

## Conference Paper

# Microstructural Characterization of Natural Fibers: *Etilingera elatior*, *Costus comosus*, and *Heliconia bihai*

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Received 1 August 2013; Accepted 8 September 2013

Academic Editors: R. Fangueiro and H. Hong

This Conference Paper is based on a presentation given by José R. M. d'Almeida at “International Conference on Natural Fibers—Sustainable Materials for Advanced Applications 2013” held from 9 June 2013 to 11 June 2013 in Guimarães, Portugal.

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This work describes the structural and morphological characteristics of fibers obtained from the stem of three ornamental plants, namely, *Etilingera elatior*, *Costus comosus*, and *Heliconia bihai*. The stems of these plants are long and nowadays do not have any use, being disregarded. The results obtained showed that the three fibers have a crystalline index of around 58% and are thermally stable to approximately 230°C, 240°C, and 255°C for *E. elatior*, *C. comosus*, and *H. bihai*, respectively. The fibers present an average humidity amount of less than 9% and the thermal degradation peak for the cellulose component varies from 358°C for *E. elatior* to 379°C for *C. comosus*. The morphological analysis showed that the fibers present a large variability of the shape of their cross-sections, which are preferentially elongated. These morphological characteristics were used to estimate the error made when one considers the fibers having a circular cross-section.

## 1. Introduction

From very ancient times natural fibers were used in several applications, such as sacks, but starting at the middle of the XX century they began to be largely replaced by synthetic fibers. These man-made fibers present several advantages such as uniformity of properties, including the mechanical ones. However, the increasingly concern of the society with a sustainable development promoted a come back to lignocellulosic materials, and today natural fibers are replacing synthetic ones, such as glass fibers, for example, at the automotive industry [1].

In fact, lignocellulosic fibers are a very attractive option both economically and ecologically, since they are not toxic, have normally a low price, have low density, and are less abrasive to the molds and processing equipment. Besides, they consume less energy to be produced and are biodegradable and neutral with respect to CO<sub>2</sub> emission [2].

Although one can cite several advantages, as above, the use of lignocellulosic fibers also presents several disadvantages. One can highlight the hydrophilic behavior of these fibers, which can hinder the fiber to matrix adhesion, once several common polymeric matrices are hydrophobic in character. Also, the hydrophilic nature of the lignocellulosic fibers can contribute to fast humidity absorption, leading to a consequent loss of dimensional stability of the manufactured part.

Another disadvantage of using lignocellulosic fibers is the intrinsic variability of their properties, due to several causes, encompassing variables ranging from the age of the plant and its harvesting time, to soil fertility and weather variations. Besides, due to the fact that they have a natural origin, the cross-section shape and size can largely vary from fiber to fiber and also along the length of a single fiber. This is an important aspect, since it is usual to determine a typical fiber “diameter” and to assume that the fibers have a circular



FIGURE 1: Procedure used to obtain sharp cross-section. After soaking the fibers in water, the use of a doctor blade enables a cut without deformation artifacts due to the cutting procedure.

cross-section when one wants to characterize the cross-section of a lignocellulosic fiber [3]. This approach can be a crude one and can have influence on the evaluation of the mechanical properties of the fibers, since it can generate cross-section areas far apart from the real ones [4–6].

Besides the more common lignocellulosic fibers, largely studied and in many instances already used in several commercial products, such as jute (*Corchorus capsularis*), sisal (*Agave sisalana*), and flax (*Linum usitatissimum*), several other less common fibers also have a great potential to be used as reinforcement in polymer matrix composites. Less common fibers here meaning fibers that are not yet largely exploited due to being restrict to a certain ecosystem and/or region or simply because they are only obtained as a by-product of other harvests. Several examples can be cited here, such as fibers extracted from palms (piassava (*Attalea funifera*) [7] or *Borassus flabellifer* [8]) or from other plants (e.g., from *Urtica dioica* [9]). Another source of lignocellulosic fibers is the vast market of ornamental plants and flowers. In Brazil this is a growing market with a financial turnover amounting around 2.2 billion dollars per year. As many ornamental plants have stalks as long as 1.5 m and since only their top part is commercialized, the leftover residues have a great potential to be processed to obtain long or short fibers that can be used as reinforcement in polymer matrix composites.

