Charpy Impact Tests of Epoxy Composites Reinforced with Giant Bamboo Fibers

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The giant bamboo fiber is among the strongest in the *Bambusa* species with a potential for application as engineering material. Its properties have been evaluated but there is limited information on the impact resistance of epoxy composites incorporated with giant bamboo fibers. Therefore, this study evaluated the Charpy impact energy of epoxy matrix composites reinforced with up to 30 vol% of giant bamboo fibers. Specimens with Charpy configuration were press-molded with continuous and aligned giant bamboo fibers reinforcing a DGEBA-TETA epoxy as the composite matrix. The energy absorbed by the composites was obtained in standard impact tests and the fracture surface of ruptured specimens was analyzed by scanning electron microscopy, SEM. The impact energy was found to increase exponentially with the amount of incorporated fiber. SEM observations revealed the mechanism of crack propagation both in the brittle epoxy matrix and in the fiber interface of the composites.

Keywords: Charpy test, epoxy composite, giant bamboo fiber, fracture analysis

1. Introduction

Owing to the growing concern about the environmental degradation associated with industrial activities, our society is increasingly using biodegradable and renewable natural materials. In this view, cellulose-based natural fibers, known as lignocellulosic fibers, become a promising solution. Currently, these fibers are being considered as possible substitute for synthetic fibers, mainly the glass fiber¹⁻³, which has since last century been used in large industrial scale but also contributing to pollution. In addition, the engineering application of lignocellulosic fibers is motivated by several advantages like low density, superior toughness and less wear of equipment used in the processing of composites4. Moreover, lignocellulosic fibers are environmentally friendly because of their characteristic of being neutral with respect to CO₂ emissions, the main responsible for global warming and climate changes5.

The interest in engineering applications of lignocellulosic fibers as polymer composite reinforcement is translated into numerous published papers in the past decades. Review articles⁶⁻¹⁹ have contributed to disseminate and discuss investigations concerning the composites and their lignocellulosic fibers. Among them, the bamboo fiber has a potential for reinforcing polymer composites to be used in structural applications. Bamboo is a well-known grass-type plant, with a hard and stiff culm that can reach, in some species, more than 10 cm in cross section diameter and stand several meters height. Owing to its low density, of approximately 0.9 g/cm³,

bamboo culms have been used in building construction from scaffoldings to house furniture. One of the limitations of bamboo culm for direct use in engineering systems is its cylindrical shape. Therefore, bamboo fibers stripped off from the culm have been investigated as reinforcement of polymer composite²⁰⁻³¹. The common bamboo (Bambusa vulgaris) fiber is reported to present4 tensile strength of 106-204 MPa and density of 1.03-1.21 g/cm³. Furthermore, according to Thwe & Liao²⁵, bamboo fiber-epoxy laminates can be made into specific sizes and shapes, preserving the natural microstructural properties. In fact, the use of the fibers composites can overcome constraints of the culm's cylindrical macrostructure. As a further advantage, the authors indicated that cracking and bioerosion caused by insect pests are prevented. One species of giant bamboo, Dendrocalmus giganteous, has recently attracted attention for its mechanical properties^{32,33}. Stripped off fiber of giant bamboo were found to present tensile strength in the range of 236-411 MPa³² and elastic modulus of 5.3-21.6 GPa³³. In particular, the giant bamboo fiber strength is significantly higher than that of Bambusa vulgaris⁴. Polymer composites reinforced with giant bamboo fibers have also been investigated for their properties³⁴⁻³⁶, including the Izod impact resistance of polyester matrix composites³⁶. However, a complete characterization of the impact behavior also requires Charpy impact tests of other polymeric matrices. Therefore the objective of this work was to evaluate the Charpy impact toughness of epoxy matrix composites reinforced with continuous and aligned giant bamboo fibers by means of impact tests.

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2. Experimental Procedure

The precursor material used in this work was the culm of giant bamboo ($Dendrocalmus\ giganteous$) kindly supplied by Prof. Khosrow Ghavami from Pontifical Catholic University of Rio de Janeiro, PUC-Rio, Brazil. Large bamboo bushes, Figure 1a, are cultivated in the Campus of PUC-Rio. Fibers were manually stripped off from dried culms, Figure 1b, with a sharp razor blade. The longitudinal direction of the fiber coincides with that of the culm and corresponds to the natural direction of the bamboo cellulose fibrils. The as-stripped bamboo fibers were dried in a laboratory stove Pro-Lab, with oven dimensions $68 \times 57 \times 53 \text{ cm}^3$, at $60 \,^{\circ}\text{C}$ for 24 hours to remove the natural moisture.





