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Elastic properties of thermo-hydro-mechanically modified bamboo (*Guadua angustifolia Kunth*) measured in tension.

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Abstract. *Guadua angustifolia Kunth* (Guadua) was subjected to thermo-hydro-mechanical (THM) treatments that modified its microstructure and mechanical properties. THM treatment was applied to Guadua with the aim of tackling the difficulties in the fabrication of standardised construction materials and to gain a uniform fibre density profile that facilitates prediction of mechanical properties for structural design. Dry and water saturated Guadua samples were subjected to THM treatment. A densified homogenous flat sheet material was obtained. Mechanical properties of small clear specimens of THM modified Guadua were evaluated by testing in tension and compared to the results of the same test on a control specimen. Samples were tested in the elastic range to determine values for Modulus of Elasticity (MOE) and Poisson's ratio.

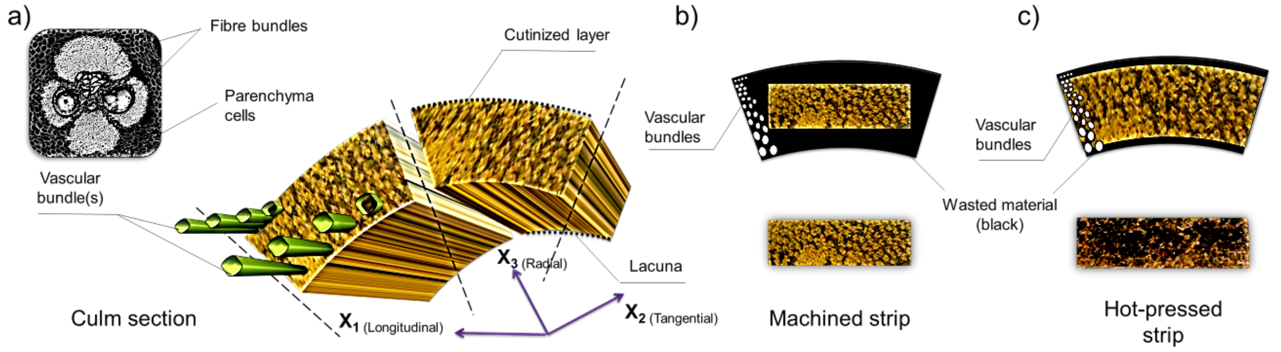
There was a significant increase in the tensile MOE values (parallel to the direction of the fibres) for densified samples. MOE values measured were 16.21 GPa, 22.80 GPa and 31.04 GPa for control, densified dry and densified water saturated samples respectively. Oven dry densities for these samples were 0.54 g/cm^3, 0.81 g/cm^3 and 0.83 g/cm^3. Despite a 50 % reduction in the radial Poisson's ratio for the water saturated sample, no further variation in the Poisson's ratio as a result of densification was observed for control and densified dry samples.

This paper presents the results of the first phase of a study focussed on the manufacturing of flat Guadua sheet (FGS) by THM treatment and the characterization of its mechanical properties. The achievement of a dimensionally stable FGS by THM modification, with a uniform density and achieved with reduced labour effort during manufacture, will be of key importance for the development of structural applications, and could have a significant impact in the bamboo industry. The final aim of the research at the University of Bath is the development of Cross Laminated Guadua (CLG) panels using THM modified and laminated FGS glued with a high performance resin.

1. Introduction.

As in wood, the mechanical properties of bamboo are density dependent. Its considerable strength to weight ratio is greatly influenced by its relatively high density (around 0.81 g/cm^3). The specific modulus of bamboo is usually compared to that of mild steel where the value of elastic modulus per unit density is about 25 x 10^6 m^{2}s^{-2}. However, factors such as the bamboo species, anisotropic properties and density variations across and along the culm hinder its use in stiffness-driven applications where steel has been widely used. Values of MOE in tension for Guadua range from 8 GPa for the basal section to 16.25 GPa for the top part of the culm which also depends on the presence or not of nodes and the fibre volume fraction [1]. Similarly, Poisson's ratio varies from 0.19 for the bottom part to 0.35 for the top part [1, 2].