In this work the structural and morphological characteristics of fibers obtained from three ornamental plants (*Etlingera elatior*, *Costus comosus*, and *Heliconia bihai*) are studied. The morphological aspects of the fibers were studied by both scanning electron microscopy and digital image analysis, and the structural characteristics were accessed by X-rays diffraction and thermogravimetric analysis. Special emphasis was given to the digital image analysis technique to characterize the true cross-section shape and area of these fibers and to discuss the results obtained with those calculated considering the fibers as with a circular and uniform cross-section.

## 2. Methods and Materials

The fibers used in this work came from a farm located at Rio Bonito county, RJ State, Brazil. These fibers were obtained from the leftover stems of the following ornamental plants:

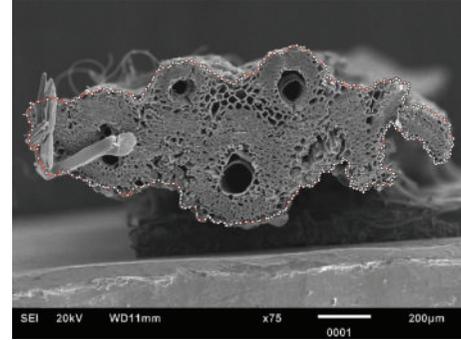


FIGURE 2: Manual outline of the fiber's perimeter. Example for a *C. comosus* fiber.

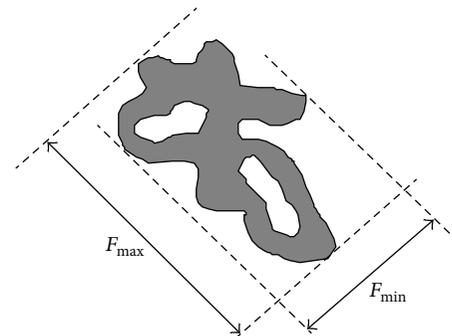


FIGURE 3: Definition of the size parameters of an object.

*Etlingera elatior*, *Costus comosus*, and *Heliconia bihai*. The common names of these plants are, respectively, torch ginger, red tower ginger, and heliconia. The fibers were obtained using a manual molasses mill. This simple device removes very efficiently the sap of the residues, has a very low energetic consume, and produces fibers as long as the processed residue. Fibers averaging about 40 mm were obtained in this work. This equipment was already successfully used to process fibers from the leftover residue of pejobaye palms [10].

These fibers were then suitably processed to the analysis to be performed. They were powdered to the thermogravimetric and X-rays diffraction analysis. The X-rays analysis was performed from  $2\theta = 5^\circ$  to  $2\theta = 80^\circ$ , with increments of  $0.02^\circ$ , using Cu- $\kappa\alpha$  radiation ( $\lambda = 1,5406 \text{ \AA}$ ). The analysis was performed using an equipment setup of 40 kV and 30 mA. The crystalline index, CI, of the fibers was calculated using the following relationship [11]:

$$CI = \left[ \frac{(I_{002} - I_{am})}{I_{002}} \right] \times 100, \quad (1)$$

where  $I_{002}$  is the intensity of (002) plane reflection and  $I_{am}$  is the intensity of the amorphous material at  $2\theta = 18^\circ$ .

The thermogravimetric analysis was performed from  $28^\circ\text{C}$  e  $750^\circ\text{C}$ , using a heating rate of  $10^\circ\text{C}/\text{min}$ ,  $\text{N}_2$  atmosphere, and a gas flow of  $20 \text{ mL}/\text{min}$ . The mass of the samples ranged from 8.67 mg to 11.79 mg.

