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Mechanical, thermal and microstructural analyses of *Dendrocalamus giganteus*

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Abstract: The flexural mechanical behaviour of *Dendrocalamus giganteus* strips machined from three different regions along the culms' height was analyzed as a function of the aging time in distilled water. For the as-machined strips, the flexural properties did not vary with the culms' height. For the aged specimens the modulus of rupture decreased in relationship to the values of the unaged specimens and also decreased from the bottom to the top of the culms. The flexural modulus also decreased when unaged and aged specimens are compared, but did not vary within the same immersion time with the specimens' position along the culms. Optical microscopy and digital image processing techniques were employed to evaluate the variations of the microstructure along the culms. A mosaic technique was used to analyze the entire cross section enabling quantitative measurements of a large number of fibers. The results showed that main structural parameter, such as the number of fibers per area, was statistically identical along the culms. The shape of the fibers was, however, dependent on the position, with more elongated fibers positioned at the top of the culms. The thermal behaviour of *D. giganteus* was also determined by thermogravimetric analysis. An activation energy of 166 kJ/mol was obtained for the thermal degradation process, which occurred on a single stage, and begins at 280.1°C.

Keywords: Thermal analysis, activation energy, mechanical properties, microstructure, *Dendrocalamus giganteus*.

INTRODUCTION

Bamboo is an outstanding engineering material and in many countries it is largely used for building purposes. The use of bamboo laminates is also of great interest, since they can replace with advantage wood based laminates, and can thus decrease the pressure over natural forests due to the increasing demand for the wood based products (Anwar *et al.*, 2004). Exploitation of bamboo and its correlated products can in fact be a source of development for many underdeveloped countries, since as many products as cosmetics, cellulose and timber like products can be obtained from bamboo

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culms. Regardless to say, the fast growing rate of several bamboo species turns this material into a potential to fix atmospheric carbon, contributing to the reduction of greenhouse gases. Besides, several bamboo species can be used to recover depleted soils, both by fixing nutrients or inhibiting erosion (Sujatha *et al.*, 2004; Venkatesh *et al.*, 2005). Moreover, several species of bamboo are spread all over the world, in tropical and sub-tropical areas, and therefore, they are a widespread natural resource that can favour communities all over the world.

Although the use of bamboo as a structural material is as old as human civilization, recently its use has became attractive in many western countries where more traditional materials like steel bars for concrete, or natural materials such as timber logged from natural forests became either costly and energy consuming or environmentally banned.

Among the several species of bamboos growing at present in Brazil, *Dendrocalamus giganteus* has several advantages, including high growth rate and large culm diameter, and although being an exotic species from the Brazilian biota it is now widespread throughout the country.

However, the mechanical properties of bamboo can be affected by microorganisms and by water swelling (Wahab *et al.*, 2004; Godbole and Lakkad, 1986) which can be a disadvantage when bamboo is used as a structural material. Therefore, study was undertaken to determine the effect of humidity on the flexural mechanical properties of bamboo strips machined from the culms of *D. giganteus*. To determine if the properties measured are affected by the position of the strips along the height of the culms, specimens were taken from three different heights, and the microstructure of each of these three regions was analyzed by digital optical microscopy. Since chips from the manufacturing procedure of the bamboo strips can be used as fillers or reinforcement for polymer-matrix composites, the thermal behaviour of grinded bamboo chips was also evaluated, and the activation energy of the thermal degradation process was determined.

EXPERIMENTAL METHODS AND MATERIALS

Mechanical behaviour

Strips 300 mm long, 25.4 mm wide and 5 mm thick were machined from 3-year-old culms of *D. giganteus* using a strip and shaping machine. The flexural specimens were taken from three different heights, from the base of the culms (1.5 m long starting from above the second node), from the mid region (1.5 m long atop the base region) and from the top region (1.5 m long atop the mid region), (Fig.1). The flexural specimens were 120 mm long, the other two dimensions being unchanged in respect to the initial dimension of the strips.