Engineered bamboo products are scarce and require complex manufacturing processes. The most common practices focus on the extraction of the technical fibres by mechanical and chemical means and the machining of longitudinal strips for the production of Plybamboo. During the machining of these strips, the trapezoidal-like section with round faces is sanded down to a rectangular one after removal of about two thirds of the total material which are regularly discarded or used for fuelling furnaces (Figure 1) [3]. Part of this wasted material is the strongly consolidated tissue of the outer layer which makes the bamboo profile denser and stiffer towards the outside with more vascular bundles and thus mechanical fibres. Therefore, by removing this layer the specific gravity of bamboo is reduced and its mechanical properties are no longer comparable to steel. The negative influence on the mechanical properties of bamboo due to removal of the outer skin as well as the considerable material wasted as a result has been reported by Nakajima *et al.* [4] and Tanaka *et al.* [5]. Both studies have undertaken modifications to the cell structure of bamboo by thermal softening, the former without pressure and the latter with elevated temperature and pressure. These treatments have been widely applied to wood for tissue consolidation and in particular to softwoods to increase the density, improve weather resistance and reduce fungal decay and hygrospicity [6-9]. Thermal softening of lignin is achieved through THM modification. Wood and bamboo have similar chemical compositions [10, 11] with comparable lignin contents which make the use of THM treatments feasible for bamboo.



(a) section of bamboo divided into strips showing the increase in size and the reduction in number of vascular bundles from the cortex to the lacuna; a microscopy image shows the distribution of fibres and vessels which are surrounded by parenchyma cells. b) in black the material wasted by the machining of bamboo strips during the conventional processing. (c) in black the material discarded prior to the THMT.

Figure 11. Diagram of the traditional sectioning and machining of bamboo into rectangular strips and the proposed THMT to trapezoidal strips.

Comparison between the thermal softening properties of bamboo *Phyllostachys bambusoides* (madake) and Japanese cypress *Chamaecyparis obtusa* (hinoki) have been reported by Nakajima *et al*. [4]; where non-pressed samples of bamboo showed thermal softening of lignin at around 60^{o} C with a considerable decrease in the relative relaxation modulus. Cherdchim *et al*. [12] studied the thermal softening of black-sweet bamboo culms in linseed oil and produced flat sheets after applying load. Kitazawa *et al*. [13] used 1.17 kW microwave irradiation to thermally soften bamboo *Madake* and forced it to pass through a conical rig with the aim of modifying its macrostructure and densify its circular section. The viscoelastic behaviour of bamboo immersed in water and its effect on the mechanical properties has also been studied by Amada & Lakes [14] where a basic hydro-modification plasticized the material.

Overall, these studies have made use of the viscoelastic behaviour of bamboo, facilitating the flow (plasticization) of lignin by increasing the moisture content which leads to the reduction of the glass transition temperature (Tg). This has a positive effect on the modification of bamboo's macrostructure and microstructure when applying elevated temperature and pressure. The modification of the structure of bamboo through THM treatment can lead to the production of engineered materials.

This study aims to provide a wider understanding of the use of THM processing of bamboo and its potential for construction applications by transforming the cylindrical shape of bamboo into a flat sheet material suitable for cross lamination in structural panels. The elastic properties were evaluated to provide data for finite element modelling (FEM).

2. Materials and methods.

The species of bamboo used for this study is *Guadua angustifolia Kunth* (Guadua), a bamboo species endemic to South America. Sections in between the nodes of dry Guadua culms from the middle part (in height) were cut to produce the samples and no preservative treatment was applied to them. The outer skin (cutinized layer) situated in the cortex and the inner lacuna (pith) was removed by mechanical means (Figure 1).

Samples were classified into A, B and C, depending on the treatment undertaken (Table 1). THM treatment was performed on dry (Sample B) and pre-soaked samples (Sample C) whilst Sample A was the control and was not subjected to any treatment. Specimens for sample C were immersed in water for 24 hours preceding THM modification. Subsequently, samples were oven-dried at 105^o C and machined to the required shape and stored at a controlled temperature (27^o C ± 2^o C) and relative humidity (70 ± 5 %) in a conditioning room, enabling them to reach equilibrium at 12% moisture content. Dimensions and weight of each specimen were checked with digital callipers and scales before the test in order to calculate the average density (Table 1).