To be analyzed at the scanning electron microscope (SEM), fibers 25 mm long were used. These fibers were first

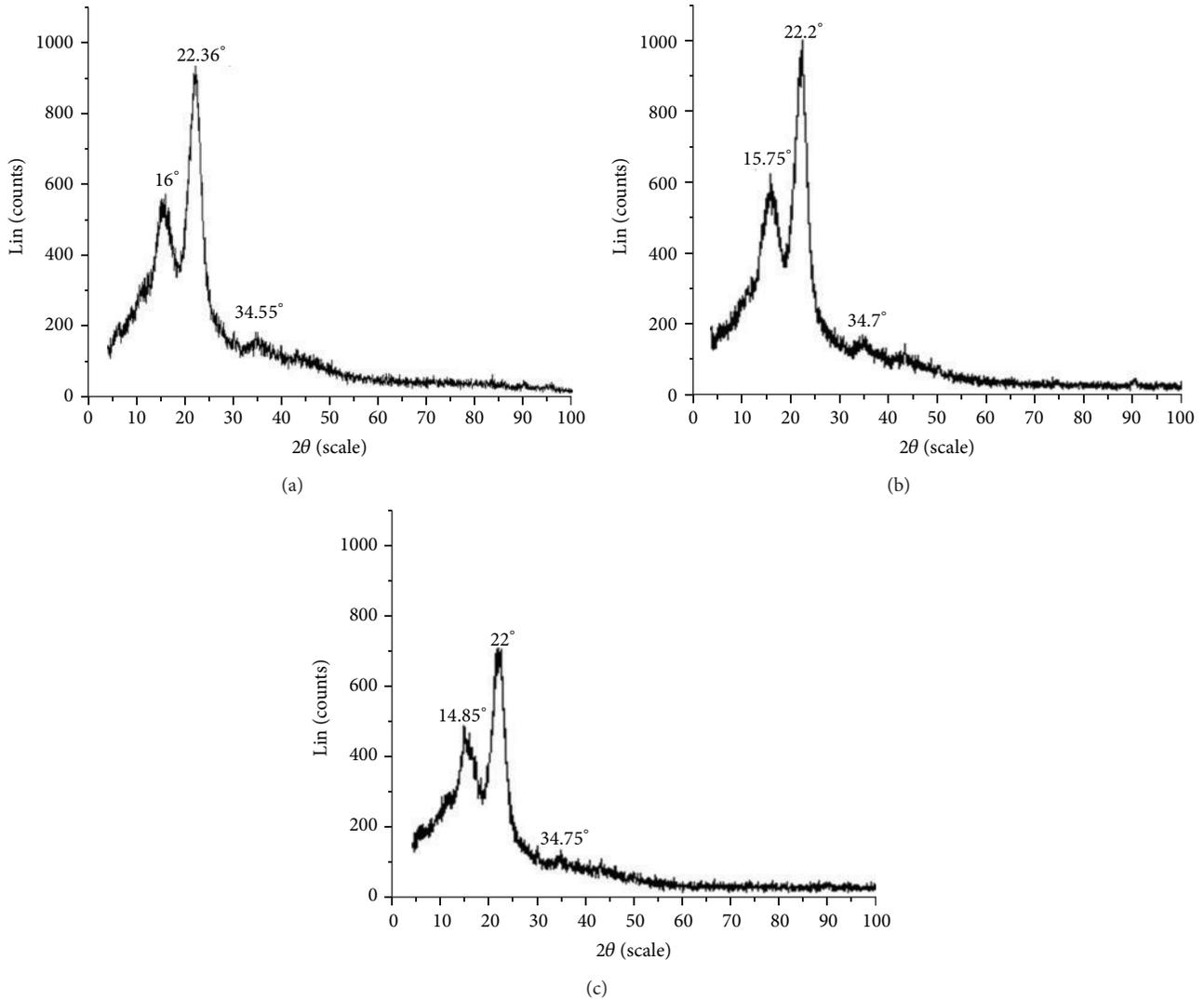


FIGURE 4: X-rays diffractogram of the fibers: (a) *E. elatior*; (b) *C. comosus*; and (c) *H. bihai*.

immersed in water for around 6 hours and were then cut using a doctor blade, as depicted in Figure 1. This simple procedure guarantees a smooth cross-section, without deformation artifacts usually observed when dry fibers are cut. The cut fibers were dried at  $70^{\circ}\text{C} \pm 5^{\circ}\text{C}$  until constant weight and were subsequently mounted vertically at a specimen holder to have their cross-section analyzed at the SEM. At this step, the fibers were individually mounted at the side of the cylindrical SEM specimen holder with a double face tape, carefully aligning them with the vertical axis of the specimen holder. This guarantees that the fibers' cross-section rests transversally to the microscope axis, since the specimen holder is perfectly aligned with this axis.

