

# Effect of Smoke Treatment on Flexural Strength of Bamboo Hierarchical Structure

Raviduth Ramful<sup>a,b,\*</sup> and Atsushi Sakuma<sup>c</sup>

Despite bamboo's noteworthy durability, the incidental effects of smoke treatment on the mechanical properties of bamboo culms, including its underlying mechanisms, have not been fully investigated. This study investigated the effects of smoke treatment on the flexural strength of Madake bamboo's (*Phyllostachys bambusoides*) hierarchical structure. Results in small clear specimens displayed an asymmetrical flexural behaviour regardless of the applied treatment, and the parameters of flexural strain and specific energy absorption, demonstrated by modulus of elasticity and modulus of rupture, were found to differ. Concerning compression, parenchyma cells had good ability to absorb large deformation, indicated by their large increase in specific energy absorption. In addition, a distinct difference was found between smoke-treated bamboo and untreated bamboo as the capacity of its outermost fibres to withstand greater tensile load was impaired, indicated by the reduction in flexural strain. Thermal degradation caused an increase in the hydrophobicity of bamboo's outermost layers, thereby engendering higher brittleness in the smoked bamboo. This work highlights critical changes in the mechanical properties of smoked bamboo, which can be addressed in future studies to improve its strength as a sustainable construction material.

*Keywords: Smoke treatment; Madake bamboo; Flexural strength; Flexural strain; Specific energy absorption; Hierarchical structure*

*Contact information: a: Graduate School of Science and Technology, Kyoto Institute of Technology (KIT), Matsugasaki, Sakyo-ku, Kyoto, 606-8585, Japan; b: Mechanical and Production Engineering Department, Faculty of Engineering, University of Mauritius, Reduit, 80837, Mauritius; c: Department of Advanced Fibro-Science, Faculty of Fiber Science and Engineering, Kyoto Institute of Technology (KIT), Matsugasaki, Sakyo-ku, Kyoto, 606-8585, Japan; \*Corresponding author: r.ramful@uom.ac.mu*

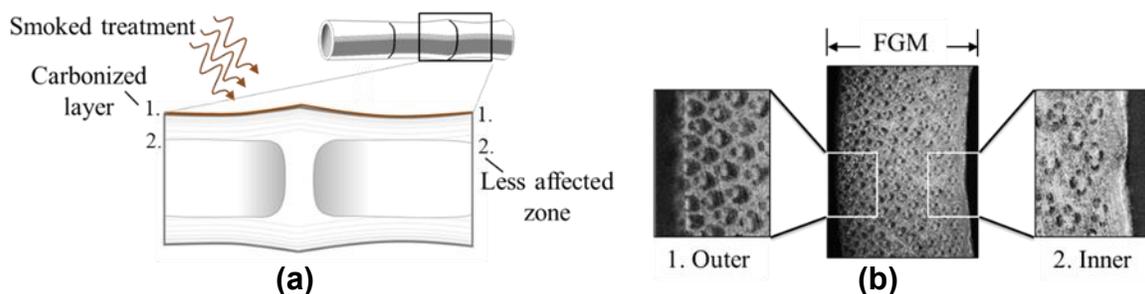
## INTRODUCTION

Bamboo has been extensively used in the past in its raw state as a prominent construction material, as it requires little modification. Being a sustainable material, bamboo is regarded as an excellent alternative to conventional building materials, such as steel and concrete, given its excellent structural efficiency based on an exceptionally high modulus of elasticity to density ratio (Ashby *et al.* 1995). Bamboo's high growth rate allows mature bamboo to be harvested for structural application after 3 to 5 years, whereas the fastest growing softwoods have a harvest cycle of at least 10 years (Liese 1987). To adapt to its natural environment, bamboo has developed a functionally graded material (FGM) structure in both the longitudinal and transverse directions. Its tapered geometry and wall thickness, which is thickest at its base, enables bamboo to withstand large bending loads (Ghavami 2016). Transversely, longitudinally oriented vascular bundles are smaller but denser around the outer-wall section, and larger but fewer around the inner-wall section, as shown in Fig. 1b (Amada *et al.* 1996). This hierarchical distribution of vascular

bundles effectively increases the moment of inertia of bamboo section (Ghavami 2016). However, despite its outstanding structural efficiency, bamboo is prone to natural degradation if untreated, and its durability is limited when subjected to environmental conditions such as sun and rain (Trujillo *et al.* 2012; Correal 2016).

To fully maintain its structural integrity as a reliable construction material, adequate treatments are required to control its natural degradation against insect attacks, fungal decay, and hygrothermal effects. Presently, chemical preservatives are widely used. Non-fixing-type preservatives—namely, boron salts—provide good protection against termites and fungi, but they leach out when exposed to rain (NMBA 2006). Fixed-type preservatives, such as copper chrome compounds, provide enhanced outdoor durability but at the expense of toxicity (Kaur *et al.* 2016a). For long-term durability, a sap-replacement technique is often used *via* a pressurized system to maximize the efficiency of chemical treatments (Rao 2001). Despite the challenges, there is much interest in research concerning bamboo durability. Recent studies show efforts to improve its resistance against natural degradation, dimensional stability, and crack reduction (Silviana and Petermann 2014; Kaur *et al.* 2016a; Wu *et al.* 2018; Rao *et al.* 2019).

One treatment that stands out from the rest is traditional smoke treatment, which substantially enhances bamboo durability against hygrothermal effects as well as improves its resistance against fungal decay and termite attack (Liese 2003; Kaur *et al.* 2016a). Smoke treatment is an efficient method of bamboo modification that has been used for centuries in the construction of Japanese houses and is a proven eco-friendly alternative to conventional chemical treatments. In Colombia, a similar smoking system as applied traditionally in Japan has been further developed for large-scale commercial operations. In that setup, semi-dry culms arranged inside a furnace were subjected to heat treatment at a temperature of 55 °C generated from the combustion of organic matter for a period of 15-30 days until the moisture content was reduced to about 12% (Liese 2003). In smoke treatment, the moisture content is efficiently reduced as bamboo culms undergo partial carbonization when subjected to a combustion smoke of wood and bamboo (Fig. 1a). The heat generated by smoke treatment damaged parenchyma cells and decreased both the starch and moisture content 35% and 69%, respectively (Liese 2005; Kaur *et al.* 2016b). Despite its long-term durability when protected from exterior exposure, smoked bamboo becomes susceptible to split as the treatment causes it to lose ductility and toughness.



