Original Article

Statistical analysis of notch toughness of epoxy matrix composites reinforced with fique fabric

Michelle Souza Oliveira a,*, Fabio da Costa Garcia Filho a, Fernanda Santos da Luz a, Artur Campos Pereira a, Luana Cristyne da Cruz Demosthenes a, Lucio Fabio Cassiano Nascimento a, Henry Alonso Colorado Lopera b, Sergio Neves Monteiro a

a Military Institute of Engineering - IME, Department of Materials Science, Praça General Tibúrcio, 80, Praia Vermelha, Urca, RJ, CEP 22290-270, Urca, Rio de Janeiro, RJ, Brazil
b University of Antioquia, Faculty of Engineering, Block 20, 53-108, street 67, Medellin, Colombia

Article history:
Received 29 April 2019
Accepted 30 September 2019
Available online 10 November 2019

Keywords:
Fique fabric
Impact tests
Epoxy matrix
Natural fiber composite

ABSTRACT

Reinforcement of polymer matrices with natural lignocellulosic fibers (NLFs) is today presenting great potential for the reduction of both product weight and cost in many industrial sectors as well as the advantage of being renewable and eco-friendly materials. For some industrial applications, especially in ballistic armor, the materials are expected to meet technical and mechanical requirements in terms of impact behavior, which motivate the present work. This work conducted a notch toughness assessment by Izod and Charpy impact tests of epoxy matrix composites reinforced with different volume fractions of fique fabric, up to 50 vol%. Statistical analyses indicated significant differences between composites with distinct amount of fique fabric. Failure mechanisms were analyzed by scanning electron microscopy (SEM). The results revealed that composite reinforced with 40 vol% of fique fabric presented the best notch toughness performance. Composites with 15 and 30 vol% of fique fabric display predominantly brittle fracture associated with lower impact energies. With higher amount, 50 vol%, of fique fabric reinforcement both Izod and Charpy impact energy suffered a small decrease according to the Roger and Plumtree model.

© 2019 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

1. Introduction

Synthetic fiber reinforced composites have been a successful class of engineering materials since last century [1,2]. Advanced composites reinforced with modern synthetic nanofibers [3,4] and nanoparticle [5] are still today being investigated for a novel high-performance technological development. Based on sustainability issues, however, these composites with synthetic reinforcements are being questioned due to non-biodegradability, poor recyclability and, especially, high costs associated with their production. This motivates researches on the use of natural lignocellulosic fiber...
as composites (NLF) reinforcement [6], as can be seen by the number of publications in ISI Web of Science with the keyword “natural fiber composite”, which was over 1,300 in 2018. Characteristics such as eco-friendly, biodegradable, renewable resource and low-density products are the most claimed advantages in the use of NLFs reinforced composites [7–17]. Geometry, shape, chemical composition, orientation and the relative amount of the fibers are of major importance in the load transfer between matrix and reinforcement, and consequently, in the mechanical behavior of NLF composites [18]. Indeed, some remarkable progress in polymer matrix composites reinforced with NLFs has been achieved and their use are today seen in the automotive [19,20] and aeronautical industries [21] as well as packing [22] and even bulletproof vests [23–28], which requires impact resistance.

Among the promising NLFs, the fique fiber, a relatively unknown fiber extracted from the leaves of an Andean plant Ficus carica andina, is noteworthy. Surface modification, thermal properties, bending resistance, tensile strength, and ballistic protection have been preliminarily investigated in fique fibers [29–34]. Furthermore, the low cost of production and average density, in the range from US$0.36 to US$0.45 and about 0.67 g/cm³, respectively [34], are both economical and technical advantages of the fique fibers. These characteristics can be pointed as an important motivation to the continuous investigation about the properties and application of polymer matrix composites reinforced with fique fibers.

Works on polyester [35–37] matrix composites reinforced with fique fabric were recently published. The main reported results were related to maximum incorporation of 30 vol% reinforcement. In these previous works, the relationship between the absorbed impact energies and the reinforcement incorporation was found to be linear. Indeed, the values obtained for 30 vol% fique fabric reinforced polyester composites both Izod, 260 ± 58 J/m [36], and Charpy, 293 ± 78 J/m [37], notch toughness are marked higher than plain polyester with 25 ± 2 and 24 ± 1 J/m, respectively. Moreover, the performance of these 30 vol% fique fabric/polyester composites are significantly above those of other NLFs composites, such as 40 vol% coir fiber/polyester, 121 J/m [36] in Izod test. Epoxy matrix composites have not yet been investigated in similar tests.