Although this was a very time-consuming step it proved to be feasible, and the cross-section of the vast majority of the fibers was at a horizontal plane perpendicular to the microscope vertical axis. Therefore, measurements errors at the fibers cross-sections geometrical parameters were minimized.

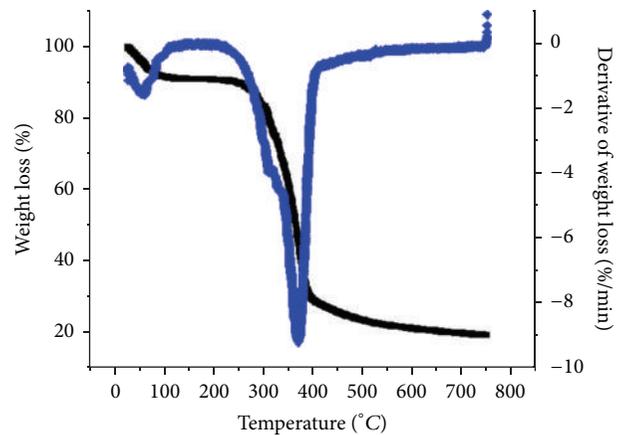


FIGURE 5: Thermogravimetric behavior of the *H. bihai* fiber. The same overall behavior was also observed for *E. elatior* and *C. comosus* fibers.

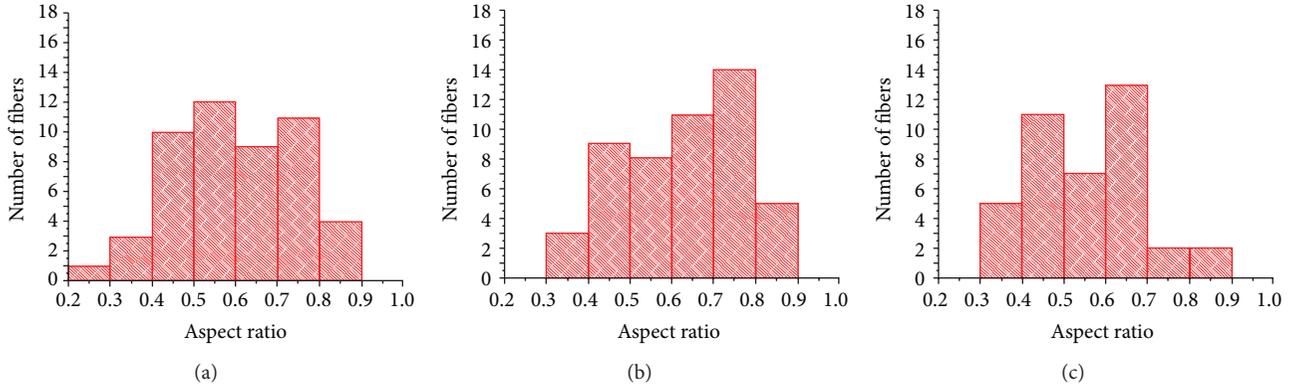


FIGURE 6: Aspect ratio histograms from (a) *E. elatior*, (b) *C. comosus*, and (c) *H. bihai* fibers.

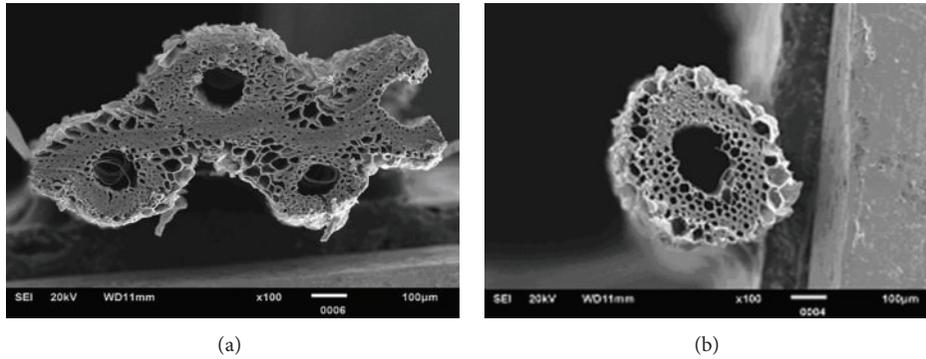


FIGURE 7: Examples of the large fibers' cross-section variability found for *C. comosus*.