2.1. THM modifications of Guadua.

An open THM system was utilized for the densification of the samples. The compression was applied by a 200 mm x 200 mm Moore manual press with heated platens mounted on four vertical columns. The press was equipped with analogue gauges for pressure measurement, a digital temperature control and a 40 tonne capacity manual jack.

As seen in Table 1, the THM treatment was applied to two samples of Guadua (B and C) which had different pre-treatments. Compression was applied across the radial direction. A control sample (A) from the same region (middle part) was used to compare the results of mechanical testing. The compression set value for samples B and C changed due to the natural variability in thickness of the material; however the target value was about 45%. The compression set value is defined as:

$$C = (Ro-Rc)/Ro \qquad (1)$$

where Ro and Rc are the thickness of the samples before and after compression respectively.

Table 11. THM treatments applied to Guadua

Bamboo species: *Guadua angustifolia Kunth*		Section: Middle (in height)	Age: Mature (3-5 years old)
	Samples A Raw un-processed	Samples B Hot pressed + Dried	Samples C Pre-soaked + Hot pressed + Dried
Time	0	20 min	20 min
Pressure	0	60 kg/cm^2	60 kg/cm^2
Temperature	0	150^o C	150^o C
Compress. Set (C)	0	46.08%	42.51%
Oven dried density	0.54 g/cm^3	0.81 g/cm^3	0.83 g/cm^3

Figure 2 shows the diagram of THM treatment and a picture of the press used. The temperature used for pressing was 150^oC and the pressure applied was 60 kg/cm^2 (6.2 MPa). Sample C was pre-soaked in water for 24 hours prior to THM pressing. The process started with a 10 minute period to allow the plasticisation of the sample where both pressure and temperature increased to 6.20 MPa and 150^{o}C respectively. This temperature and pressure were held for 10 minutes during the densification stage. Before cooling the sample down, a final increase in pressure was applied to achieve the compressive set ratio or preferred thickness.

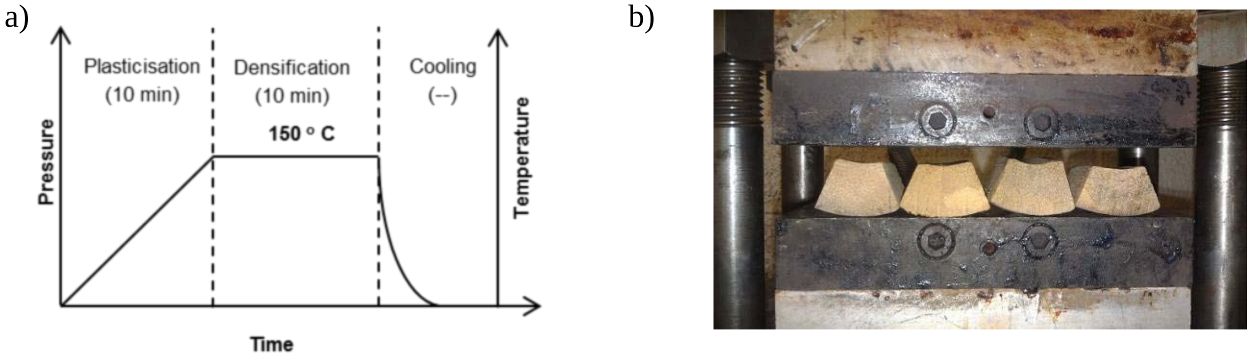


Figure 22. a) Diagram of the THMT, b) Samples in the press before the treatment.

2.2. Microscopy.

Microstructural modifications of Guadua tissues were observed using optical microscopes. A Leica DM ILM inverted reflected-light microscope and a Leica M 205C stereo microscope were used to record the changes which occurred during the densification process. Some samples were embedded in epoxy resin.

2.2. Testing and determination of MOE and Poisson's ratio.

An experimental programme was devised with the aim of characterizing the mechanical properties of the modified Guadua. A longitudinal tensile test was carried along the X_1, L (longitudinal) direction and strain measured in this direction. As seen in Figure 3 (a) during the same test, passive strains in the X_2, T and X_3, R directions (tangential and radial respectively) were also recorded. Modulus of Elasticity (MOE) and Poisson's ratio (υ) were calculated from the experimental data.