The flexural mechanical properties were determined with as-machined specimens,

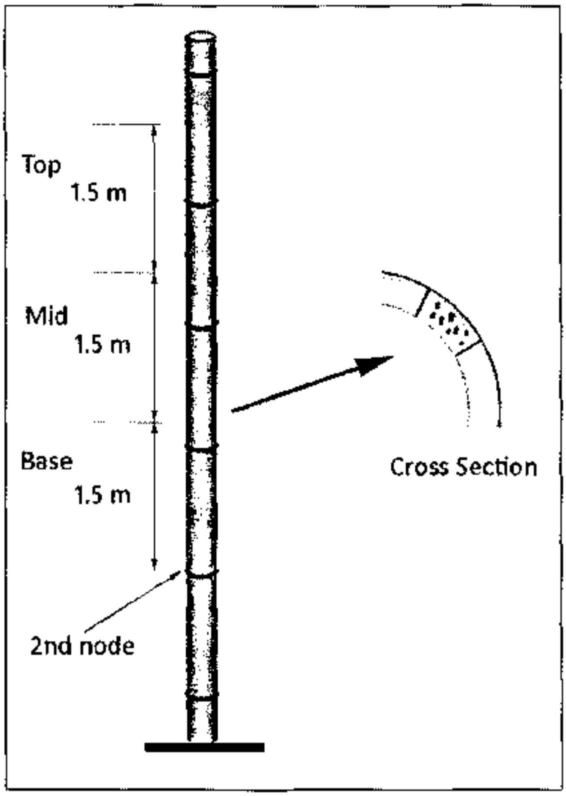

Figure 1. Distribution of the specimens along the height of the culms and orientaion of the cross-section in relationship to the specimens' length.

and also with specimens aged by immersion in distilled water. As water can readily be absorbed by wood and wood related materials, it was decided that the immersion of bamboo specimens in distilled water would act as an accelerated aging treatment. The specimens were hanged in distilled water in closed containers, at room temperature (23 ± 2°C) and were removed and tested after 10 and 54 days of immersion.

The flexural tests were conducted using a mechanically driven machine, with 10 kN capacity. A span to depth ratio of 16 was used, following the procedure recommended by the ASTM D 790 Standard. The test velocity was of 2.56 mm/min, and at least 6 specimens were tested per height or aging condition.

Microstructure characterization

Optical microscopy and digital image processing techniques were employed to evaluate differences in the microstructure along the bamboo culms. First, cross-sections (Fig. 1) from the three regions of the culms (base, mid and top) were prepared using the usual metallographic techniques of cutting, grinding and polishing to reveal their structures clearly. The samples were then analyzed by a fully automated optical microscope (Axioplan 2 Imaging, Zeiss). The control software (Axiovision 4.6, Zeiss) allows a complete scanning of sample surface through the formation of a mosaic image. The mosaic images were composed typically by 3 x 6 tiles. Each tile of the mosaic image was captured with a CCD camera (Axiocam HR) at a resolution of 1300 x 1030 pixels, using 5x objective lens. The mosaic image has a great advantage for the analysis of heterogeneous materials (Paciornik and d'Almeida, 2009) since it provides an overall view of the entire cross-section. The mosaic image was digitally processed to obtain quantitative information on the structure. A special routine Axiovision platform was developed, including pre-processing, segmentation, post-processing and measurements operations (Paciornik and Mauricio, 2004). The quantitative parameters analyzed were the number of fibers per area, their area fraction, and their aspect ratio (minor axis length/major axis length).

Thermal analysis

For the thermal analysis, bamboo strips were grinded using a laboratory cutting mill. The thermal properties of the powder obtained were studied by thermogravimetric analysis (TG) using a Perkin Elmer TGA-7 equipment. The analysis was performed from 30 to 800°C, using several heating rates, namely 5, 10, 15, 20, 30 and 40°C/min, to evaluate the activation energy of the degradation process of the bamboo. The calculations were made using the method proposed by Kissinger (1957), using the following equation:

$$\ln\left(\frac{\phi}{T_m^2}\right) = \ln\left(\frac{AR}{E}\right) - \frac{E}{R}\cdot\frac{1}{T_m} \qquad (1)$$

where ϕ is the heating rate, T_m is the peak temperature in degrees Kelvin, A is the pre-exponential constant describing a solid → solid+gas reaction of order n, R is the universal gas constant (R = 8.314 J mol^{-1} K^{-1}) and E is the activation energy. The experiments were conducted under N_2 atmosphere with a gas flow of 20 ml/min. The average mass of the samples was of 5 mg and all the results presented are the average of two measurements per heating rate.