The present work proposes a statistical assessment of epoxy matrix composites reinforced with up to 50 vol% of fique fabric in terms of impact tests. Izod and Charpy configurations were also used to evaluate the absorbed impact energy, associated with notch toughness, by the composites. The results of both tests were statistically analyzed by ANOVA and Tukey tests. A comparison between tests was made based on Rogers and Plumtree approach [38]. Fractured surfaces were analyzed by scanning electron microscopy (SEM) in order to determine the main failure modes in the investigated composites. It is important to emphasize that, dealing with the use of fique fabric epoxy composites, impact tests in the same conditions exhibit by the present work were not found in the literature.

2. Materials and methods

Fique fabric commercially available in Medellin, Colombia, was purchased by one of the authors (HAC) from a local commercial outlet. Fig. 1 (a) illustrates a bundle of fique fibers while in Fig. 1 (b) one may observe a piece of as-received fique fabric as well as an insert that details the microscopic aspect of the fabric.

The area density of the plain-woven fabric was found to be 0.860 kg/m², in accordance with the NBR 10591/98 [39]. This result is greater than that previously reported [35], which presents lower fiber count, i.e., a more open woven. The polymer used as the matrix was a commercial epoxy resin diglycidyl ether type of bisphenol A (DGEBA) cured with triethylenetetramine (TETA), using the phr of 13 parts of hardener to 100 parts of resin, both supplied by Epoxyfiber, Brazil. The choice for DGEBA/TETA epoxy for a composite matrix was based on its properties and engineering applications [40]. Moreover, based on a comparison with polyester, Gopinath et al. [41] indicated that epoxy composites display, for some applications, a better performance than polyester composites. The fique fabrics were dried in an air oven at 60 °C for 24 h and subsequently mixed in amounts of 15, 30, 40 and 50 vol% with epoxy resin to prepare the composites. Plates of these composites with 10 mm thickness were fabricated in a rectangular steel mold with dimensions 150 x 120 mm. All composites were fabricated by intercalating layer of fique fabric with still fluid epoxy resin, already mixed with hardener, inside the mold. A maximum of 4 layers was possible to accommodate, corresponding to 50 vol% fabric embedded with resin, before applied compression onto the mold. The curing process of the composite plates took place under 3 MPa pressure for 24 h, at room temperature (25 ± C).

After unmolded, composites specimens were cut with standard dimensions for impact tests, according to ASTM D6110 and ASTM D256 [42,43]. The specimens were notched with 45° of angle, transversely to the direction of compression molding. A total of 5 specimens per each volume fractions of fique fabric for Charpy and Izod impact tests were obtained. Fig. 2 illustrates schematically the dimensions of the specimens and the produced specimens in perspective.

Impact tests were performed in a PANTEC instrumented pendulum, model XC-50, using an 11 J impact hammer. According to the ASTM D256 [43], there are significant differences between tests, Izod and Charpy, which can lead to distinct results. Therefore, after the tests, the results were statistically evaluated by ANOVA and Tukey test as well as compared with a similar approach to that proposed by Rogers and Plumtree [38]. After the impact tests, the fractured surfaces of the specimens were analyzed by scanning electron microscopy (SEM) in a model Quanta FEG FEI equipment.

3. Results and discussion

Fig. 3 shows the macroscopic aspect of fracture composites samples with different amount of incorporated fique fabric, up to 50 vol%.

By visual analysis, one should notice that completely brittle fracture occurred for the 15 vol% fique fabric. Such behavior is typically observed for the epoxy resin in a neat condition [44]. By contrast, as the amount of fabric reinforcement increases, apparently an evolution in the fracture mode of the composites occurs. The fiber behavior, in the fabric, influences the
Fig. 1 – (a) Bundle of fique fibers and (b) as-received piece of fique fabric with SEM microscope magnifications details of 1,600× in (a) and 80× in (b).