The test was undertaken on small clear samples of Guadua following standards for bamboo from ISO 22157 parts 1 and 2 [15, 16], NTC 5525 [17] and for wood from BS 373-1957, BS EN 408-2010 and BS EN 789-2004 [18-20]. Micro-strain gauges, with a grid resistance of 350.0±0.3% ohms, were attached to the specimens for measuring the deformation (Figure 3-d); the electrical signal was recorded continuously during loading. Four specimens in each sample were used for the test and five repetitions per each 500 N increase in load were recorded.

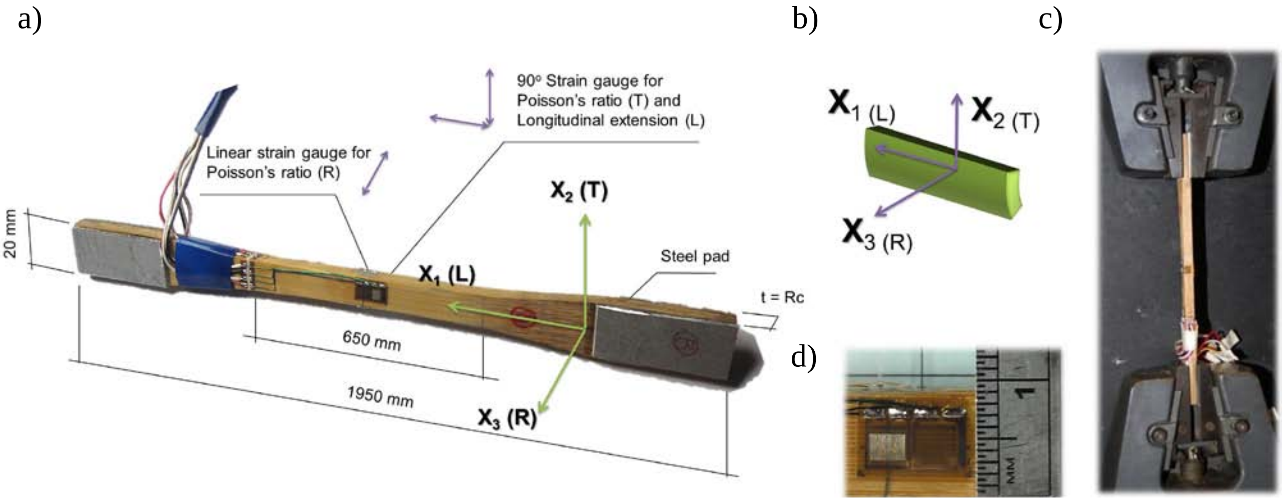


Figure 33. (a) Dimensions of the tensile test sample (b) Specimen orientation (c) Specimen in the Instron wedge grips (d) Detail of the strain gauges used.

An INSTRON 5585H floor model testing machine with a 200kN load cell was used for the tensile tests and a Vishay System 6000 (Model 6100) Data Logger was used to record the data from the micro-strain gauges. The rate used for the test was 0.5 mm/min which complied with the standard's requirement of reaching maximum load within 300±120s, and avoided creep. Steel pads were glued to the ends of the sample to improve the grip in the auto-adjustable wedge jaws. Figure 3 illustrates the specimen orientation, the dimensions of the samples and the test set up.

Strain was recorded below the proportional elastic limit. One specimen per sample was tested to failure to determine the maximum load and the proportional limit was determined. The effect of the presence of internodes was neglected as the material for the proposed Cross-Laminated Guadua (CLG) panels is considered to be an orthotropic continuum flat sheet with symmetry axes identified [21].