Figure 1. Bamboo trees (a) and its fibers manually stripped off from dried culms (b).

Figure 2 presents the histogram corresponding to the diameter distribution of the as-stripped giant bamboo fibers. The equivalent diameter of each fiber was actually the average value obtained by 10 different measurements performed in a profile projector at five distinct locations (two with 90° rotation at each location). This histogram discloses a relatively large dispersion in the diameter (0.1 to 0.7 mm), which is a consequence of the non-uniform cut procedure and physical characteristics of all lignocellulosic fiber⁴. It should be noticed that the giant bamboo fiber diameter range displays an average of 0.40 mm. Based on the histogram of Figure 2, the tensile strength of the giant bamboo fibers was measured for each interval of diameter in 20 selected fibers using the Weibull statistical analysis. As aforementioned, strength values from 236 to 411 MPa were found³² for the largest and thinnest giant bamboo fibers, respectively, with a mean proportional value of 262 MPa for a homogeneous mixture of fibers.

Composites with up to 30% in volume of giant bamboo fibers were fabricated by placing the mixture of fibers longitudinally aligned inside a steel mold and then pouring the still fluid diglycidyl ether of the bisphenol-A (DGEBA) epoxy resin in stoichiometric proportion, phr = 13, with triethylene tetramine (TETA) hardener into the mold. A pressure of 20 MPa was applied to the mold during the cure of the composite at room temperature (RT) for 24 hours. Standard specimens for Charpy impact test, with $125 \times 12.7 \times 10 \text{ mm}^3$, were prepared according to the ASTM D6110-10 norm³⁷ with giant bamboo fibers aligned along the length¹⁷.

Figure 3 illustrates (a) the Charpy impact pendulum and (b) a schematic Charpy specimen with standard ASTM dimensions. The notch, with 2.54 mm in depth as well as an angle of 45° and a tip curvature radius of 0.25 mm, was machined with a special milling tool. For each volume fraction of giant bamboo fiber, 10 specimens were machined to assure a statistical validation and then impact tested in the PANTEC hammer pendulum, shown in Figure 3a.

The impact fracture surface of the specimens was gold sputtered and analyzed by scanning electron microscopy, SEM, in a model SSX-500 Shimadzu microscope with secondary electrons imaging at an accelerating voltage of 15 kV.

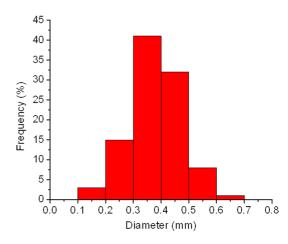


Figure 2. Histogram of the distribution of diameter of the stripped giant bamboo fibers.

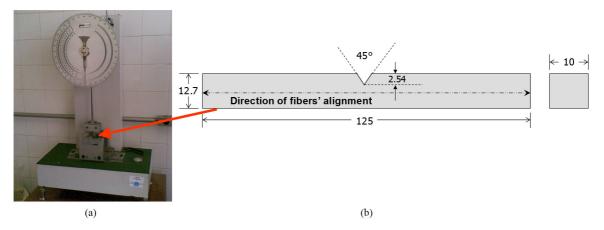


Figure 3. Charpy equipment (a) and standard specimen schematic (b) with dimensions in mm.

3. Results and Discussion

Figure 4 shows the variation of the Charpy impact energy with the volume fraction of continuous and aligned giant bamboo fibers reinforcing epoxy matrix composites. In this figure, one should notice the significant increase in impact energy with incorporation of up to 30 vol% of giant bamboo fibers. An exponential mathematical adjustment was applied to fit the points and also to define the boundaries corresponding to the limits of standard deviation associated with the error bars. The increasing dispersion of the values in Figure 4, given by the error bars, is due to the heterogeneous characteristics of the stripped giant bamboo fibers shown in the histogram of Figure 2. Since no selection was previously conducted on the fibers used to fabricate the composites, the mixture of distinct diameters implies in dispersion on mechanical behavior. This dispersion becomes more accentuated the greater the volume fraction of fibers, as seen in Figure 4. Results of Izod impact tests in polyester matrix composites reinforced with similar continuous and aligned giant bamboo fibers³⁶, showed comparable values as in Figure 4. However, the increase in impact energy followed a linear tendency in the case of Izod polyester composites³⁶ rather than the exponential increase of Charpy for epoxy composites in the present work. It is worth mentioning that not only polyester and epoxy matrices have different impact properties but also Izod and Charpy tests have distinct configurations that could justify these differences.