RESULTS

A characteristic low-temperature mass loss attributed to moisture content loss, commonly observed when lignocellulosic materials are analysed was obtained (Fig. 2). The value of 8.9 per cent obtained is comparable to those found for several other lignocellulosic materials such as 10.2 per cent for jute (Das *et al.*, 2000) or 14.1 per cent for *Bambusa tulda* (Deka *et al.*, 2003). After that, the thermal decomposition occurred on a single stage, beginning at 280.1°C, T_{onset}. This temperature is in the range of onset temperatures usually reported for other bamboo species. For example, for *D. strictus* T_{onset} is close to 300°C (Rajulu *et al.*, 2005).

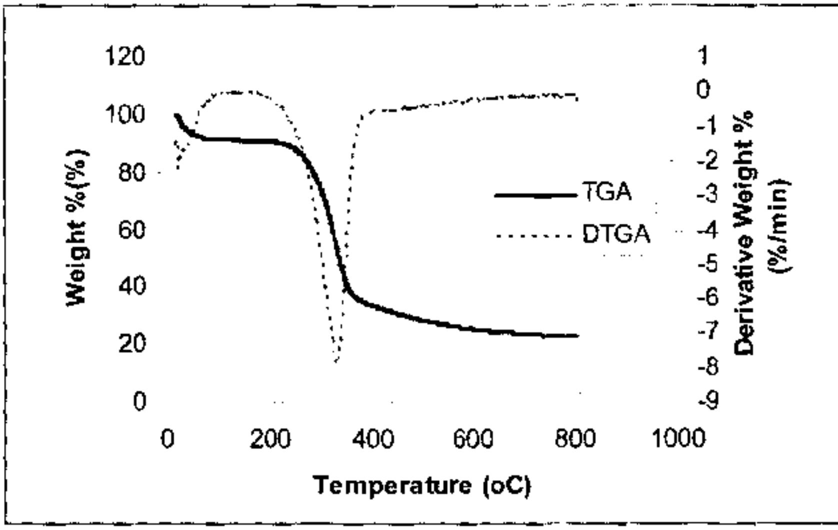

Figure 2. TG and DTG curves for *Dendrocalamus giganteus.*

The mass loss observed up to the peak temperature is common to lignocellulosic materials and lignocellulosic fibers, and can be attributed to the thermal decomposition of hemicellulose, to the rupture of ether linkages originated from the thermal degradation reactions of lignin, to the rupture of the glycoside link of the cellulose molecule, and to the decomposition of cellulose oligomers (Ramiah, 1970; Le Van, 1989). The peak temperature of 335°C is also close to the values reported for other lignocellulosic materials (Kissinger, 1957) and is close to that of pure α cellulose, 322°C (Hornsby *et al.*, 1997). Slight differences between peak temperatures from one lignocellulosic material to the other are, however, to be expected, since the amounts and interactions between the several polysaccarides present at each material vary from material to material. A char residue of 23.6 per cent at 700°C was obtained. This value correlates very well with values reported for other bamboos *Guadua angustifolia, Phyllostachys aurea* and *P. pubescens* (d'Almeida *et al.*, 2006).

Figure 3 illustrates the plot of Equation 1 using the TGA experimental data obtained from the runs made with several heating rates. A good correlation was obtained (r = 0.990), and the calculated activation energy was 166 kJ/mol. This value was similar to the values reported for other bamboo species, such as *B. tulda* (Deka *et al.*, 2003) and *D. strictus* (Rajulu *et al.*, 2005). From TGA and DTG results, it can be concluded that the thermal behaviour of *D. giganteus* is similar to those found for other bamboo species. Following the results from Rajulu *et al.*(2005) the reaction order of the thermal degradation of *D. giganteus* is also close to 1.