For the determination of MOE the graph load-deformation response was plotted and a linear regression analysis performed. The longest portion of the graph with a correlation coefficient ≥ 0.99 was used to determine the MOE. Poisson's ratio (υ) was obtained from the ratio of passive strain (along tangential = X_T and radial = X_R directions) to active strain (along the direction of the load applied = X_L) which is denoted by:

$$\upsilon_{LR} = \varepsilon_R / \varepsilon_L \tag{2}$$

Poisson's ratio in the LR plane caused by a load applied in X_L (radial Poisson's ratio).

$$\upsilon_{LT} = \varepsilon_T / \varepsilon_L \tag{3}$$

Poisson's ratio in the LT plane caused by a load applied in X_L (tangential Poisson's ratio).

3. Results and discussion

3.2. THM modification of Guadua.

The main aim of the THM process applied to Guadua was to create a more even distribution of fibres across the section to improve its mechanical properties. In its natural state bamboo exhibits variation in its mechanical properties across its wall thickness and also along its height [11, 22-26]. Li & Shen [27] reported an average 50% increase in the crosswise volume fraction of vascular bundles and a factor of two increase in MOE from the inner to the outer surface.

Guadua was densified in the radial direction following the treatments defined in section 2. Sample C had a relatively lower degree of densification than sample B, set at 42.51% and 46.08% respectively. The oven dry density for sample C was 35% higher than the density of the control sample (from 0.54 g/cm^3 to 0.83 g/cm^3); these results can be seen in Table 1 and Figure 4b. THMT of these samples reached a slightly higher level of densification using the same time and temperature condition used for sample B.

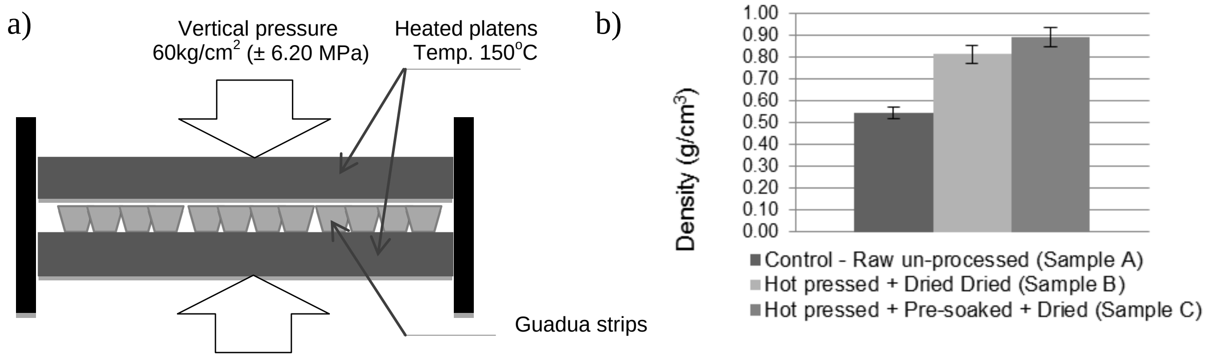


Figure 44. (a) Diagram of the densification process (b) Graph of the oven dry density of Samples A, B, and C.

THM treated samples (B and C) were darker in colour (Figure 5) which is due to the hydrolization of polysaccharides and lignin [9]. The temperature used (150^{o}C) was adequate for the treatment and no external carbonization or degradation of the cell wall components was observed.

Previous experimentation with densified Guadua samples at the University of Bath also showed a considerable increase in the hardness of inner and outer layers of Guadua after the hot press treatment.

3.3. Microscopical analysis of the microstructure of Guadua after THMT.

The degree of densification applied to Samples B and C in comparison to the control sample (Sample A) can be observed in Figure 5.