SEM images were captured using a beam electron voltage of 20 kV on gold sputtered samples and at the secondary electrons imaging mode.

Digital image analysis was used to characterize the fiber cross-sections. However, due to the very irregular shape of the fibers and to the fact that their images were collected separately—one by one—it was not possible to use a completely automated analysis process, as described in a previous work [6]. Here, the first step of the digital image analysis was a manual outline of the fiber's perimeter, as shown in Figure 2. After this border was delineated all the following analyses of the fibers could be fully automated. Fifty fibers from *E. elatior* and *C. comosus* and 40 from *H. bihai* were analyzed.

The parameters used to the morphometric analysis of the fibers were the true fiber area ( $A_T$ ), obtained just by counting the image pixels contained inside the outlined perimeter; the maximum and minimum calipers ( $F_{max}$ ) and ( $F_{min}$ ), corresponding, respectively, to the longest and to the shortest projection of the fiber, Figure 3 [12]; and the aspect ratio (AR) defined as the ratio between ( $F_{max}$ ) and ( $F_{min}$ ), considered as a good measure of the cross-section elongation.

The measurement of both maximum and minimum calipers has also the objective to estimate a circular area of the cross-sections, since it is a common place to assume that the fibers have a circular cross-section when traditional measurements methods, such as when micrometers or other calipers, are used. Since the image analysis methodology

permits the direct measurement of the true area of the cross-section it is also possible to estimate the error committed when the maximum or the minimum calipers are used as representative measures of an “apparent fiber diameter.” These errors can be calculated using the following equations [6]:

$$E_{F_{max}} = \left| \frac{A_T - A_{F_{max}}}{A_T} \right| \times 100, \quad (2)$$

$$E_{F_{min}} = \left| \frac{A_T - A_{F_{min}}}{A_T} \right| \times 100,$$

where  $A_{F_{max}}$  and  $A_{F_{min}}$  are the circular areas calculated, respectively, using  $F_{max}$  or  $F_{min}$  as the fiber diameters.

### 3. Results and Discussion

Figure 4 shows the results of the X-rays analysis. The spectra of the three fibers are, as expected, similar and show the presence of the three characteristic peaks of native cellulose; namely, [13, 14] (i) the peak with the highest intensity at  $22.2^\circ$  corresponds to the diffraction of the (002) plane; (ii) a broad peak between  $14^\circ$  and  $16^\circ$  corresponds to the diffraction of