**Fig. 1.** (a) Schematic illustration of smoke treatment in a longitudinally sliced section of bamboo culm; (b) FGM structure in a transversally sliced section of bamboo culm

Moreover, modification by various treatment methods can often engender inhomogeneous effects in the FGM structure of bamboo because of its unique morphology (Fig. 1b). For instance, the radial flow of preservatives is considerably restricted by simple

soaking methods due to an outer skin with a high silica content, an inner culm wall consisting of suberin layers, and an absence of ray cells (Janssen 2000; Liese and Kumar 2003). In submerged treatment, preservatives travel through the culm in metaxylem vessels, slowly diffusing through the surrounding varying volume fraction of parenchyma and fibres (Liese 2003, 2004). Bamboo has high hygroscopicity due to the presence of polar hydroxyl groups in its cellulose fibres (Li *et al.* 2016). An increase in moisture content (MC) below the fibre saturation point (FSP) promotes ductile behavior in bamboo (Ota 1953; Jiang *et al.* 2012; Xu *et al.* 2014). Additionally, the mechanical properties of the lignin-hemicellulose matrix are more sensitive than cellulose to changes in MC (Jiang *et al.* 2012). At high MC, cellulose fibrils tend to decouple from the weakened matrix because of the reduced stiffness in hemicellulose (Cousins 1978). In contrast, thermal effects due to smoke treatment, which occur at temperatures between 140 and 150 °C, cause permanent change to bamboo's chemical constituents, including cellulose, lignin, and hemicellulose. Notable reduction in the water absorption of lignocellulosic materials has been linked to lignin's cross-linking reaction that stems from thermal modification (Tjeerdsma *et al.* 1998; Boonstra and Tjeerdsma 2006). Thermal degradation of hemicellulose further reduces MC, as well as increases bamboo's brittleness due to an increase in relative proportion of crystalline cellulose (Tjeerdsma *et al.* 1998; Boonstra *et al.* 2007; Tang *et al.* 2019).

For the purpose of construction safety and reliability, it is essential to properly understand the alterations of bamboo's physical and mechanical properties caused by administered treatments that result in microstructural changes. The current study investigated the effect of smoke treatment on the flexural strength of Madake bamboo's (*Phyllostachys bambusoides*) hierarchical structure. The flexural strength of smoked bamboo was compared with similar bamboo species subjected to other treatments. The effect of smoke treatment on bamboo's hierarchical structure and the alterations to its chemical structure was further investigated by microscopy analysis and Fourier transform infrared (FTIR) analysis. In addition to this study's utility when treating full-culm bamboo, its results can also be applied to efficiently treat bamboo used for engineered bamboo materials.

## EXPERIMENTAL

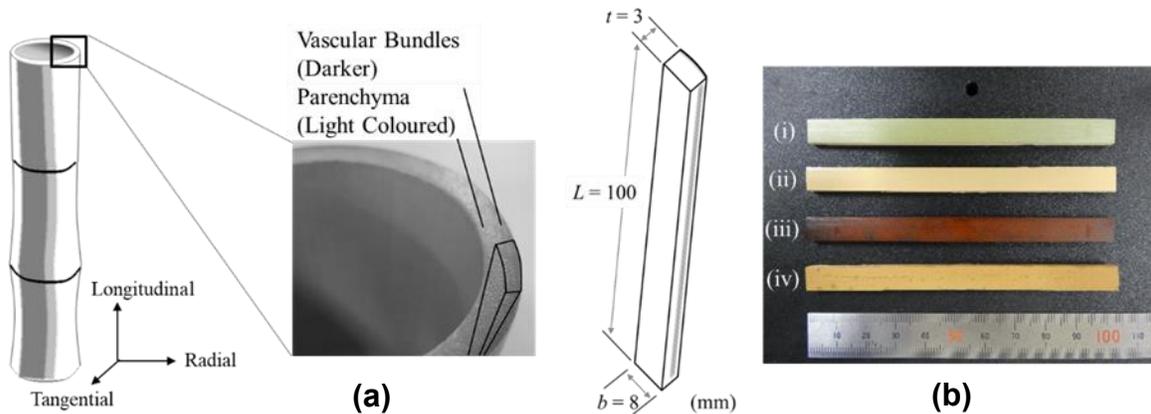
### Materials

#### *Sample preparation*

In this study, untreated samples of Madake bamboo (*Phyllostachys bambusoides*) (harvested from the Kameoka and Ohara regions in western and northern Kyoto, Japan) were used as controls. Traditionally, smoked specimens were prepared in a partial carbonization process (shown in Fig. 1a) by subjecting dried bamboo to an organically derived combustion smoke from wood and bamboo over 24 h at a temperature not exceeding 150 °C. To compare the mechanical properties of smoke-treated bamboo, two other types of modified bamboo of the same species, having alternative treatment methodologies and mediums, were prepared. These bamboos were dried and dyed. The former was prepared by natural drying in a forest for two months at autumn temperatures, and twelve months inside a factory environment, followed by a heat treatment to remove oil from its hard, waxy outer surface. Dyed bamboo was prepared from dried samples in a

submerged chemical-bath treatment involving a mixture of coloured pigments (Dianix blue E-GR and Miketon polyester yellow, DyStar, Osaka, Japan).

All four categories of bamboo specimens used in this study were procured from Yokoyama Bamboo Products & Co. in Kyoto, Japan (Fig. 2). The specimens had an average maturity of 3 years. They were selected at various positions along the culm length and were prepared from the outermost wall section inclusive of the skin layer into dimensions of 100 mm (longitudinal)  $\times$  8 mm (tangential)  $\times$  3 mm (radial). The span-to-depth ratio was no less than 20 to avoid shear stresses. Only longitudinal sections without nodes were considered (Dixon and Gibson 2014; Chen *et al.* 2020).



**Fig. 2.** (a) Schematic illustration of specimen sizing; (b) types of bamboo used in this study: (i) untreated, (ii) naturally dried, (iii) dyed, and (iv) smoked

### Physical Characterization

The density of each specimen was determined after treatment by the mass and volume method, recording the mass and measuring dimensions with a digital scale and digital calipers (Dixon and Gibson 2014). The density of specimens, which were chosen from various culms, ranged between 600 and 900 kg/m<sup>3</sup> (Fig. 3). Moisture content measured by the EXTECH MO280 pin-free moisture meter (FLIR Commercial Systems Inc., Extech, Nashua, NH, USA) after a 3-month conditioning at room temperature of 25 °C and a relative humidity below 20%, ranged between 12.4% in untreated bamboo and 4.5% in smoked bamboo (Table 1).

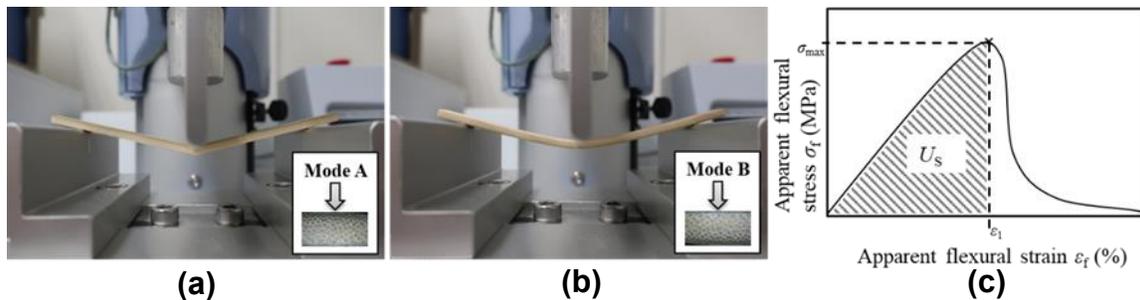


**Fig. 3.** Mean values of density variation in untreated and modified bamboo; Mode A:  $\diamond$   $n = 8$ , Mode B:  $\blacklozenge$   $n = 5$ ; error bars represent the standard deviation

## Flexural Test

In this study, the influence of treatment methods on the mechanical properties of bamboo was investigated using a bending test as employed in previous studies (Obataya *et al.* 2007; Habibi *et al.* 2015). A flexural test was conducted using a three-point bending test on the Shimadzu EZ-S (Shimadzu Corporation, Kyoto, Japan) equipped with a load cell of 500 N. The cross-head speed and distance between supports were maintained at 2 mm/min and 80 mm, respectively, throughout all tests. The supports and punch had radii of 2.5 mm.