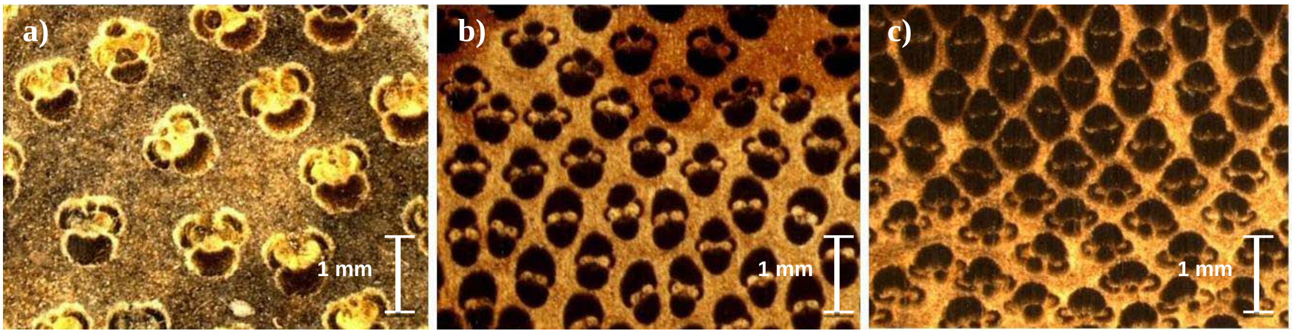


Figure 5. Stereo microscope images of Samples A (a), B (b) and C (c)

Optical microscopy of the cross section shows the closing of the lumen (vascular conducts) as it occurs in wood. Collapse of empty cavities such as metaxylem vessels, protoxylem and phloem has also been observed (Figures 5-7). Volume reduction of void spaces is due to the buckling of parenchyma cells whereas fibre bundles come closer as seen in Figure 8.

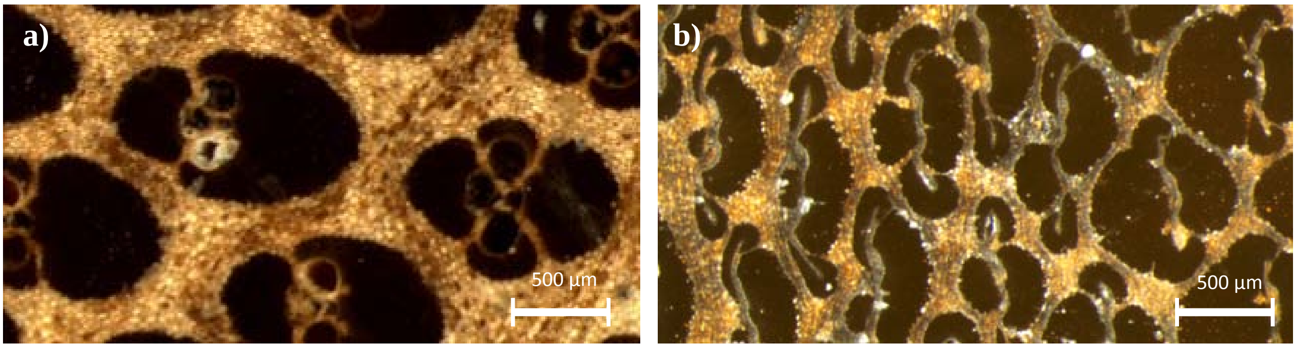


Figure 6. Stereo microscope images (a) before densification and (b) following THMT.

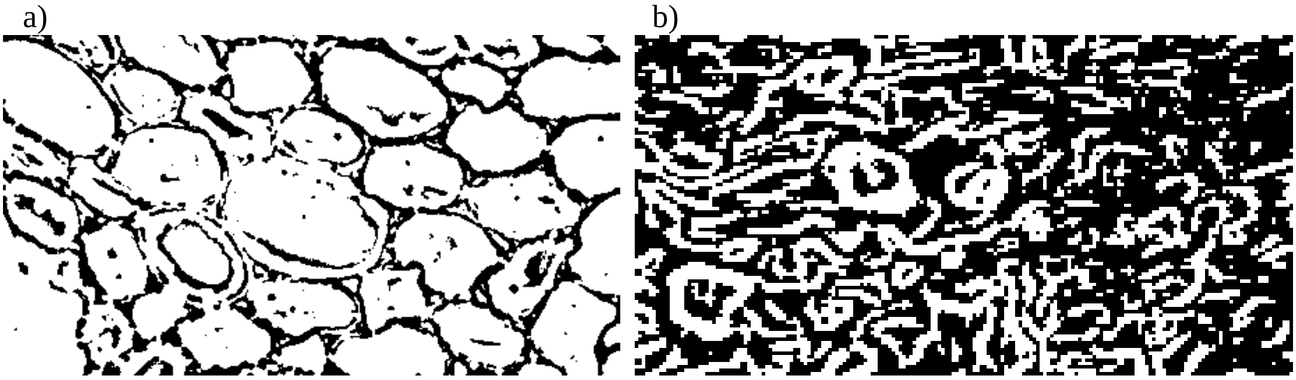


Figure 7. Digitally contrasted pictures from two inverted reflected-light microscope images of the same region before (a) and after (b) densification.