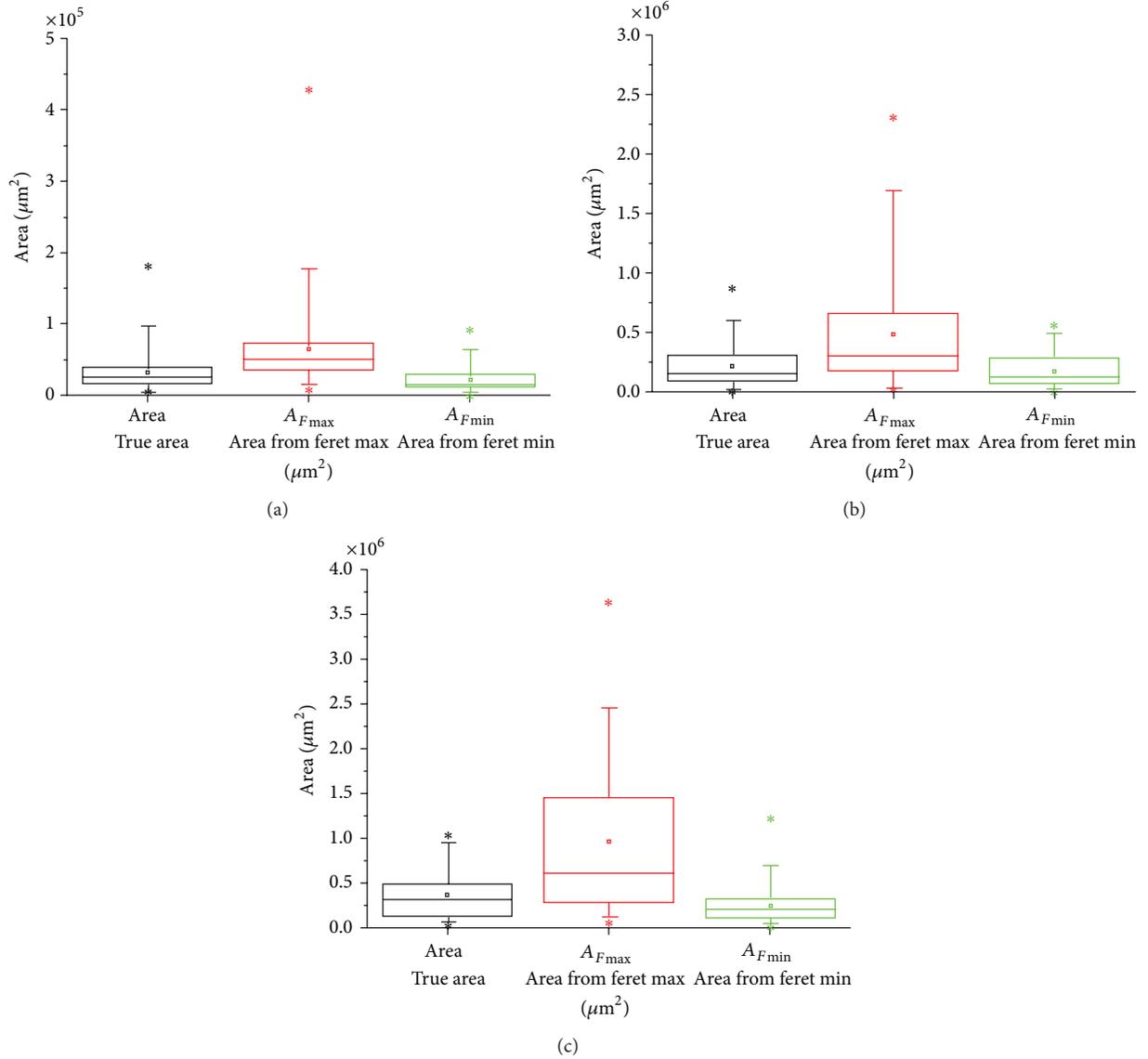


FIGURE 8: Comparison between the true fiber area directly measured by image analysis and the areas calculated assuming circular cross-sections: (a) *E. elatior*, (b) *C. comosus*, and (c) *H. bihai* fibers.

both (101) and (10 $\bar{1}$ ) planes; and (iii) the peak around 34° corresponds to the (040) plane.

The crystalline index of the fibers, evaluated from (1), showed no significant statistical difference between the fibers. The values obtained were 58.5%, 57.8%, and 58.5% for, respectively, *E. elatior*, *C. comosus* and *H. bihai* fibers. These values are similar to values reported for several other lignocellulosic fibers [15].

Figure 5 shows the thermogravimetric behavior observed for the *H. bihai* fiber, and it is also representative of the behavior of the other two fibers. At the temperature range between around 30°C and 90°C there is a mass loss attributed to loss of humidity. The values measured for the three types of fibers are listed in Table 1 and agree with the values reported for several other lignocellulosic fibers [7, 16, 17].

TABLE 1: Humidity loss and peak temperature of the cellulose decomposition of *E. elatior*, *C. comosus*, and *H. bihai* fibers.

Fiber	Humidity loss, %	Peak temperature, °C
<i>E. elatior</i>	8.9	358
<i>C. comosus</i>	7.8	379
<i>H. bihai</i>	8.8	370

The onset of the thermal degradation of the fibers, defined as the temperature where a mass loss of 1% occurs at the plateau following the humidity mass loss, began at around 230°C for *E. elatior*, 240°C for *C. comosus*, and 255°C for *H. bihai*. The subsequent mass loss is mainly attributed to the thermal

TABLE 2: Statistical comparison between the true area and the circular areas calculated using the maximum and minimum caliper values. Student's  $t$ -test between the average values.