To investigate the effect of treatment on the flexural strength of the hierarchical graded structures, two modes on bending, namely Modes A and B, were considered. Perpendicular loads were applied to the outermost and innermost layers of bamboo, as shown in Fig. 4a and 4b. In each bamboo category, the number of specimens evaluated in Mode A and Mode B were 8 and 5, respectively, making a total of 52 evaluated specimens in this study.



**Fig. 4.** Untreated bamboo specimen in a 3-point bending test (displacement,  $\delta$  of 15 mm) in (a) Mode A, and (b) Mode B; (c) calculation of specific energy absorption from the stress-strain curve

In this study, the flexural strength was assumed to be same as the modulus of rupture (MOR). Both the MOR and flexural modulus (MOE) were calculated based on an assumption of a homogeneous material with a neutral axis at the center, and hence was termed as apparent MOR and apparent MOE. The apparent average MOR, the apparent flexural strain, and the apparent MOE were determined by the following equations, respectively,

$$\sigma_f = (3FL) / (2bt^2) \quad (1)$$

$$\epsilon_f = (6\Delta t) / L^2 \quad (2)$$

$$E_f = (FL^3) / (4\Delta bt^3) \quad (3)$$

where  $\sigma_f$  is the flexural strength (MPa);  $F$  is the force capacity (N) of the bending test jig;  $\epsilon_f$  is the apparent flexural strain;  $E_f$  is the apparent flexural modulus (MPa);  $\Delta$  is the deflection of the center of beam; and  $L$ ,  $b$ , and  $t$  represent the length (m) between supports, breadth, and thickness of the specimen, respectively.

## Specific Energy Absorbed

The specific energy absorption,  $U_s$ , was calculated to further analyse the effects of treatment modifications on asymmetrical flexural behavior resulting from bamboo's hierarchical structure. The specific energy absorbed gives a measure of the energy absorbed per unit mass during deformation (Priem *et al.* 2014; Mou *et al.* 2016). It was calculated

by considering non-linear deformation, measured as the area under the stress-strain curve up to the maximum flexural strain,  $\varepsilon_1$  (Fig. 4c) as follows,

$$U_s = \frac{1}{\rho} \int_0^{\varepsilon_1} \sigma_f(\varepsilon_f) d\varepsilon_f \quad (4)$$

where  $U_s$  is the specific energy absorbed (J/kg),  $\sigma_f$  is the flexural stress (MPa), and  $\rho$  is the density (kg/m<sup>3</sup>).

### Microscopy Analysis

A scanning electron microscope (SEM) (JSM-6010LA SEM; JEOL Ltd., Tokyo, Japan) was used to observe the fractured samples of Modes A and B. A digital microscope Keyence VHX VH-Z20R (Keyence Corporation of America, Itasca, IL, USA) of lower magnification was used to observe the extent of microstructure affected by the treatment modification.

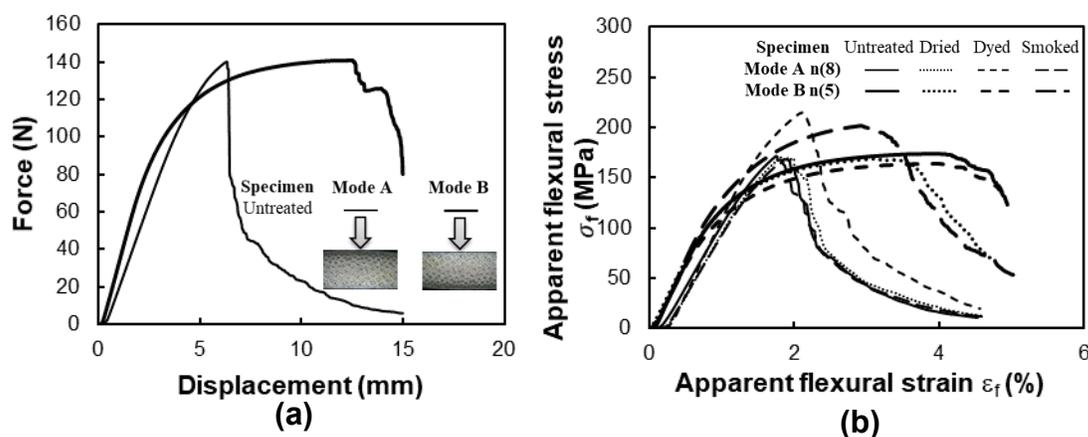
### FTIR Analysis

To assess the effect of partial carbonization on bamboo's cellular structure during the smoke treatment between 140 and 150 °C, FTIR spectroscopy was performed *via* attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR, FTIR-4700 with ATR PRO ONE equipped with a diamond prism; Jasco Co., Tokyo, Japan) with a resolution of 4 cm<sup>-1</sup> and 100 scans. Commercial software (Origin 8.5, OriginLab Co., Northampton, MA, USA, and LabSpec, Horiba/Jobin-Yvon, Kyoto, Japan) were used for spectral acquisition and to pre-process raw data by baseline subtraction, smoothing, normalization, and fitting methods.

## RESULTS AND DISCUSSION

### Flexural Results

The typical force-displacement curves of small clear specimens of untreated bamboo in 3-point bending Modes A and B are shown in Fig. 5a. In both Modes, ultimate failure occurred on the section subjected to tensile loads.