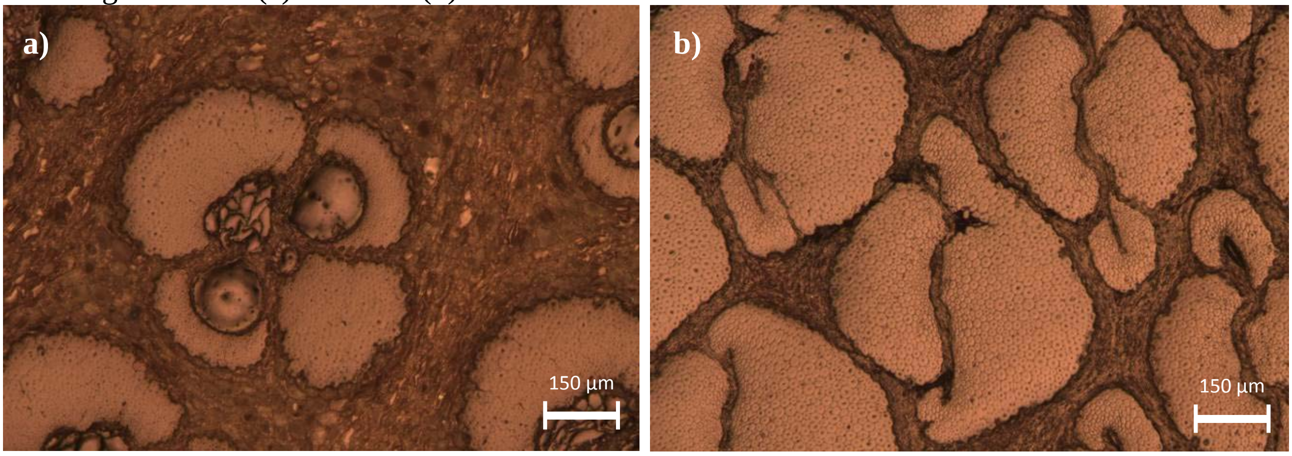


Figure 8. Inverted reflected-light microscope image (a) Sample A (b) Sample C

Density of samples increased from 0.54 g/cm^3 (A) to 0.81 g/cm^3 (B) and 0.89 g/cm^3 (C).

3.4. Determination of MOE and Poisson's ratio by longitudinal tensile test.

As in case for timber, issues regarding the natural variability of bamboo and its anisotropy complicate characterization of its mechanical properties. Bamboo also shows viscoelastic behaviour [28] and behaves semi-plastically at high strain. Graphs of load versus displacement in the elastic region showed loops that never returned to the initial zero position, however the stress-strain gradient was constant which illustrates the consistency of the results.

The typical stress-strain response obtained from the longitudinal tensile tests and MOE results for Samples A, B and C are shown in Fig. 9 (a) and (b) respectively. The higher volume fraction of the densified samples has strongly influenced the stiffness of the THM treated Guadua.