	Area calculated using the maximum caliper	Area calculated using the minimum caliper
<i>E. elatior</i>	Yes ( $P = 1.8E - 7$ )	Yes ( $P = 2.5E - 6$ )
<i>C. comosus</i>	Yes ( $P = 9.4E - 7$ )	Yes ( $P = 4.2E - 5$ )
<i>H. bihai</i>	Yes ( $P = 7.1E - 7$ )	Yes ( $P = 2.5E - 5$ )

95% confidence interval. Yes means a statistically significant difference.

decomposition of hemicellulose, as well as to the rupture of glycoside link of the cellulose molecule and the rupture of  $\alpha$  and  $\beta$  aryl-alkyl-ether linkages originated from the thermal degradation reactions of lignin [18, 19].

The last thermal degradation step is associated to the degradation of cellulose and the value where the peak temperature of the cellulose thermal degradation occurred—obtained from the DTG curve—is also listed in Table 1. The values obtained closely agree with the values reported for the thermal degradation of cellulose from other several lignocellulosic fibers [20].

Figure 6 shows the histograms of the aspect ratio of the fibers obtained from digital image analysis results. These histograms show that the fibers' cross-section has a large variety of forms, since AR presents a large range of values. In fact, one can observe that for *E. elatior* values ranging from 0.2 to 0.9, with an average of 0.60 and a standard deviation (SD) of 0.15, were obtained. *C. comosus* fibers showed a similar trend with AR values ranging from 0.3 to 0.9, with an average of 0.63 and a SD of 0.14. *H. bihai* fibers also showed values from 0.3 to 0.9. The average value here was of 0.55 with a SD of 0.13. Figure 7 shows some cross-sections, where one can clearly observe the very different shape of the fibers. Besides, the histograms of Figure 6 show that the cross-section of the fibers is preferentially elongated, since AR values are smaller than 1. AR values approaching unity mean a more equiaxial fiber' cross-section.

In Figure 8 the graphs compare the true area, measured by image analysis, with the circular areas calculated from the minimum and maximum calipers. The Student's  $t$ -test was applied assuming a 95% confidence interval and has indicated that there exists a significant difference between the true area and the calculated circular areas for the three analyzed fibers. These results are listed in Table 2 and show that the circular area calculated both from the maximum or from the minimum caliper is statistically different from the true measured cross-section area.

The errors between the circular areas inferred using the calipers as "diameters" and the true area were calculated using (2). Therefore, for each specimen of each fiber two errors were calculated, namely,  $E_{F_{\max}}$  and  $E_{F_{\min}}$ , when the maximum caliper and the minimum caliper were used, respectively. For the population of each fiber, maximum, minimum, and average values of each error were then calculated. These values are listed in Table 3, where one can see that errors as large as 300% can be generated assuming the fibers as circular objects.

TABLE 3: Values of  $E_{F_{\max}}$  and  $E_{F_{\min}}$  for the three fibers species.

		<i>E. elatior</i>	<i>C. comosus</i>	<i>H. bihai</i>
$E_{F_{\max}}$ (%)	Maximum	273	267	337
	Minimum	28	37	48
	Average	108	116	142
$E_{F_{\min}}$ (%)	Maximum	69	59	61
	Minimum	2	5	2
	Average	31	23	31

## 4. Conclusions

The thermal and structural characteristics of fibers obtained from the stems of three ornamental plants were analyzed in this work. These stems are, nowadays, considered just as waste at the economic branch represented by the flower and ornamental plants market. However, the obtained results indicate that these fibers have thermal stability and crystalline index similar to the values found for other lignocellulosic fibers already used as reinforcement in polymer matrix composites. Therefore, from their structural characteristics, these fibers are possible candidates to be used as reinforcement in composites, for example, at interior door panels.

The fibers' cross-section morphology was also analyzed, and the results indicate that the values obtained by digital image analysis can be strongly different from the ones obtained when the fibers' cross-sections are considered as circular. Determination of the aspect ratio showed that the fibers have, in fact, an elongated shape, meaning that the circular cross-section hypothesis can be a crude approximation. The image analysis approach was shown to be an important tool, since its use significantly increases the accuracy of the measurements made on each fiber, and also enhanced the statistical significance of the morphometric parameters measured, since the number of fibers analyzed can be increased without a considerable increase on the analysis time requested. This is a key point about digital image analysis, since using usual techniques involves time-consuming steps, and the number of parameters measured and/or the number of objects analyzed is usually low.