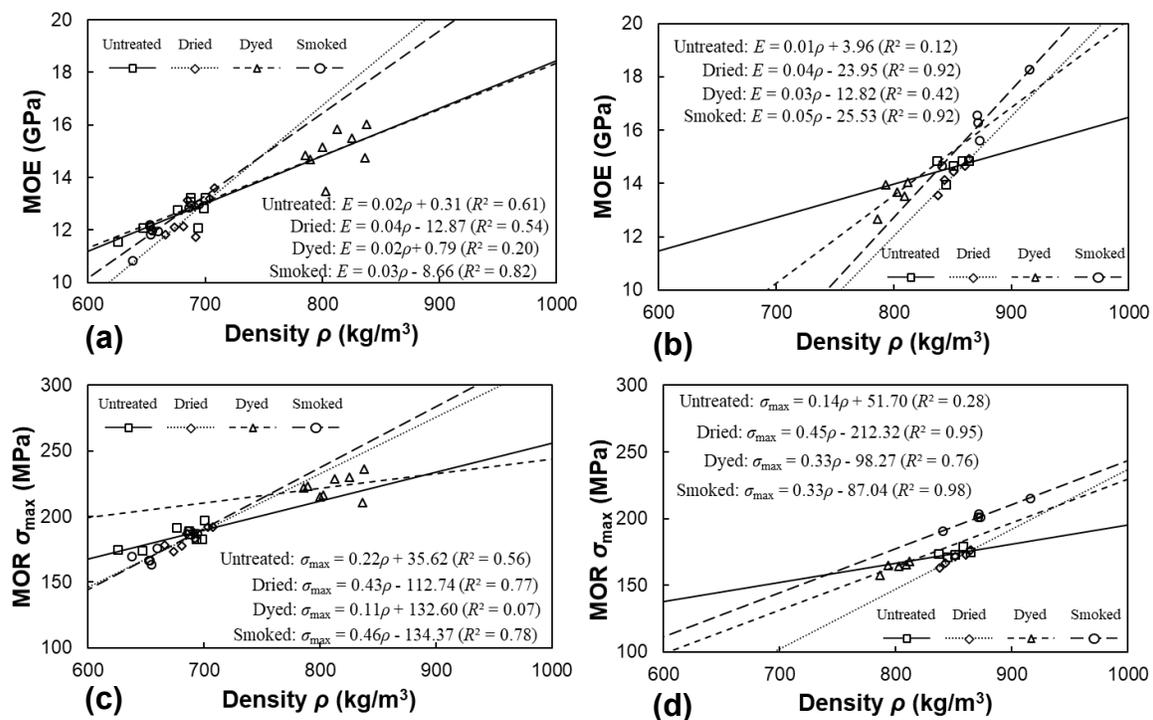


**Fig. 5.** (a) Typical force-displacement curves in 3-point bending of small clear specimens of (i) untreated bamboo ( $\rho = 625$  kg/m<sup>3</sup>) in Mode A, and (ii) untreated bamboo in Mode B ( $\rho = 837$  kg/m<sup>3</sup>); (b) arithmetic mean of multiple stress-strain curves in each batch of bamboo

Figure 5b shows the average stress-strain curves of each type of treated bamboo in Modes A and B. Each curve was computed from the arithmetic mean of multiple curves obtained from each batch of bamboo. A distinct difference in terms of the region up to fracture point, which was twice as big in Mode B than Mode A, was observed and corresponds to the observations of Chen *et al.* (2020) and Habibi *et al.* (2015). Mode B showed a larger region of non-linear deformation and delayed fracture, as the outermost section of concentrated fibres and inner softer inner tissues were, respectively, in tension and compression (Obataya *et al.* 2007).

### Correlation of Flexural Properties

Figure 6 displays the results of commonly assessed flexural parameters of MOE and MOR in relation to their density in both Modes A and B. A linear increasing trend in MOE and MOR was observed with density in most cases except for dyed specimens in Mode A (Fig. 6a and 6c) and in MOE results of untreated specimen in Mode B (Fig. 6b). The slope of trendlines of MOE and MOR results for untreated bamboo, which was steeper in Mode A than Mode B, was consistent with Chen *et al.* (2020) and Habibi *et al.* (2015). Moreover, the slope of the trendline of MOE and MOR of smoked bamboo was distinctly steeper in comparison to untreated bamboo in both bending Modes A and B.

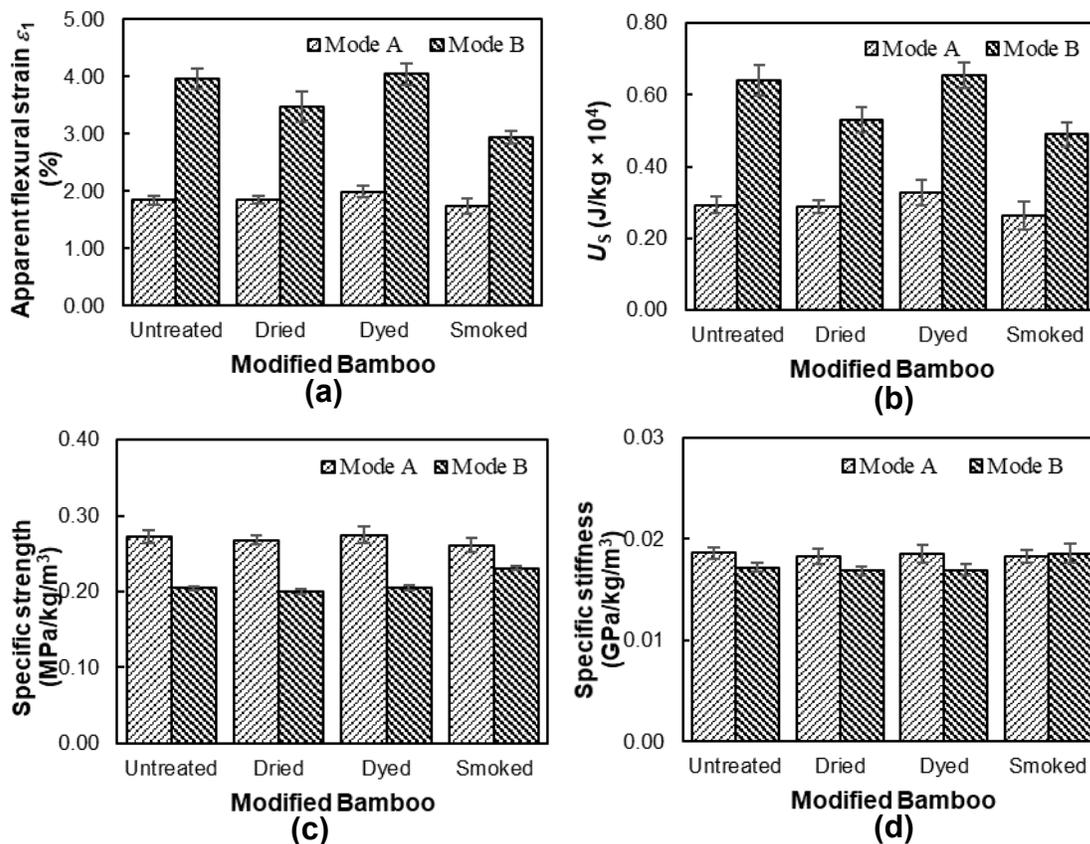


**Fig. 6.** Correlation between MOE and  $\rho$  in (a) Mode A, (b) Mode B; correlation between MOR and  $\rho$  in (c) Mode A, and (d) Mode B

As demonstrated in Figs. 7a and 7b,  $\epsilon_1$  and  $U_s$  were greater in Mode B than in Mode A, contrary to the previous observation made about MOE and MOR from Fig. 6. Additionally, the latter observations further demonstrated in Fig. 7c and 7d indicated an increase in specific strength and specific stiffness in Mode A compared to Mode B (Habibi *et al.* 2015; Chen *et al.* 2020). This increase remained true in all bamboo except for the

smoked specimen, which exhibited an increase in specific stiffness in Mode B. This overall increase in specific stiffness was due to the relatively narrow linear region of the stress-strain curve exhibited in Mode A.

From the results of Fig. 7a and 7b, parameters  $\varepsilon_1$  and  $U_s$  were influential in further discussing the changes in mechanical properties and FGM structure due to treatment modification. On this basis, both parameters were considered indicating properties (IPs). By comparing the change in  $\varepsilon_1$  and  $U_s$  from Mode A to Mode B, dried bamboo followed a similar decreasing trend compared to smoked bamboo, while dyed bamboo closely matched untreated bamboo. Moreover, the large variation observed in the mentioned parameters among Mode B specimens was attributed to the effect of smoked and dried treatments, which predominantly targeted the outermost surface at different intensities, hence impairing the flexibility of fibres in that section (Obataya *et al.* 2007).



**Fig. 7.** Comparative study of flexural properties of bamboo strips by considering Mode A and Mode B bending configurations: (a) apparent flexural strain; (b) specific energy absorbed; (c) specific strength; (d) specific modulus as a function of bamboo modification

The average MOE and MOR results of both Modes correlated well with the material property chart of modulus-specific strengths by Ashby *et al.* (1995) and indicated a performance index of material that allowed large, recoverable deformations. The detailed results of MOR, MOE,  $\varepsilon_1$ , and  $U_s$ , including their standard deviations and coefficients of variance, is summarized in Table 1.