The results of the mechanical tests are given in Table I. For the as-machined, raw bamboo, the values did not vary as a function of the height of the culms. The box plots of both the modulus of rupture (MOR) and the elastic modulus indicate only

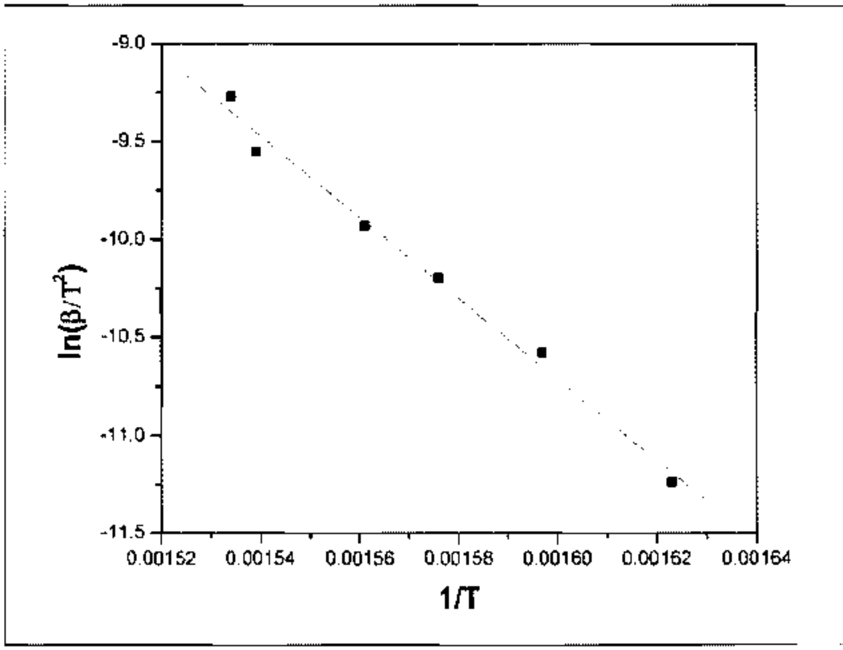

Figure 3. Plot of the experimental points using the Kissinger equation to evaluate the activation energy of the thermal decomposition process.

different maximum and minimum values, but the average values are almost the same, (Fig. 4). At the box plot, the boxes are limited by the first and third quartile and crossed by a solid line indicating the value of the median. The symbol "o" denotes the average value, and "×" the minimum and maximum values obtained. Moreover, statistical analysis using the t-Student test, at a significance level of 5 per cent also shows that the average values are not significantly different. The same behavior was found for *P. pubescens* (Li, 2004). However, for *D. strictus* an increase of both MOR and elastic modulus from the bottom to the top of the culms was observed (Ahmad, 2000). In respect to the numerical values obtained, *D. giganteus* shows a higher flexural mechanical behaviour than many common bamboo species. For example, it has a higher elastic modulus than *D. strictus*, although showing the same average MOR (Ahmad, 2000). Average values for *B. vulgaris* are comparatively lower: 60 MPa and 7 GPa for the MOR and the elastic modulus, respectively (Mohmod *et al.*, 1990). *P. pubescens* and *B. tulda* also present lower flexural mechanical properties with MOR values attaining 97 MPa and 120 MPa, respectively (Ahmad, 2000). It is worth saying,

Table 1 . Flexural mechanical properties of *Dendrocalamus giganteus*

Height	MOR (MPa)	E (GPa)
Base	156.5 ± 21.9	14.3 ± 3.0
Mid	166.8 ± 20.3	15.4 ± 1.9
Top	160.0 ± 18.5	15.0 ± 1.3

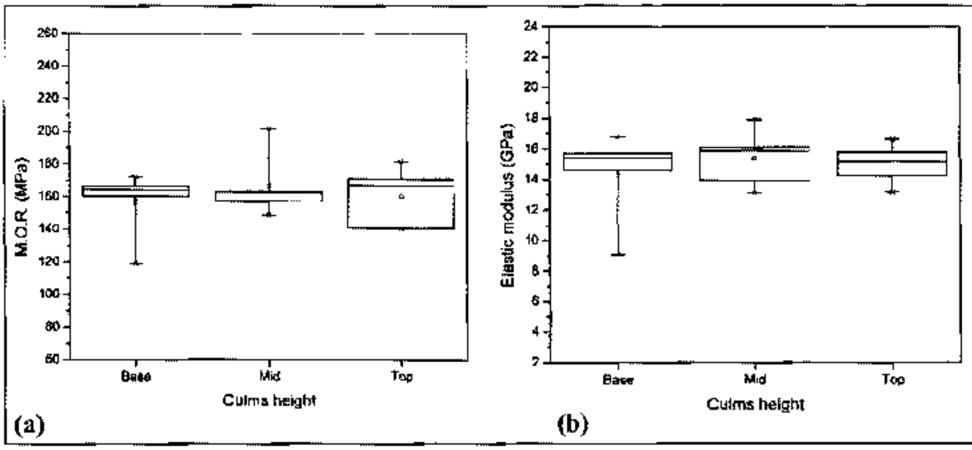

Figure 4. Raw bamboo specimens variation of : a) modulus of rupture; b) elastic modulus.

however, that the same bamboo species can present very different physical properties depending on its growing sites. For *D. strictus*, for example, MOR values ranging from 50 MPa to 153 MPa are reported to culms coming from different growing sites (Ahmad, 2000).

