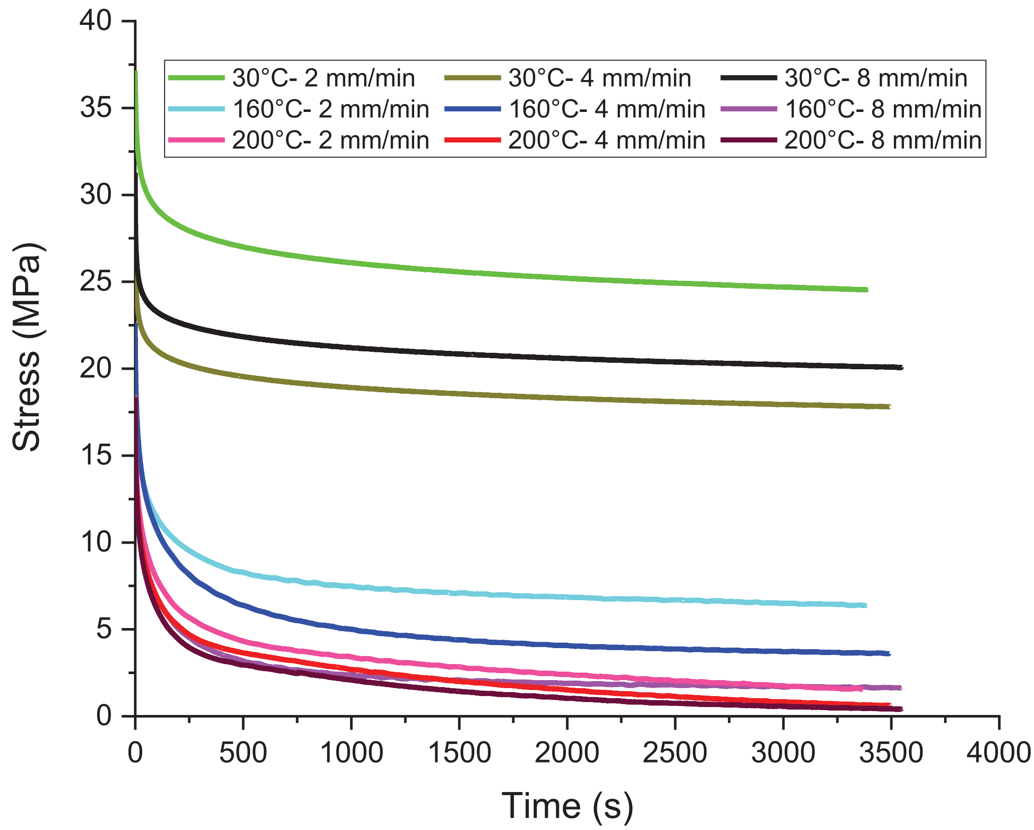


Figure 16 - Force relaxation curves of bamboo samples

4. Conclusions

The present study deals with obtaining an optimum thermo-mechanical treatment to achieve the highest critical densification degree and the best dimensional stability for bamboo strips. The dimensional stability analysis for the samples 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 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, 200ºC, and the lowest compression rate, 2mm/min, are the optimum parameters to achieve the maximum densification degree (DD); the densification degree was more dependent upon temperature than the 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 200ºC, a 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 h and 24 h soaking, and TS after 1 h and 24 h soaking was predicted to be 4.72%, 4.35%, 23.80%, 6.46%, and 17.70% respectively. The obtained optimized parameters are advised for future attempts to produce densified bamboo-based products with improved quality and stability.

5. Funding

The first author thanks the financial support from São Paulo Research Foundation (FAPESP), Grant # 2019/24253-1 and Grant # 2020/00827-6 for developing this work. The second author also thanks FAPESP for the Grant # 2016/26022-9. The last author is grateful to Brazilian National Agency CNPq for the financial aid (Grant # 307723/2017-8).

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