A statistical test, illustrated in Table 2, was conducted to determine whether the differences, which prevailed between the means of evaluated parameters of treated

specimens and untreated bamboo, were statistically significant. An unpaired t-test was selected to compare the means of the independent groups of specimens. For a level of significance of 0.05, the difference in means of  $U_s$  and  $\varepsilon_1$  of both dried and smoked bamboo were considered highly statistically significant with respect to untreated bamboo in Mode B as indicated by the p-values displayed in Table 2. However, the difference in means of the formerly mentioned parameters of dyed bamboo could not be statistically distinguished with respect to untreated bamboo in Mode B. The similarity between dyed and untreated bamboo was assumed to be related to its uniform submerged treatment, which did not trigger an inhomogeneous effect on its microstructure.

**Table 1.** Flexural Test Results of Untreated and Modified Bamboo Subjected to Bending Modes A and B

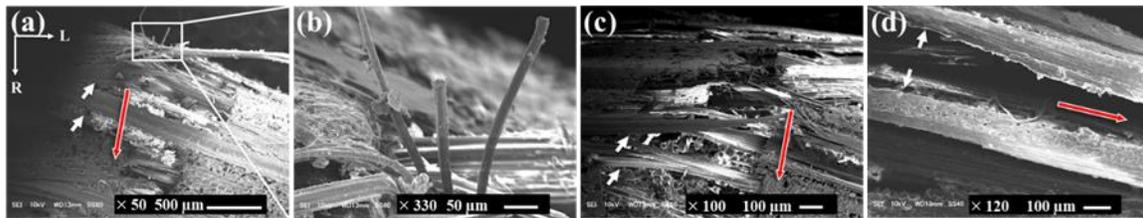
Specimen	Mode Tested	n	MC (%)	$\rho$ (kg/m <sup>3</sup> )		MOR (MPa)		MOE (GPa)		$U_s$ (J/kg × 10 <sup>4</sup> )	
				Mean	COV	Mean	COV	Mean	COV	Mean	COV
Untreated	A	8	12.4	677	0.04	185	0.04	12.6	0.05	0.292	0.08
Dried	A	8	6.0	688	0.02	184	0.04	12.6	0.06	0.288	0.06
Dyed	A	8	4.9	811	0.02	223	0.04	15.0	0.05	0.328	0.11
Smoked	A	8	4.5	661	0.03	173	0.06	12.1	0.05	0.263	0.15
Untreated	B	5	12.4	851	0.01	174	0.02	14.6	0.03	0.641	0.07
Dried	B	5	6.0	851	0.01	170	0.03	14.3	0.04	0.530	0.07
Dyed	B	5	4.9	801	0.01	164	0.02	13.6	0.04	0.655	0.05
Smoked	B	5	4.5	875	0.03	202	0.04	16.3	0.08	0.490	0.07

**Table 2.** Summary of p-values From Unpaired t-test Comparing Modified Bamboo to Untreated Bamboo in Mode B

	Untreated Specimen			Smoked Specimen			p-value
	n	Average	COV	n	Average	COV	
$U_s$ (J/kg × 10 <sup>4</sup> )	5	0.641	0.07	5	0.490	0.07	0.0003
$\varepsilon_1$ (%)	5	3.961	0.05	5	2.948	0.04	0.0001
	Dried Specimen						p-value
$U_s$ (J/kg × 10 <sup>4</sup> )				5	0.530	0.07	0.0024
$\varepsilon_1$ (%)				5	3.470	0.08	0.0096
	Dyed Specimen						p-value
$U_s$ (J/kg × 10 <sup>4</sup> )				5	0.655	0.05	0.5920
$\varepsilon_1$ (%)				5	4.042	0.04	0.4970

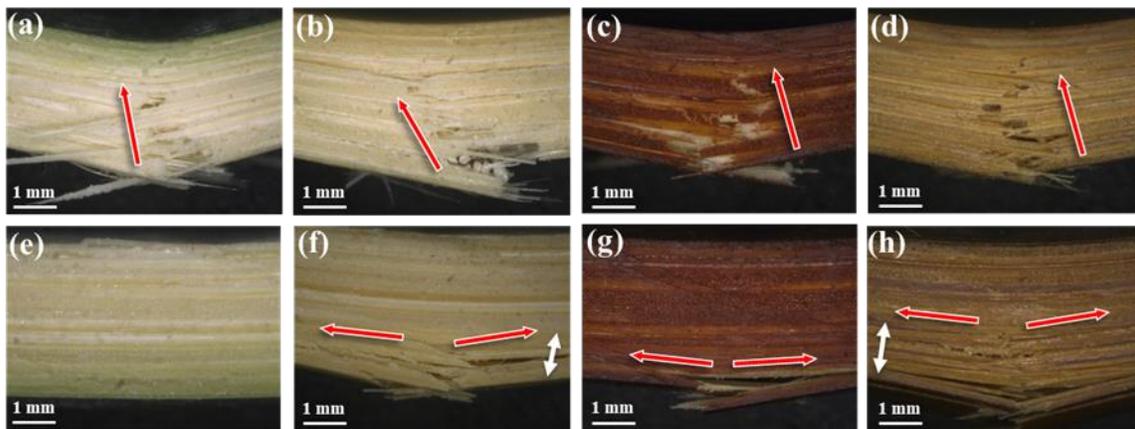
### Microscopy Analysis

In this section, microscopy results are used to explain the difference between Modes A and B and to discuss the visual changes in the microstructure following treatment modification. Figure 8 shows close-up SEM observation of the fracture modes in Modes A and B. In Mode A (Figs. 8a and 8c), radial crack propagation, which was predominant across the layers, followed a zigzag pattern, as indicated by red arrow and as reported by Song *et al.* (2017). Propagation was easier across the softer matrix component than across fibres, which led to fibre debonding. In the wake of these cracks (Fig. 8a and 8c), side debonding of parenchyma matrix is seen to occur from main fibre bundles as indicated by the white arrows. Fibre pull-out, as shown in Fig. 8a and 8b, intertwined to induce an arresting effect of transverse crack propagation.



**Fig. 8.** SEM observations of fracture modes of untreated bamboo in: (a) (b) and (c) Mode A, and (d) Mode B [magnification: (a) 500  $\mu\text{m}$ , (b) 50  $\mu\text{m}$ , (c) 100  $\mu\text{m}$ , and (d) 100  $\mu\text{m}$ ]

Conversely, in Mode B (Fig. 8d), the direction of the crack propagation differed from Mode A, and it propagated orthogonally to the direction of loading. This difference occurred due to the disproportionate volume fraction of fibres to the parenchyma matrix in the outermost layers. In bending Mode B (Fig. 9e and 9f), the parenchyma-rich section of the innermost layers prevented a large-scale buckling of fibres, given their foam-like structure that provided good ability to absorb large deformation, as evidenced by the large non-linear deformation regime prior to fracture in the stress-strain curves (Obataya *et al.* 2007; Habibi *et al.* 2015). Additionally, the morphology of the parenchyma cells, which are tightly packed near the outer section, was believed to further accentuate this asymmetric flexural behaviour (Akinbade *et al.* 2019). The difference between Modes A and B was evidenced by results of  $U_s$  and  $\varepsilon_1$ .