In previous works on Charpy impact test of epoxy matrix composites reinforced with other lignocellulosic fibers³⁸⁻⁴⁵, an exponential increase in impact energy was also observed. Table 1 compares Charpy impact energy results of epoxy matrix composites reinforced with different lignocellulosic fibers. In this table it is observed that the giant bamboo fiber reinforced epoxy composites presents the lowest value of Charpy impact energy. Although these values in Table 1 correspond to the maximum fraction of 30%, smaller fractions also follow the same trend. The reason for giant bamboo composites to have a relatively lower Charpy toughness might be due to several reasons. One important point is the fact that, different from the other fibers, the bamboo was manually extracted by cutting the hard culm

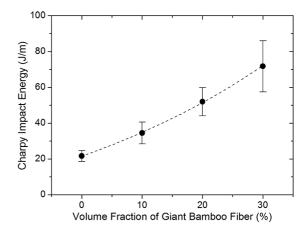


Figure 4. Charpy impact energy as a function of the amount of giant bamboo fibers.

with a razor blade. This certainly introduces comparatively more defects in the microfibrils that constitute each fiber. The other lignocellulosic fibers³⁸⁻⁴⁵ are extracted from the corresponding plant by softer natural processes such as retting (water immersion) and drying. In these separation processes, the fiber structure remains practically intact¹⁷. The fracture resulting from the Charpy test is another point, which might differentiate the impact toughness of the giant bamboo fiber composites from the other lignocellulosic fiber ones, as further discussed in the present work.

Table 2 indicates the exponential equations corresponding to the mathematical adjustment of the Charpy impact energy (E_i) as a function of the volume fraction (V) for each lignocellulosic fiber reinforced epoxy matrix composites presented in Table 1. As shown in Table 2, the Charpy impact energy dependence with the volume fraction of previously investigated lignocellulosic fiber epoxy composites can be adjusted to exponential equations with good statistical precision (R²). In this table, it is worth noticing that the softly extracted fibers display higher toughness tendency, for a predicted 50% reinforcement, than the manually cut giant bamboo fiber composites. In any case, the values in Tables 1 and 2

| Lignocellulosic fiber reinforcing epoxy composites | Volume Fraction (%) | Impact Energy (J/m) | Reference |
|--|------------------------|------------------------|--------------|
| Giant Bamboo | 30 | 72 ± 14 | Present work |
| Curaua | 30 | 139 ± 38 | 38 |
| Coir | 30 | 241 ± 45 | 39 |
| Piassava | 30 | 302 ± 90 | 40 |
| Ramie | 30 | 212 ± 24 | 41 |
| Jute | 30 | 197 ± 59 | 42 |
| Malva | 30 | 310 ± 98 | 43 |
| Buriti | 30 | 128 ± 14 | 44 |
| Sisal | 30 | 336 ± 35 | 45 |

Table 1. Charpy impact energy of epoxy matrix composites reinforced with different lignocellulosic fibers.

Table 2. Exponential adjustment for the variation of Charpy impact energy (E_i) and the volume fraction of lignocellulosic fibers.

| Lignocellulosic Fiber Reinforcing Epoxy Composites | $E_{_i}(J/m)$ and V (%) [R^2] | Predicted Charpy Impact Energy for 50% Fiber (J/m) | |
|---|--|--|--|
| Giant Bamboo | $E_i = 53.69 \exp(0.022V) - 32.11 (R^2 = 0.999)$ | 129 | |
| Curaua | $E_1 = 91.96 \exp(0.028V) - 77.60 (R^2 = 0.992)$ | 295 | |
| Coir | $E_1 = 57.55 \exp(0.053 \text{V}) - 44.30 \text{ (R}^2 = 0.999)$ | 238 | |
| Buriti | $E_1 = 99.71 \exp(0.025V) - 85.77 (R^2 = 0.999)$ | 262 | |
| Ramie | $E_1 = 35.80 \exp(0.063 \text{V}) - 22.92 (\text{R}^2 = 0.999)$ | 813 | |
| Jute | $E_i = 163.97 \exp(0.022V) - 120.13 (R^2 = 0.974)$ | 372 | |
| Malva | $E_i = 177.33 \exp(0.032V) - 150.96 (R^2 = 0.990)$ | 727 | |
| Piassava | $E_i = 28.18 \exp(0.080V) - 11.08 (R^2 = 0.998)$ | 1527 | |
| Sisal | $E_i = 41.63 \exp(0.070 \text{V}) - 9.62 (\text{R}^2 = 0.970)$ | 1369 | |

for continuous and aligned fibers are significantly higher than those reported for Charpy and Izod results for chopped and randomly distributed lignocellulosic fiber reinforcing other polymeric matrices⁴⁶.

