Short beam strength of curaua, sisal, glass and hybrid composites

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ABSTRACT

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In this study, the short beam strength (SBS) characteristics of randomly oriented composites were comprehensively investigated. The following parameters were varied: fiber used (curaua or sisal), fiber washing or surface chemical treatment with sodium hydroxide/sodium borohydride, fiber length (from 5 to 60 mm), hybridization with glass fiber, and the pre-processing of the polyester resin. The overall fiber volume fraction was kept constant (30 vol.%). In all configurations, the composites containing curaua fiber obtained higher short beam strength than those with sisal. An increase in fiber length yielded higher SBS. The optical and electronic micrographs showed mostly horizontal cracks, typical of shear failure. In addition, it was carried out a study of the ASTM D2344 standard regarding the span-to-thickness (s:t) ratio recommended for testing, the measured strength decreased for higher s:t ratio for specimens with higher length and width, and the failure mode changed to bending around s:t > 12.

Keywords: Interlaminar shear strength; Short beam strength; Natural fibers; Alkali treatment; Hybridization; span:thickness ratio.

1. Introduction

Glass, carbon and aramid are generally costly synthetic fibers mainly used for structural composites, due to their high mechanical performance[1]. But, the use of natural fibers for non-structural applications is attracting growing attention in many sectors, such as civil naval and automotive. In the latter, they have already been used in door panels, trunk liners, parcel shelves, upholstery, rear parcel shelf, seat cushion parts[2], among others.

Among the vegetable fibers, sisal (*Agave sisalana*) is commonly used in composites, due to its abundance, easy processing, relatively good specific mechanical properties and low density. Curaua (*Ananas erectifolius*) fiber is not as easily available as sisal, however it has similar cost and superior strength compared to other vegetable fibers[2].

The wide use of vegetable fibers in composites is limited mainly due to comparatively low mechanical properties, high moisture absorption and lower compatibility to polymers in comparison with synthetic fibers. One way of increasing mechanical performance of vegetable fiber composites regards the concomitant use of a stronger fiber, producing hybrid composites[3]. For instance, Almeida Júnior et al.[4] reported that hybrid composites in which 30 vol.% of glass was replaced by curaua fiber was able to achieve similar results in hardness, impact strength, and dynamic mechanical properties.

The chemical treatment of vegetable fiber surfaces may be employed to improve fiber/matrix compatibility and to reduce water absorption[5,6]. The most common treatment uses NaOH, which is an inexpensive and effective way to minimize these drawbacks[5]. This treatment has recently been improved with the addition of sodium borohydride ($NaBH_4$) to the NaOH solution so that the $NaBH_4$ can act as a reducing

agent for the end-group (i.e. aldehyde) present in the C-1 free polysaccharides chain during treatment under alkaline conditions[7].

The mechanical properties of the composites are very dependent on the fiber/matrix interfacial strength[8]. The interface behaves as an important connecting link among the constituents, transferring stress from the matrix to the reinforcement[9]. The short beam test, formerly known as interlaminar shear strength (ILSS) test, is primarily used to evaluate strength of the composite when subjected to out-of-plane shear stress[10], but it has also be used to indirectly evaluate fiber/matrix adhesion and the overall quality of the composite. For instance, Karbhari[11] analyzed E-glass/vinylester composites processed under different curing temperatures and concluded that SBS decreased for higher temperatures due to disruption of the fiber/matrix interface and also to fiber degradation.

On this context, the aim of this work is to evaluate the effect of parameters like fiber treatment and length, and polyester pre-processing on the short beam strength of pure (curaua, sisal and glass) and hybrid (curaua/glass and sisal/glass) composites. Besides, a deeper study of the ASTM D2344M-00 standard applied to vegetable fibers was carried out to investigate the material response to the span/thickness ratio variation.

2. Materials and methods

2.1 Materials

The materials used in this study were:

- Curaua fiber rope, supplied by small local producers from the North Region of Brazil; Sisal fiber rope, purchased in the local market; Glass fiber roving (ME 3050, areal density: of 4000 g.km^{-1}) from Owens Corning. Based on the references, curaua fiber is expected to have higher tensile strength and modulus (900 MPa and 36 GPa,

respectively[12]) than sisal fiber (640 MPa and 15 GPa, respectively[13]), being both fibers inferior to E-glass (2000-3500 MPa GPa and 86 GPa, respectively[13]).

- Orthophthalic polyester resin, Polydyne 7001-04, supplied by Cray Valley; Butanox M-50 (methyl-ethyl-ketone peroxide) curing agent; Resin degassing agent (BYK-A 515).
- Sodium hydroxide (NaOH) and sodium borohydride ($NaBH_4$) for the fiber chemical treatment.