**Fig. 9.** Microscopy analysis of fractured surface of bamboo in: (a), (b), (c), and (d) Mode A; and (e), (f), (g), and (h) in Mode B by digital microscope Keyence VHX VH-Z20R (magnification x30)

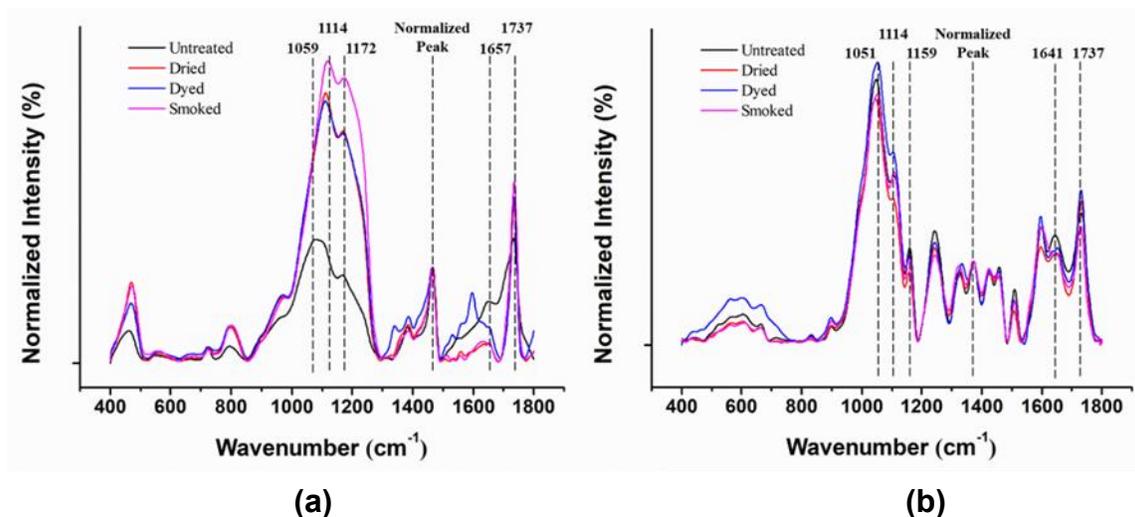
In addition to this hierarchical graded microstructure, the inferior interfacial strength between the fibres and parenchyma matrix promoted premature matrix failure, which pulled open the interface (indicated by the white arrows in Fig. 8d). Hence, this resulted in rapid crack propagation along the interface, leading to extensive delamination, which is evidenced by the major serrations in the fracture region of the stress-strain curves in Fig. 5b. The extent of radial delamination across the layers of Mode B–tested specimens was observed by a digital microscope. As indicated by the white arrows in Fig. 9f and 9h, the depth until which delamination occurred in dried and smoked bamboo accounted for approximately 30% and 40% of their respective thickness. Red arrows indicate the distinct direction of crack propagation. Both smoking and drying treatments were assumed to have

affected the capacity of bamboo's outermost layers to withstand greater tensile load in bending Mode B, as confirmed by the decrease in  $U_s$  and  $\varepsilon_1$ .

The increased toughness in Mode B has been associated with an increase in parenchyma content in the compressive region (Chen *et al.* 2020). However, as reported by Chen *et al.* (2018), the toughness of the parenchyma matrix is affected by change in moisture content. Even so, in Mode B, the increased volume fraction of fibres ( $E_f = 46$  GPa,  $\rho_f = 1160$  kg/m<sup>3</sup>) in the outermost layers contributed more notably to the stiffness of bamboo in comparison to parenchyma cells, which have lower density and stiffness ( $E_m = 2$  GPa,  $\rho_m = 670$  kg/m<sup>3</sup>) (Amada *et al.* 1996).

### FTIR Analysis

The FTIR results provided an effective means to qualitatively compare and assess the effect of heat-treated and untreated samples. The FTIR spectra in the range of 400 to 800 cm<sup>-1</sup> (seen in Fig. 10a and 10b) demonstrate the prevailing difference between the cellular structure of outer and inner layers of bamboo. Figure 10a shows a major increase in the FTIR spectra of dried, dyed, and smoked specimens from the peak at 1114 cm<sup>-1</sup>, corresponding to C–H functional group, assigned to guaiacyl and syringyl (lignin) (Meng *et al.* 2016). Unlike untreated bamboo, the outermost layers in dried bamboo was subjected to oxidation, as it was exposed to the elements of air and ultraviolet light over a one-year period of natural drying. This period was followed by a heat treatment applied to its outermost surface to remove the oil from the waxy hard layer. Because dyed and smoke-treated bamboo were derived from the dried bamboo, the trend of the peak at treatment, is, at higher temperatures, a further increase in the lignin content (Meng *et al.* 2016).



**Fig. 10.** FTIR spectra of untreated and modified bamboo in bamboo's (a) outer layer, and (b) inner layer

This increase after heat treatment was associated with the formation of new alcohols and esters linked to lignin. This change was assumed to reduce the number of free hydroxyl groups and decrease the hygroscopicity of bamboo, hence improving the dimensional stability and durability of bamboo (Meng *et al.* 2016). The reduction in hygroscopicity of smoked bamboo was further confirmed by the decrease in the peak at 1657 cm<sup>-1</sup> as observed by Huang *et al.* (2012), which represents the C=O functional group that interacts with adsorbed water at the surface. Furthermore, under oxidising conditions,

an increase in carbonyl groups in lignin occurred in smoked and dried bamboo, as indicated by the increase in the peak at  $1737\text{ cm}^{-1}$  (Hoseinzadeh *et al.* 2019). No notable changes were observed at the peaks at  $896\text{ cm}^{-1}$ , which represent the C–H bending vibration of  $\beta$ -glucosamine bond in cellulose (Wang *et al.* 2020). Cellulose decomposition was also lower than other components due to its crystalline structure (Zaman *et al.* 2000).

## Mechanics of Smoke Treatment

### *Effect of smoke treatment on mechanical properties*

In flexural tests, modified bamboo exhibited substantial asymmetrical bending behavior, resulting in an increase in its mechanical properties ( $U_s$ ,  $\varepsilon_1$ ). The mechanical properties ( $U_s$ ,  $\varepsilon_1$ ) were controlled by its hierarchical graded structure as observed between Mode A and Mode B (Fig. 7a and 7b). The effect of treatment modifications was more pronounced and distinct in Mode B specimens, which was linked to the contributions of fibres and displayed excellent load-carrying capacity in tension ( $E_f = 46\text{ GPa}$ ,  $\rho_f = 1160\text{ kg/m}^3$ ) (Amada *et al.* 1996). The evident decline in the mechanical properties of smoked bamboo ( $U_s$ ,  $\varepsilon_1$ ) is associated with its non-uniform method of treatment application that predominantly targeted the outer layers of concentrated volume fraction of fibres. The thermal effect resulting from smoke treatment affected the chemical constituents of bamboo, namely lignin, which in turn altered the physical and mechanical properties (Kaur *et al.* 2016b; Wang *et al.* 2020).

### *Effect of smoke treatment on microstructure*

As observed by SEM, the outermost layers in bamboo's graded structure plays a vital structural role given its primary composition of fibres having high tensile load-bearing capacity. In addition to their low volume fraction, parenchyma cells are tightly packed in the outer periphery of the wall section. Previous studies show the toughness of bamboo was sensitive to change in MC (Chen *et al.* 2018; Akinbade *et al.* 2019). Smoke treatment, which principally targets bamboo's outer surface, is amplified by bamboo's graded structure that is denser on the outer periphery, shown by the schematic in Fig. 1b. Consequently, the thermal effect of smoke treatment directly affects the fibre-rich areas and parenchyma cells by triggering irreversible chemical changes in the microstructure.