The constancy of the flexural properties along the height of the culms agrees with the microstructural characteristics observed. (Fig. 5). The particular mosaic shown here was taken from the base region, but the same overall microstructural characteristics were observed for all samples analyzed. Figure 6 shows the values obtained for the area fraction of the fibers and the fiber density (number of fibers per area) in each region. The values of these microstructural parameters are slightly different between the three regions, and do not show any trend along the bamboo culms height. It is worth noticing, however, that the aspect ratio of the fibers seems to vary along the height. The fiber aspect ratio for base (a), mid (b) and top (c) cross sections is given in Figure 7. There is a clear difference in the top section with respect to others. To confirm this difference, a two-population independent t-Student test, at a significance level of 5 per cent, for each pair of distribution was done (Tables 2, 3). These results confirm the difference from the top section with others, in respect to fiber aspect ratio distribution.

The flexural properties of *D. giganteus* after immersion in distilled water are given in Table 4. Figure 8 shows the box plot for the MOR as a function of the specimens'

Table 2. Fiber axis ratio: mean value, variance and number of measured fibers

Height	Mean	Variance	Number of fibers
Base	0.623	0.017	487
Mid	0.628	0.020	581
Top	0.589	0.025	599

Table 3. Two population independent t-test at the 0.05 confidence level applied on fiber axis ratio distributions of the base, mid, top cross sections

Axis ratio distributions (pairs)	t	p
Base and Mid	0.614	0.5393
Base and Top	-3.851	1.25E-4*
Mid and Top	-4.549	5.95E-6*

* Significantly different

position and the immersion time. A clear trend of the MOR to decrease from the base to the top can be observed. Moreover, comparing the results from Tables 1 and 4, a decrease of at least 20 per cent between the MOR before and after water absorption can be observed. This result can be attributed to structural modifications caused by water absorption when lignocellulosic materials are exposed to humid environments. Cellulose, the main component of lignocellulosic materials, has a large amount of

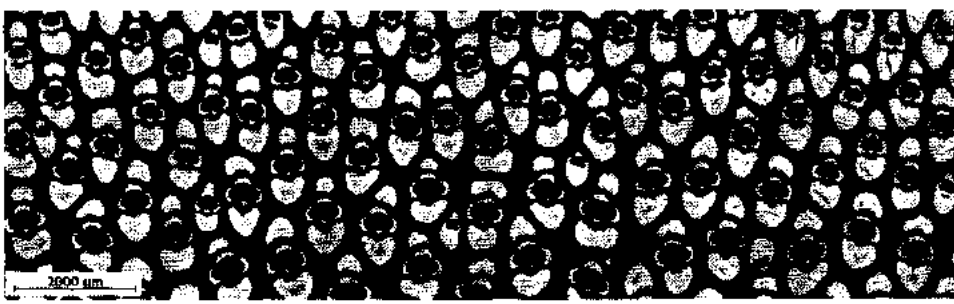

Figure 5. Mosaic image composed of 3 x 6 assembled images (base region).