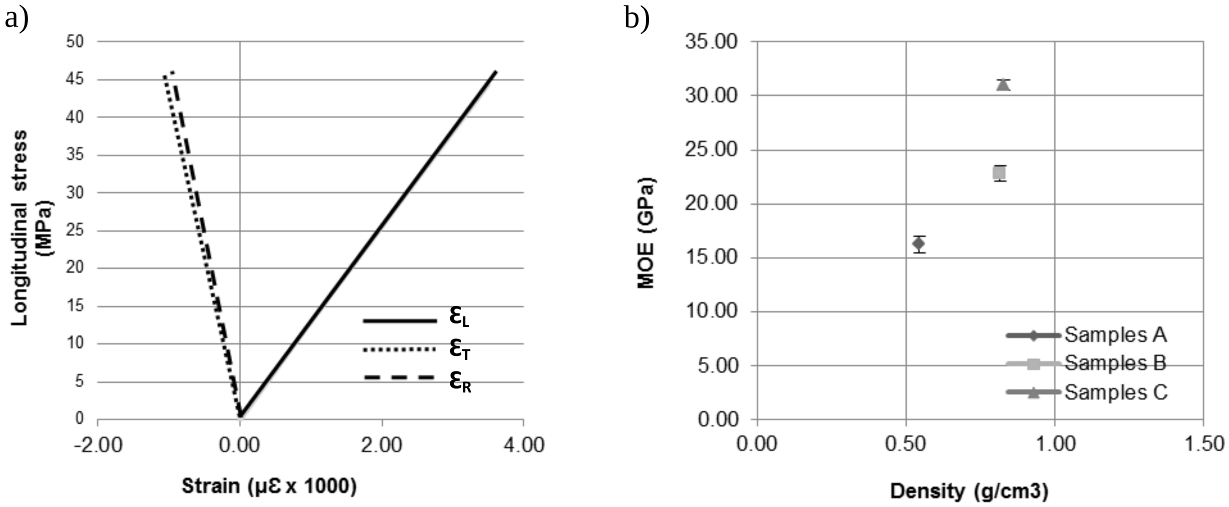


Figure 9. (a) Typical strain-stress graph of the tensile test carried on samples A, B and C (b) MOE versus density for samples A, B and C.

Results for MOE longitudinal to the direction of the fibres (E_L) show an increase of almost 200% compared to the value of the control sample. Despite the small difference in the density ratio between Samples B and C, a significant increase in the MOE values for sample C indicates effective plasticization of Guadua resulting in a densified profile with improved mechanical properties. Thus, the specific stiffness (specific modulus) of Guadua has also increased.

Table 22. MOE and Poisson's ratio results for samples A, B, C.

	Sample A	Sample B	Sample C
E_L	16.21 GPa	22.80 GPa	31.04 GPa
STA DEV	0.76 GPa	0.73 GPa	0.47 GPa
Density (ρ)	0.54 g/cm^3	0.81 g/cm^3	0.83 g/cm^3
Specific stiffness (average)	29.84	27.99	37.58
ν_{LT}	0.27	0.33	0.27
STA DEV	0.01	0.01	0.01
ν_{LR}	0.29	0.33	0.14
STA DEV	0.02	0.01	0.02

Despite a significant decrease in ν_{LR} for sample C (50 % reduction), no further variation in the Poisson's ratio as a result of densification was observed for samples A and B. Values for the tangential and radial Poisson's ratio of the control sample (A) were similar to those reported in the literature [1,2]. Some of the strain values for Poisson's ratio recorded for sample C were very low and some had a positive tendency. This might be due to the prior saturation in water applied to sample C that influences the way cell structures are modified during THM processing.

Although some indentation of the jaws was observed on the samples, no-failure at the grips was experienced. It was also observed after THM pressing that the surface roughness of Guadua samples (B, C) was lower than that of non-densified Guadua samples (A).

4. Conclusions and recommendations.

The main aim of this work was to demonstrate that THM treatments similar to those applied to timber can be applied to bamboo for improving its mechanical properties and providing a new and more predictable material for structural cross-laminated Guadua (CLG) which is under development at the University of Bath. These panels address the difficulties of using round bamboo in construction [29] through the use of a straight-forward densification method that can be easily applied in industry. Furthermore, 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 sample C.

A well-known benefit from THM modification is the reduction of the water uptake in timber [12]. However, further studies need to be undertaken to determine whether a similar behaviour can be expected for Guadua and bamboos in general. Relaxation behaviour in wood, the effects of moisture content on the Tg and the changes in the cell wall structure and chemical composition have been widely studied [30], however, little investigation into the subject has been carried out for bamboo to date.

The set temperature for the densification was decided after previous trials to be 150^{o}C, which seemed appropriate for the treatments However, longer times might need to be considered to avoid the collapse of cell structures.

Further testing and microstructural analysis of the water saturated samples subjected to THM modification need to be undertaken to validate the low values reported for radial Poisson's ratio and understand the microstructural changes which occurred during the treatment.

5. Acknowledgments

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