### *Effect of smoke treatment on chemical structure*

The FTIR analysis showed the effect of thermal changes from smoke treatment at  $150\text{ }^\circ\text{C}$  on the main chemical constituents of bamboo's outermost surface. The results confirmed an increase in lignin content after heat treatment, followed by a reduction of free hydroxyl groups, which decreased smoked bamboo's hygroscopicity (Meng *et al.* 2016; Hoseinzadeh *et al.* 2019; Wang *et al.* 2020). Above  $120\text{ }^\circ\text{C}$ , lignin undergoes structural changes. Poly-condensation reactions resulted in further cross-linking of the lignin network and led to increased lignin content, as evidenced by the FTIR results in Fig. 10a (Boonstra *et al.* 2007; Kaur *et al.* 2016b). Lignin, which typically accounts for approximately 21% of bamboo's chemical constitution, exerts an important function in consolidating matrix components (Amada *et al.* 1996). Lignin's observed increase in peripheral tissues of the outer wall was assumed to further contribute to the strength of smoked bamboo, hence supporting the observation made from Fig. 7d about the increase in MOE.

However, lignin's cross-linking reaction is assumed to further accentuate the brittleness of smoked bamboo. This reaction caused notable reduction in water absorption (Tjeerdsma *et al.* 1998; Boonstra and Tjeerdsma 2006). In thermal treatment which lasted

for a 24-hour period above 150 °C, the degradation of hemicellulose reduced bamboo's affinity to water, and led to its selective transformation into a hydrophobic network (Tjeerdsma *et al.* 1998; Boonstra *et al.* 2007; Tang *et al.* 2019). Reductions in MC have been linked to adverse effects on the ductile characteristics of bamboo (Ota 1953; Jiang *et al.* 2012; Xu *et al.* 2014; Chen *et al.* 2018). This change in cellular structure was assumed to increase smoked bamboo's brittleness characteristics, thus supporting the observation made about the reduction in  $\epsilon_1$  (Fig. 7a).

### **Future Recommendation**

As a material having good durability, further research is still required on bamboo to optimize its smoke treatment. Research investigating how to relieve excessive brittleness and reduce its propensity to cracking is needed so it may be considered as a more attractive sustainable building material.

### **CONCLUSIONS**

1. Bamboo displayed noticeable asymmetrical bending behaviour during flexural tests as a result of its hierarchical graded structure. The treatment modification effects were more pronounced and distinct when the bamboo was loaded in the less fibre-dense section. In this setup, smoked bamboo displayed a noticeable increase in modulus of elasticity (MOE) and modulus of rupture (MOR), but a noticeable decrease in specific energy absorption ( $U_s$ ) and maximum flexural strain ( $\epsilon_1$ ).
2. This change in smoked bamboo was associated with its non-uniform method of treatment application, which by targeting predominantly the outer layers of concentrated volume fraction of fibres, caused an inhomogeneous effect in the microstructure. The high tensile load-bearing capacity of the latter was affected, while the less-affected parenchyma cells in the compressive region maintained their ability to absorb large deformation.
3. Changes due to higher temperature in smoke treatment altered the chemical constituents of the outermost surface. These changes caused an increase in lignin and a decrease in free hydroxyl groups, thus enhancing the strength while reducing the hygroscopicity. In this experiment, the elevated temperature and combustion smoke derived from wood and bamboo in smoke treatment increased the dimensional stability and durability of bamboo respectively at the expense of increased brittleness characteristics.

### **ACKNOWLEDGEMENTS**

The authors gratefully acknowledge Mr. Y. Yokoyama for providing bamboo specimens and the Ceramic Physics Laboratory at the Kyoto Institute of Technology in using FTIR.

## REFERENCES CITED

- Akinbade, Y., Harries, K. A., Flower, C. V., Nettleship, I., Papadopoulos, C., and Platt, S. (2019). "Through-culm wall mechanical behaviour of bamboo," *Construction and Building Materials* 216, 485-495. DOI: 10.1016/j.conbuildmat.2019.04.214
- Amada, S., Munekata, T., Nagase, Y., Ichikawa, Y., Kirigai, A., and Zhifei, Y. (1996). "The mechanical structures of bamboo in viewpoint of functionally gradient and composites materials," *Journal of Composite Materials* 30(7), 800-818. DOI: 10.1177/002199839603000703
- Ashby, M. F., Gibson, L. J., Wegst, U., and Olive, R. (1995). "The mechanical properties of natural materials. I. Material property charts," *Proceedings of the Royal Society A* 450(1938), 123-140. DOI: 10.1098/rspa.1995.0075
- Boonstra, M. J., Acker, J. V., Kegel, E., and Stevens, M. (2007). "Optimisation of a two-stage heat treatment process: Durability aspects," *Wood Science and Technology* 41(1), 31-57. DOI: 10.1007/s00226-006-0087-4
- Boonstra, M. J., and Tjeerdsma, B. (2006). "Chemical analysis of heat treated softwoods," *Holz als Roh- und Werkstoff* 64, 204-211. DOI: 10.1007/s00107-005-0078-4
- Chen, G., Luo, H., Yang, H., Zhang, T., and Li, S. (2018). "Water effects on the deformation and fracture behaviors of the multi-scaled cellular fibrous bamboo," *Acta Biomaterialia* 65, 203-215. DOI: 10.1016/j.actbio.2017.10.005
- Chen, M., Ye, L., Li, H., Wang, G., Chen, Q., Fang, C., Dai, C., and Fei, B. (2020). "Flexural strength and ductility of moso bamboo," *Construction and Building Materials* 246, Article Number 118418. DOI: 10.1016/j.conbuildmat.2020.118418
- Correal, J. F. (2016). "Bamboo design and construction," in: *Nonconventional and Vernacular Construction Materials*, 1<sup>st</sup> Ed., K. A. Harries and B. Sharma (eds.), Woodhead Publishing, Cambridge, UK, pp. 393-431. DOI: 10.1016/B978-0-08-100038-0.00014-7
- Cousins, W. J. (1978). "Young's modulus of hemicellulose as related to moisture content," *Wood Science and Technology* 12, 161-167.
- Dixon, P. G., and Gibson, L. J. (2014). "The structure and mechanics of moso bamboo material," *Journal of the Royal Society Interface* 11, 1-12. DOI: 10.1098/rsif.2014.0321
- Ghavami, K. (2016). "Introduction to nonconventional materials and an historic retrospective of the field," in: *Nonconventional and Vernacular Construction Materials*, K. A. Harries, and B. Sharma (eds.), Woodhead Publishing, Cambridge, UK, pp. 37-61. DOI: 10.1016/B978-0-08-100038-0.00002-0
- Habibi, M. K., Samaei, A. T., Gheshlaghi, B., Lu, J., and Lu, Y. (2015). "Asymmetric flexural behavior from bamboo's functionally graded hierarchical structure: Underlying mechanisms," *Acta Biomaterialia* 16, 178-186. DOI: 10.1016/j.actbio.2015.01.038
- Hoseinzadeh, F., Zabihzadeh, S. M., and Dastoorian, F. (2019). "Creep behavior of heat treated beech wood and the relation to its chemical structure," *Construction and Building Materials* 226, 220-226. DOI: 10.1016/j.conbuildmat.2019.07.181
- Huang, X., Kocaefe, D., Kocaefe, Y., Boluk, Y., and Pichette, A. (2012). "Study of the degradation behavior of heat-treated jack pine (*Pinus banksiana*) under artificial sunlight irradiation," *Polymer Degradation and Stability* 97(7), 1197-1214. DOI: 10.1016/j.polymdegradstab.2012.03.022