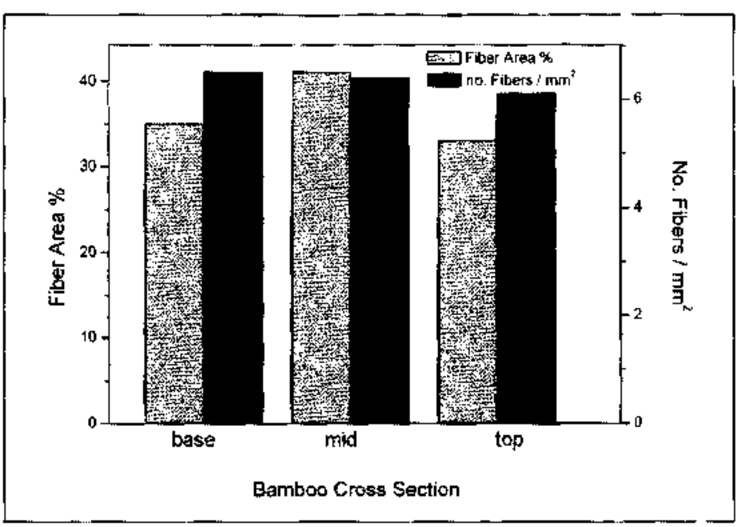

Figure 6. Percent area occupied by fibers and density of fibers (number of fibers per area) along the bamboo cross section.

Table 4. Variation of the flexural mechanical properties with the immersion time

Height	Immersion time (days)			
	10		54	
	MOR (MPa)	E (GPa)	MOR (MPa)	E (GPa)
Base	127.2 ± 28.3	11.9 ± 1.1	133.4 ± 19.9	12.6 ± 1.2
Mid	114.9 ± 26.9	10.4 ± 2.0	123.9 ± 22.3	11.8 ± 3.1
Top	101.3 ± 12.1	10.8 ± 2.4	95.3 ± 11.7	10.0 ± 2.0

hydroxyl groups, which favours both water absorption and moisture diffusion. The other two main components, hemicellulose and lignin, are also hygroscopic and partially soluble in water (Lincoln *et al.*, 2008). The high amount of absorbed water causes both swelling and plasticization that contributes to lowering the mechanical performance of lignocellulosic materials after their exposure to water absorption.