Figure 5 illustrates the typical macroscopic rupture aspect of Charpy specimens of epoxy composites reinforced with up to 30 vol% of giant bamboo fibers. In this figure, rupture is predominantly transversal to the specimen length and nucleates, as expected, at the notch. In the case of the pure epoxy (0%) a flat fracture surface is observed, while for the composites (10, 20 and 30%) fibers are seen sticking out of the broken surface. This indicates that the crack nucleated at the notch, upon the Charpy hammer impact, propagates across the brittle epoxy but is arrested at the fibers interface. The original crack either changes its trajectory or nucleates new longitudinal cracks in between the fiber/matrix interface. As a consequence, owing to the propagation of longitudinal cracks along the fibers interfacial length, a greater fracture area is created in association with higher impact energy⁴⁷.

In Figure 5 it is also important to notice that the 30% specimen is not totally separated and few long fibers are still connecting the two parts. In fact, upon impact, the specimen bent around the hammer but did not separate in two parts due to the flexibility of some unbroken fibers. Similar situation occurred for impact tests of epoxy composites reinforced with 30% of other lignocellulosic fibers³⁸⁻⁴⁵. The reason for decohesion at the fiber/matrix interface, allowing longitudinal cracks to propagate and release intact giant bamboo fibers from the epoxy matrix, can be assigned to the low interfacial shear stress of any lignocellulosic fiber¹⁷.

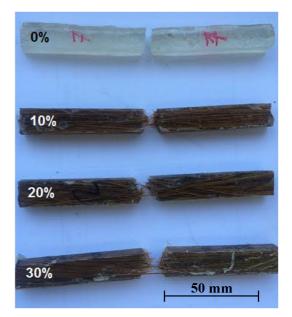


Figure 5. Typical rupture aspect of Charpy specimens of epoxy composites reinforced with different volume fractions of giant bamboo fibers.

Figure 6 shows SEM images of the impact fracture surface of a 30% giant bamboo fiber composite. With lower magnification, Figure 6a, it can be observed several fibers attached to the epoxy matrix in a specific area. Some long

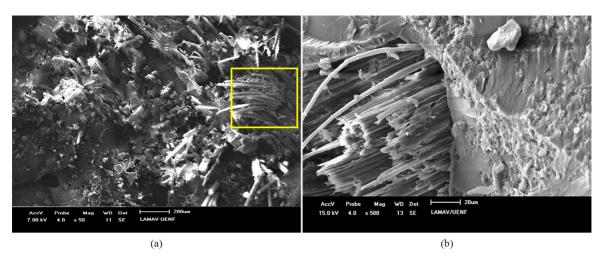


Figure 6. SEM fractographs of Charpy impact tested epoxy matrix composite reinforced with 30 vol% of giant bamboo fibers: (a) 50× and (b) 500× of magnification.

fibers are even sticking out of the transversal surface of the brittle epoxy matrix. A few holes could be caused by the giant bamboo fiber pullout due to, as aforementioned, longitudinal cracks that propagated at the low shear stress interface, releasing the fiber from the epoxy matrix. With higher magnification, Figure 6b, details of a fiber/matrix interface can be seen in a different area. The transversal rupture surface of the epoxy matrix (right side) indicates that the original crack nucleated at the specimen notch, was arrested at the fiber. The several microfibrils that constitute the fiber (left side) were longitudinally separated from the matrix by interface propagating cracks. A similar fiber/matrix interface is shown inside the insert in Figure 6a. Finally, the microfibrils were broken by tensile stresses generated upon the impact. These fiber/matrix interface longitudinal fracture and microfibrils tensile rupture provide the major contribution to the Charpy impact energy and justify its exponential increase, Figure 4 and Table 2, with the volume fraction of giant bamboo fibers.

4. Conclusions

 Epoxy matrix composites reinforced with up to 30% of continuous and aligned giant bamboo fibers display an exponential increase in toughness, measured by Charpy impact tests, as a function of the fibers volume fraction.

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- This increase in Charpy toughness is relatively smaller than those reported for other lignocellulosic fibers epoxy composites, probably due to defects introduced in the giant bamboo fiber during extraction by manual cut of the hard culm with razor blade.
- The exponential increase can be attributed to decohesion
 of the fiber/matrix low shear stress interface and
 tensile rupture of the microfibrils. These mechanisms
 result in higher absorbed energy as a consequence
 of longitudinal propagation of cracks and multiple
 broken areas of the numerous microfibrils.
- In spite of mechanisms responsible for the transversal fracture in the epoxy matrix as well as longitudinal decohesion of the fiber/matrix interface and tensile rupture of microfibrils, some giant bamboo fibers remained intact after the impact. In the 30 vol% fiber epoxy composites these intact fibers, owing to their bend flexibility, avoid total separation of the specimen in two parts.

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