The fibers were used in three conditions: (i) Dried: The as-received fibers were dried in an oven with air circulation for 1 h at 105 ± 1 °C; (ii) Washed/Dried: The as-received fibers were washed by immersion in distilled water for 1 h and dried in an oven with air circulation for 1 h at 105 °C; (iii) Treated: Sisal and curaua fibers were treated by immersion in an aqueous solution of sodium hydroxide (NaOH) (5 wt.%) with sodium borohydride ($NaBH_4$) (1 wt.%) for 60 min at room temperature. Then, the fibers were neutralized with an aqueous solution of acetic acid (0.2 wt.%), thoroughly washed with distilled water and dried in an oven for 1 h at 105 °C. More specific details on this particular treatment can be found in Moraes et al.[7].

Fiber mats were manufactured by carefully placing the fibers in a metallic mold (internal dimensions: 270 × 170 × 22 mm) in a randomly oriented way. The fibers were cut in different lengths (5, 15, 30, 45 and 60 mm), using either dried, washed or NaOH/$NaBH_4$ treated vegetable fibers. Other mats were produced using a combination of previously intimately mixed curaua and glass or sisal and glass fibers, varying the volumetric ratio between fibers.

2.2 Composite Molding and Characterization

The composites were produced using polyester resin and 1.5 wt.% initiator. However, the resin was used either as received, after manual mixing of the P-MEK initiator for 5 min, or after degassing for 5 min in an ultrasonic bath (Unique, model UltraCleaner USC-1400A). In some cases, a chemical additive (i.e. a degassing agent) was added to the resin (0.5 wt.% of resin), the mixture was homogenized using mechanical stirrer for 5 min, left at rest for 25 min, and the initiator was added to the resin and manually mixed for 4 min prior to use.

The total fiber volume fraction was kept constant in 30 vol.%. The composites were hot compression molded (under 6 ton) for 60 min at 90 ºC using a hydraulic press (Marconi – MA 098/A3030). The nomenclature of the composites was based on the volumetric ratio between curaua (C) or sisal (S) and glass (G) fiber, namely: 100:0, 75:25, 50:50, 25:75 and 0:100 (for C:G or S:G), also considering the different conditions of the fiber and resin, as mentioned above.

The composites were characterized solely by short beam testing, following ASTM D2344M standard, in universal testing machine EMIC DL30000, c.a. 500 N load cell, using the standard span:thickness (*s:t*) ratio of 4:1, unless otherwise stated. The definition of length and width of the samples followed that recommended by the standard, (6×*t*, i.e. *1.5*×*s*) and (2×*t*, i.e. *0.5*×*s*), respectively. The ASTM recommended s:t ratio of 4:1 aims to minimize flexural, tensile and compressive stresses and to maximize shear stresses. In the current version of this standard (ASTM D2344/D2344M-00(2006)), the term ‘shear’ was removed from its title, since the stress state is not pure shear due to high stress concentration on the loading area and also due to the short zone of uniform shear stress[14].

Some of these drawbacks may be partially overcome by increasing the span:thickness ratio. Thus, a deeper investigation of this standard was carried out for the pure curaua composites by varying the *s:t* ratio (namely, 4:1, 6:1, 8:1, 10:1, 12:1 and 14:1). In this study, two sets of samples were used, both with the same thickness. In the first group, called Specimen A, the length was varied (1.5×*s*) but the width of the sample was kept constant (2×*t*) and, in the second group, called Specimen B, both length (1.5×*s*) and width (0.5×*s*) were varied.

Morphological analysis of the composite samples was tested carried out using Carl Zeiss optical microscope and JEOL (Model 6060) scanning electron microscope.

3. Results and discussion

The influence of the resin condition on short beam strength (SBS) of the curaua and sisal composites (fiber length: 30 mm) may be observed in Figure 1. The curaua composites presented a trend towards higher strength in relation to sisal, suggesting better adhesion of the former with the polyester resin, providing a stronger fiber/matrix interface. The as-received resin showed lower strength than that degassed in an ultrasonic bath, so this method was not considered effective, whereas adding a degassing agent increased strength of the curaua composites. On the other hand, the sisal composites did not show any clear trend.

Indeed, the SBS can be improved by increasing strength[15] of the matrix itself which could benefit, for instance, from a lower void content in the fiber/matrix interface or within the resin itself. Voids in the polyester resin are very common and difficult to eliminate since most of them are caused by the volatilization of styrene throughout the crosslinking process[16]. Thus, SBS of the polyester resin is generally lower than that of other thermoset resins like epoxy[17].