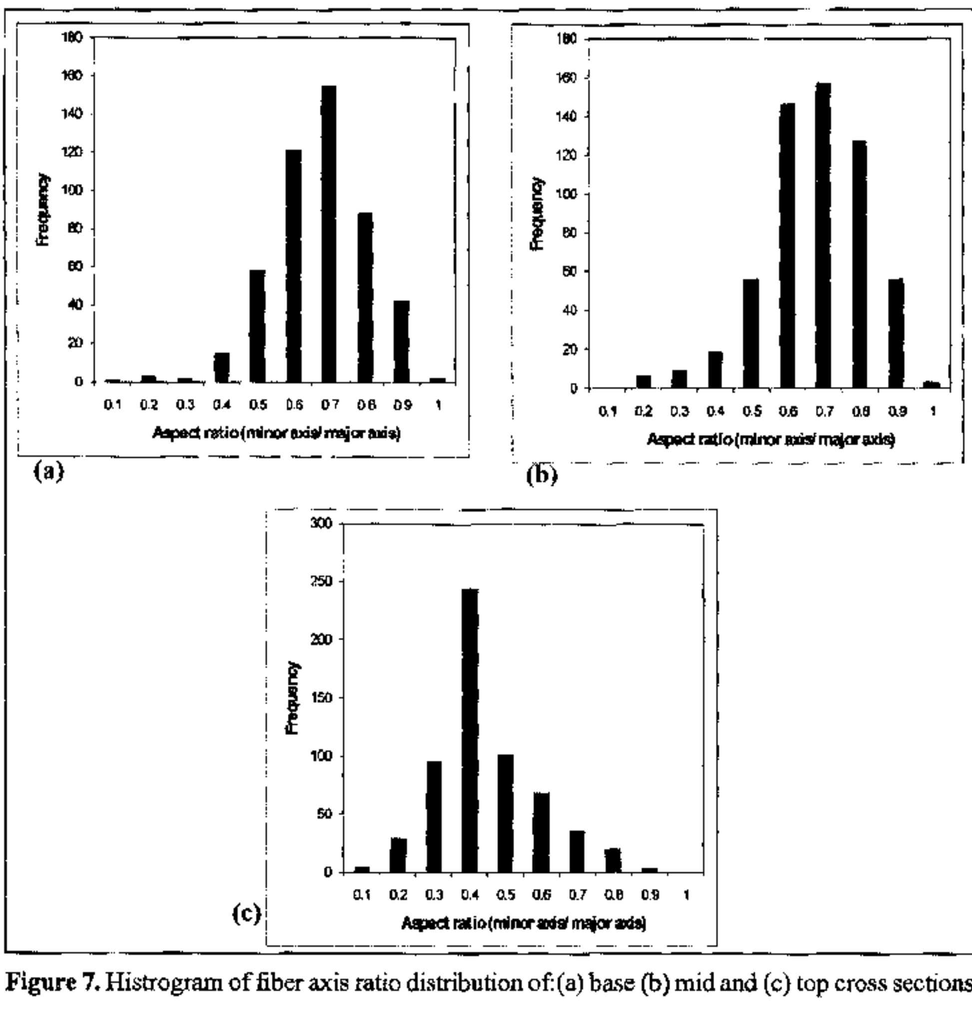

Figure 7. Histrogram of fiber axis ratio distribution of: (a) base (b) mid and (c) top cross sections.

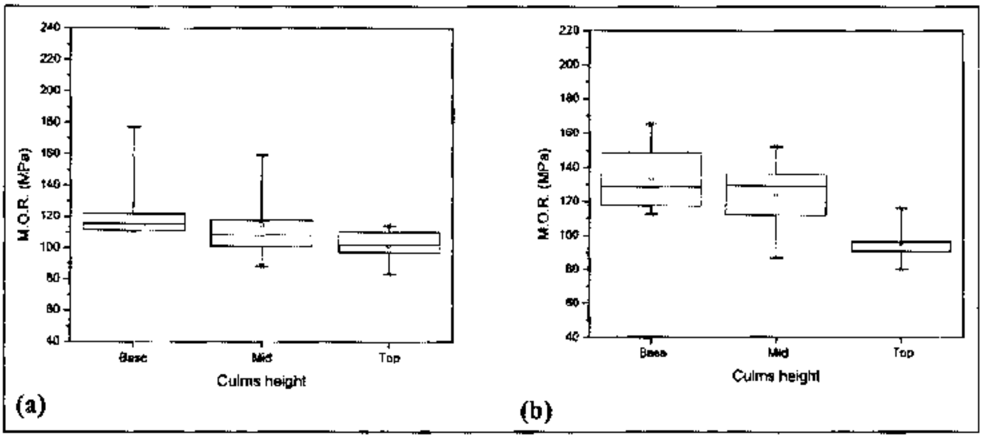

Figure 8. Variation of the modulus of rupture with the immersion time: (a) 10 days; (b) 54 days.

The variation of the MOR as a function of the position at the culms is probably due to the variation of the hemicellulose content along the culms. In fact, for *P. pubescens* it was observed that the holocellulose content increases from the bottom to the top of the culms (Li, 2004). A higher hemicellulose to cellulose ratio at the culms top can lead to higher structural changes and to a more pronounced decrease of the mechanical properties. Moreover, it was shown that hemicellulose played an important role in the bending behaviour of *P. bambusoides* submitted to moisturizing and drying cycling (Saito and Arima, 2002).

The plasticizing effect of the absorbed water is also responsible for lowering the flexural modulus with the immersion time (Tables 1, 4). However, it is to be noted that the values of the flexural modulus were not statistically affected by the position along the culms' height.

CONCLUSIONS

As expected, the thermal behaviour of *D. giganteus* is similar to those found for other bamboo species. After a low-temperature mass loss due to moisture content, the thermal decomposition occurred on a single stage, with a fairly high T_{onset} of 280.1°C. The flexural properties, MOR and flexural modulus, do not vary along the culms' height for the as-machined, unaged, material along the 4.5 m length above the second node.

Aging by immersion in distilled water causes reduction of both the MOR and the flexural modulus. This result is due to the structural modifications caused by water absorption. In fact, due to the hygroscopic nature of main bamboo components (cellulose, hemicellulose and lignin), water absorption and moisture diffusion are favoured, and both swelling and plasticization readily occur, leading to a decrease of the mechanical performance.

For the aged specimens, the observed decrease of the MOR from the bottom to the top of the culms is probably due to higher amount of hemicellulose at the top portion. Hemicellulose is partially soluble in water, and thus a more pronounced decrease of the mechanical properties is expected where the hemicellulose to cellulose ratio is greater.

The microstructural analysis showed that the main microstructural features of the bamboo cross section, such as the number of fibers per area and their area fraction, are constant along the culms height (4.5 m). Only the geometric shape of the fibers seems to vary, showing a more elongated character at the top region as verified by the aspect ratio measurements.

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