- Janssen, J. J. A. (2000). *Designing and Building with Bamboo* (Technical Report 20), International Network for Bamboo and Rattan, Beijing, China.
- Jiang, Z., Wang, H., Tian, G., Liu, X., and Yu, Y. (2012). "Sensitivity of several selected mechanical properties of moso bamboo to moisture content change under the fibre saturation point," *BioResources* 7(4), 5048-5058. DOI: 10.15376/biores.7.4.5048-5058
- Kaur, P. J., Satya, S., Pant, K. K., and Naik, S. N. (2016a). "Eco-friendly preservation of bamboo species: Traditional to modern techniques," *BioResources* 11(4), 10604-10624. DOI: 10.15376/biores.11.4.10604-10624
- Kaur, P. J., Satya, S., Pant, K. K., Naik, S. N., and Kardam, V. (2016b). "Chemical characterization and decay resistance analysis of smoke treated bamboo species," *European Journal of Wood and Wood Projects* 74, 625-628. DOI: 10.1007/s00107-016-1029-y
- Li, H., Xian, Y., Deng, J., Cheng, H., Chen, F., and Wang, G. (2016). "Evaluation of water absorption and its influence on the physical-mechanical properties of bamboo-bundle laminated veneer lumber," *BioResources* 11(1), 1359-1368. DOI: 10.15376/biores.11.1.1359-1368
- Liese, W. (1987). "Research on bamboo," *Wood Science and Technology* 21(3), 189-209.
- Liese, W. (2003). "Protection of bamboo in service," *World Bamboo and Rattan* 1(1), 29-33.
- Liese, W. (2005). "Preservation of a bamboo culm in relation to its structure," *World Bamboo and Rattan* 3, 17-21.
- Liese, W., and Kumar, S. (2003). *Bamboo Preservation Compendium*, Centre for Indian Bamboo Resource and Technology, New Delhi, India.
- Meng, F., Yu, Y., Zhang, Y., Yu, W., and Gao, J. (2016). "Surface chemical composition analysis of heat-treated bamboo," *Applied Surface Science* 371, 383-390. DOI: 10.1016/j.apsusc.2016.03.015
- Mou, H. L., Zou, T. C., Feng, Z. Y., and Xie, J. (2016). "Crashworthiness analysis and evaluation of fuselage section with sub-floor composite sinusoidal specimens," *Latin American Journal of Solids and Structures* 13(6), 1186-1202. DOI: 10.1590/1679-78252446
- National Mission on Bamboo Applications (NMBA) (2006). *Preservation of Bamboo – Training Manual*, NMBA, Department of Science and Technology, Government of India, New Delhi, India, pp. 1-60.
- Obataya, E., Kitin, P., and Yamauchi, H. (2007). "Bending characteristics of bamboo (*Phyllostachys pubescens*) with respect to its fiber-foam composite structure," *Wood Science and Technology* 41, 385-400. DOI: 10.1007/s00226-007-0127-8
- Ota, M. (1953). "Studies on the properties of bamboo stem (part 9). On the relation between compressive strength parallel to grain and moisture content of bamboo splint," *Bulletin of Kyushu University of Forestry* 22, 87-108.
- Priem, C., Othman, R., Rozycki, P., and Guillon, D. (2014). "Experimental investigation of the crash energy absorption of 2.5D-braided thermoplastic composite tubes," *Composite Structures* 116, 814-826. DOI: 10.1016/j.compstruct.2014.05.037
- Rao, J., Jiang, J., Prosper, N. K., Yang, X., Liu, T., Cai, W., Wang, H., and Sun, F. (2019). "Combination of polyethylene glycol impregnation and paraffin heat treatment to protect round bamboo from cracking," *Royal Society of Science* 6(11), Article ID 190105. DOI: 10.1098/rsos.190105

- Rao, K. S. (2001). *Transfer of Technology Model: Bamboo Preservation by Sap Displacement*, International Network for Bamboo and Rattan and the Institute of Wood Science and Technology, pp. 1-23.
- Silviana, S. K., and Petermann, M. (2014). "Durability assessment and physical properties investigation of modified petung bamboo (*Dendrocalamus asper*) as resulted on acetylation, assisted by supercritical CO<sub>2</sub>," *Procedia Chemistry* 9, 273-283. DOI: 10.1016/j.proche.2014.05.034
- Song, J., Surjadi, J. U., Hu, D., and Lu, Y. (2017). "Fatigue characterization of structural bamboo materials under flexural bending," *International Journal of Fatigue* 100, Part 1, 126-135. DOI: 10.1016/j.ijfatigue.2017.03.016
- Tang, T., Zhang, B., Liu, X., Wang, W., Chen, X., and Fei, B. (2019). "Synergistic effects of tung oil and heat treatment on physicochemical properties of bamboo materials," *Scientific Reports* 9, Article Number 12824. DOI: 10.1038/s41598-019-49240-8
- Tjeerdsma, B. F., Boonstra, M., Pizzi, A., Tekely, P., and Militz, H. (1998). "Characterisation of thermally modified wood: Molecular reasons for wood performance improvement," *Holz als Roh- und Werkstoff* 56, 149-153. DOI: 10.1007/s001070050287
- Trujillo, D. J. A., Ramage, M., and Chang, W. S. (2012). "Lightly modified bamboo for structural applications," *Proceedings of the Institution of Civil Engineers – Construction Materials* 166(4), 238-247. DOI: 10.1680/coma.12.00038
- Wang, X., Cheng, D., Huang, X., Song, L., Gu, W., Liang, X., Li, Y., and Xu, B. (2020). "Effect of high-temperature saturated steam treatment on the physical, chemical, and mechanical properties of moso bamboo," *Journal of Wood Science* 66(1), 1611-4663. DOI: 10.1186/s10086-020-01899-8
- Wu, H., Yang, X., Rao, J., Zhang, Y., and Sun, F. (2018). "Improvement of bamboo properties *via in situ* construction of polyhydroxyethyl methylacrylate and polymethyl methylacrylate networks," *BioResources* 13(1), 6-14. DOI: 10.15376/biores.13.1.6-14
- Xu, Q., Harries, K., Li, X., Liu, Q., and Gottron, J. (2014). "Mechanical properties of structural bamboo following immersion in water," *Engineering Structures* 81, 230-239. DOI: 10.1016/j.engstruct.2014.09.044
- Zaman, A., Alén, R., and Kotilainen, R. (2000). "Thermal behavior of scots pine (*Pinus sylvestris*) and silver birch (*Betula pendula*) at 200-230° C," *Wood and Fiber Science* 32(2), 138-143.

Article submitted: September 11, 2020; Peer review completed: October 25, 2020;  
Revised version received and accepted: November 10, 2020; Published: November 18, 2020.

DOI: 10.15376/biores.16.1.387-402