<< Insert Figure 1 >>

The short beam strength primarily depends on the matrix properties and the fiber/matrix interfacial strength rather than the fiber properties[18]. Nevertheless, Figures 2-3 show that fiber treatment and fiber length influence SBS of the studied composites. Figure 2 shows an increase in SBS of curaua and sisal composites (fiber length: 30 mm) which used washed/dried or chemically treated ($NaOH + NaBH_4$) fibers in comparison with those with just dried fibers. From these results, it can be concluded that fiber surface impurities and poorly bonded or water-soluble fiber material, which are partly removed during thorough washing or treatment, have a detrimental effect on SBS.

Figure 2 also shows that the ($NaOH + NaBH_4$) chemical treatment was more beneficial for sisal than for curaua composites (108% and 12% increase, respectively, in relation to the just dried fibers). This is expected since the parameters used in the treatment (agent concentration, time and temperature) have only been optimized for sisal fiber in the work of Moraes et al.[7], and these conditions do not appear to be the best ones for curaua fiber, and therefore these parameters should be investigated and optimized for each vegetable fiber prior to use. The decrease noted for alkali treated curaua fiber, comparing to washed and dried ones, could be related to cell thickening which leads to poor adhesion[19]. Seki[20] reported a similar increase in SBS of around 21% for jute/polyester after the NaOH treatment of the fibers. However, since the simpler washing/drying procedure yielded generally similar results compared to the most expensive chemical treatment, and both were better than just drying the fibers, the former was adopted for the rest of this study for both fibers.

<< Insert Figure 2 >>

Figure 3 shows that, for both composites, SBS increased in the 5 – 60 mm fiber length range (c.a. 59% for curaua and 69% for sisal composites). It is well-known that the efficiency of the matrix/fiber stress transfer in short fibers increases with fiber length under tensile loading, for instance. Nevertheless, this effect was also noticed on SBS results, perhaps because waviness of the fibers in the thick mat is more prominent for higher fiber length, and this could cause greater interference with out-of plane crack propagation during short beam failure due to a more pronounced fiber bridging effect. Besides, curaua again showed a trend towards higher SBS than sisal probably due to its superior overall strength (among other factors). Nevertheless, the 45 mm fiber length was adopted in the rest of the study to avoid issues related to poor fiber dispersion/arrangement of the random mat observed for very high fiber length.

<< Insert Figure 3 >>

Aiming to optimize the SBS of the composites based on vegetable fibers, intimate hybridization was carried out with glass fiber. Figure 4 shows the cross-sectional view of the 50:50 (C:G) hybrid composite produced. In this figure, it can be seen that the fibers were properly mixed and fiber segregation thorough the thickness of the composite was not noticed. Figure 5 shows the SBS of another batch of composites, some of them being C:G and S:G hybrid composites. SBS increased with the glass fiber content in both cases, which was expected due to the better overall mechanical performance of the glass fiber and the stronger interfacial adhesion with the polyester resin in comparison with the vegetable fibers (e.g. 2.6 MPa for sisal/polyester and 12

MPa for glass/polyester in pull-out experiments[21,22]). Nevertheless, the samples in which 25 vol.% of the glass fiber was replaced by curaua or sisal (i.e. 25:75 (C:G) and 25:75 (S:G), respectively) showed SBS values close to that of pure glass composites. Here again, the composites with curaua showed slightly higher SBS than those with sisal.

<< Insert Figure 4 >>

<< Insert Figure 5 >>

Typical load-displacement curves for these samples are shown in Figure 6. Similar patterns were noted for all composites, even though the composite with only sisal fiber showed a less prominent peak. Ahmed and Vijayarangan[18], who studied the short beam behavior of jute/glass/polyester hybrid composites, reported that the shape of the curves varied from flat plateau (for lower shear strength samples) to rounded (sharper) peaks (for higher shear strength samples), the former is somewhat similar to what was noticed in Figure 6 for sisal.

<< Insert Figure 6 >>

Early investigations reported that the failure mode and the SBS are strongly dependent on the s:t ratio[23]. Besides, it is well known that short beams (low s:t ratio) usually fail by shear and/or crushing whereas, long beams (high s:t ratio) show prevalent bending failure (tension and/or compression sides)[1]. ASTM 2344 standard indicates s:t ratio of 4:1 to produce interlaminar shear failure. Nevertheless, it is

important to investigate the effect of the variation of this ratio on the sample response so that the transition between failure modes can be identified, i.e. an optimum s:t ratio that maximizes the probability of interlaminar shear failure may be established[24], especially when new materials are being tested.

Curaua composites were chosen to study the effect of the variation of the s:t ratio (from 4:1 to 14:1) on SBS. The configuration used was fiber length of 45 mm, washed/dried fibers, with degassing agent. As mentioned before, two sample geometries were used for these tests, either varying only the length (specimen A) of the samples or varying their length and width (specimen B). Figure 7 shows the same basic trend for both geometries, i.e. SBS decreases with increasing s:t ratio, as previously reported by Markham and Dawson[23] and Adams and Busse[24]. It can also be seen that the decrease in SBS is slightly less pronounced if just the length of the specimen is varied, SBS remains nearly constant for s:t ratios equal to 8:1 or higher.