## Conflict of Interests

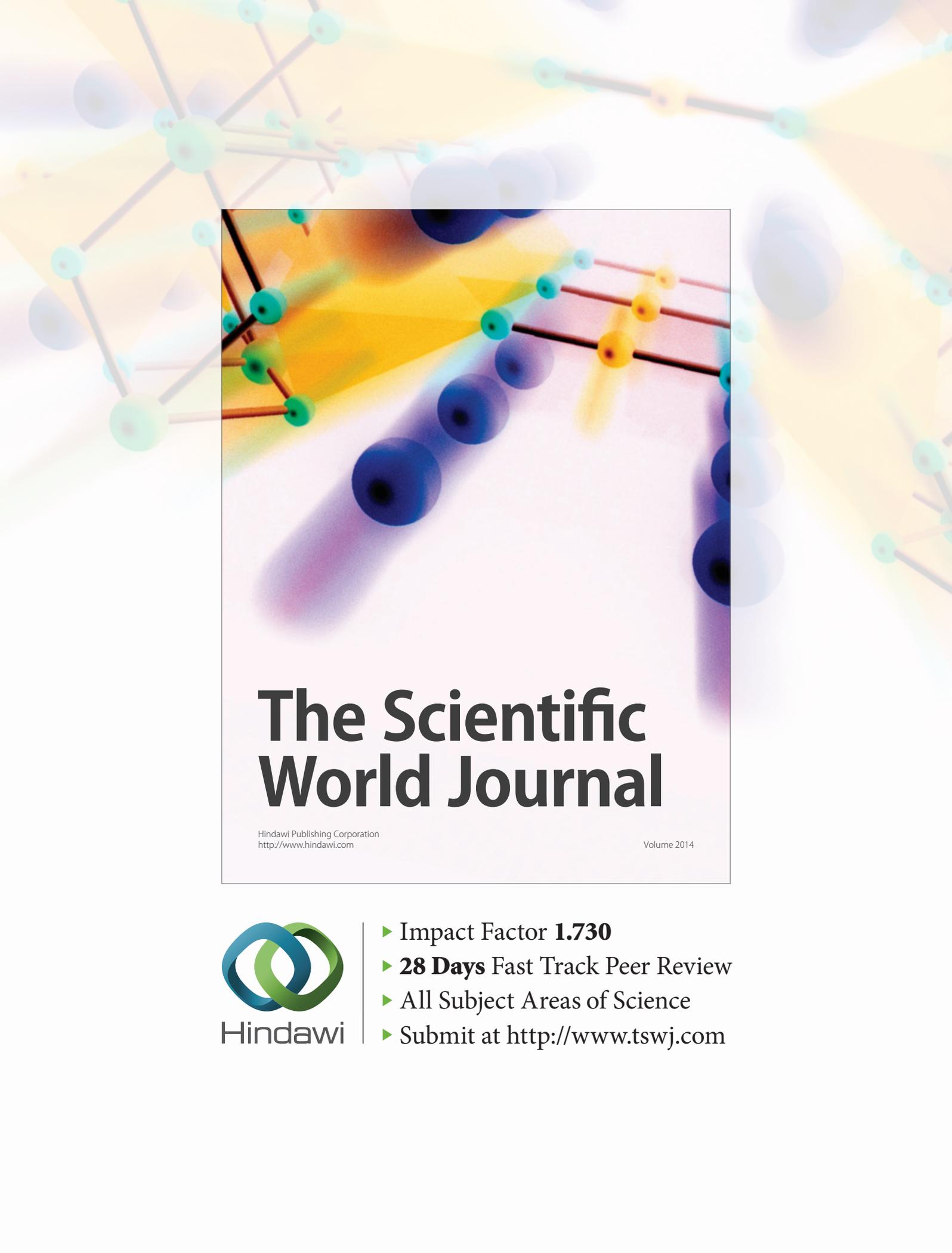
The authors declare that there is no conflict of interests regarding the publication of this paper.

## Acknowledgment

The authors acknowledge the financial support from the Brazilian Funding Agency CNPq.

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