<< Insert Figure 7 >>

Figure 8 shows typical load-displacement curves obtained for some specimens A and B previously discussed in Figure 7. The nomenclature of the curves was based on s:t ratio and specimen, e.g. 6:1-SpA refers to the sample with s:t = 6:1 and for specimen A. These curves suggest different failure modes. For s:t = 4:1, there is a sharp peak that reaches higher stress, with a clear drop after that. For higher s:t ratio, specimen A shows similar curves, less pronounced than the reference curve, and all of them reaching approximately the same maximum, still with small displacement. For Specimen B, however, the curves progressively reach slightly lower peak height (lower maximum shear stress) and much higher displacement, which is an indication that, for these

samples, rupture is more severely caused by a combination of shear and bending forces [14].

In Figure 8, short beam strength (which is calculated from the maximum value of the curve) should be constant since the same composite is being tested. However, curve profile is being seriously affected when sample geometry varies, indicating the range of applicability of this test configuration. It also indicates the difficulty of establishing a particular s:t value for testing. Nevertheless, the curves obtained for samples of constant width (Specimen A) are less affected by the s:t ratio and therefore this geometry would be preferred.

<< Insert Figure 8 >>

According to ASTM D2344 and ASTM D790, short beam strength (τ) and flexural strength (σ) are calculated by:

$$\tau = \left(\frac{3P}{4wt} \right) \quad \text{and} \quad \sigma = \left(\frac{3Ps}{2wt^2} \right) \tag{1}$$

where: P is the load at failure, w is the specimen width, t is the specimen thickness, and s is the support span. Even though width is accounted for in both equations, its variation influences the combination of forces necessary to failure. Besides, wider samples make it more difficult for a crack to follow the mid-plane path throughout the width of the sample, which is the failure mechanism expected for short beam testing. Christiansen et al.[25] also compared the short beam strength with the flexural strength in glass/polyester composites and they concluded that the SBS decrease with higher s:t ratio, whereas the flexural strength is more active at higher s:t ratio.

Figure 9 presents the visual aspect of the tested samples. Figure 8(a) suggests that the samples with s:t = 4:1 failed in shear mode, with minimum crushing. On the other hand, bending failure is predominant in samples with s:t = 12:1 and higher, as can be seen in Figure 9(b) for s:t = 14:1. Rosensaft and Marom[26] considered that the failure mode transition is not sharp, it occurs over a range of s:t ratios. This is further supported by SEM micrographs of the curaua composites shown in Figure 10. These micrographs indicate that for low s:t ratio (4:1 and 6:1), Figures 10(a-b), the sample failed mainly by shear, featuring mostly horizontal cracks, many of them along the fiber/matrix interface and near the mid-thickness of the sample. For s:t ratio ≥ 12 (Figure 10(c-d)), the samples tended to fail by bending, as indicated by cracks running vertically in the sample. Similar results have been reported by Adams and Lewis[14].

<< Insert Figure 9 >>

<< Insert Figure 10 >>

4. Conclusions

In this paper, the interlaminar shear properties of random curaua, sisal, glass and hybrid composites were investigated. The curaua composites showed generally higher short beam strength than sisal composites, suggesting better adhesion of the former with the polyester matrix. Short beam strength was found to increase when the fiber had been washed/dried in comparison to as-received (only dried) fibers. Chemical treatment with combination of NaOH + $NaBH_4$ improved SBS of sisal composites only, achieving 108% increase compared to the only dried fibers. Besides, short beam strength was

found to increase with fiber length for both fibers and with the use of a degassing agent for the polyester resin, to reduce voids in the composite.

Hybridization of the vegetable fibers with glass fiber was successful in increasing strength. Also, curaua/glass composites yielded higher strength than sisal/glass composites and the partial substitution (25 vol.%) of glass with curaua yielded short beam strength near that of pure glass composites.

The estimated short beam strength decreased for higher s:t ratio. In addition, short beam curve profile and maximum load were seriously affected when sample geometry varied, indicating the range of applicability of this test configuration. It also shows the difficulty of establishing a particular s:t value for testing. Nevertheless, keeping the width of the specimen constant was more successful in avoiding interference with the test in a wider range of s:t. Observation of the load-displacement curves and the failed specimens along with calculations of SBS suggest shear failure for s:t ratio of 4:1 (pure) and 6:1 (with little contamination), combined failure for 8:1 and 10:1, and bending failure for 12:1 and higher.

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