Fig. 2 – Specimens dimensions schematically and the fabricated (a) Charpy (b) and Izod impact test specimens.

impact energy absorption. This behavior may be associated with the non-regular rupture surface of epoxy matrix near the reinforcing fabric. The relatively stronger fique fiber [29] acts as an obstacle to crack propagation as will be further discussed. One may notice that the specimens with 40 and 50 vol% were not completely fractured after the impact test. This could be related to the relatively high tensile strength characteristic of the fique fabric, which avoids a total collapse of the material. In other words, the incorporation of fique fabric in the epoxy matrix significantly changes the fracture mode of these composites as observed in Izod and Charpy notch toughness tests. Therefore, these results are indicative of the composite higher toughness, since if a total rupture occurred, the energy absorbed would be even higher.

The toughness results of Izod and Charpy impact energy with the volume fraction of fique fabric are shown in Fig. 4. It should be noted that the incorporation of fique fabric in the epoxy matrix significantly increases the notch toughness of the composites for both Izod and Charpy. The highest values of 222 ± 52 J/m, in Izod and 485 ± 192 J/m, in Charpy toughness, were reached for 40 vol% of fique fabric.

The increase observed in Fig. 4 is 2.3 times higher for the Izod and 2.1 higher for the Charpy, considering the relation between the higher toughness for the 40 vol% and that for the lower 15 vol% incorporation. In addition, the high dispersion of the values given by the error bars could be associated with the heterogeneity characteristic of NLFs, which might cause variation in the properties of these fibers [10]. These results
were validated through the analysis of variance (ANOVA) and Tukey test.

Tables 1 and 2, present the ANOVA for the Izod impact energy of the investigated composites as well as the Tukey test to determine the significant differences between each condition of these samples, respectively.

According to the results obtained by ANOVA in Table 1, it is shown that, with a reliability level of 95%, one should reject the hypothesis that the mean impact energy of composites incorporated with different fractions of fique fabric are equal, since $F_{\text{calculated}} (8.92) > F_{\text{critical}} (3.24)$. Therefore, it is possible to affirm that the volume fraction of fique fabric in the epoxy matrix composites have different effects on the Izod impact energy. In addition, the Tukey test results in Table 2 revealed that the condition, which stands as the most energy absorbing one is related to Izod impact test. The least significant difference (LSD) obtained was 59.52 J/m, i.e., the difference between two mean values higher than this value is significantly different. In bold are highlighted the differences between mean values larger than the LSD.

Likewise, similar approach was made for the Charpy test results. These results are presented in Tables 3 and 4.

Comparing the value of $F_{\text{calculated}}$ with $F_{\text{critical}}$ (Table 3), it is rejected the hypothesis that means are equal to 5% significance level, since $F_{\text{calculated}} (5.31) > F_{\text{critical}} (3.24)$. Therefore, the type of material used also influences the energy absorbed in the Charpy impact. As for the individual means by the Tukey test, the LSD obtained was 150.16 J/m.
For both tests configuration, Izod and Charpy, the most noteworthy conditions were the 40 and 50 vol% of fique fabric reinforcement. This result evidences the assumption that the evolution of fracture mode suggested by the macrographs in Fig. 3, could be associated with the amount of absorbed energy. This energy increases with the amount of fique fabric reinforcement up to 40 vol%, as shown in Fig. 4.

Fig. 5 presents a comparison between the amount of energy absorbed by Izod and Charpy as proposed by Rogers and Plumtree approach [38]. One may verify that the proposed mode of energy absorption was similar for both test configura-

![Table 4 - Honest significant difference (HSD), Tukey test analysis for Charpy impact.](image)

<table>
<thead>
<tr>
<th>Composites</th>
<th>E-15%FF</th>
<th>E-30%FF</th>
<th>E-40%FF</th>
<th>E-50%FF</th>
</tr>
</thead>
<tbody>
<tr>
<td>E-15%FF</td>
<td>-</td>
<td>138.94</td>
<td>254.91</td>
<td>230.64</td>
</tr>
<tr>
<td>E-30%FF</td>
<td>138.94</td>
<td>-</td>
<td>115.97</td>
<td>91.71</td>
</tr>
<tr>
<td>E-40%FF</td>
<td>254.91</td>
<td>115.97</td>
<td>-</td>
<td>24.27</td>
</tr>
<tr>
<td>E-50%FF</td>
<td>230.64</td>
<td>91.71</td>
<td>24.27</td>
<td>-</td>
</tr>
</tbody>
</table>

Bold values significance the differences between mean values larger than the LSD.

![Fig. 5 – A comparison between the results obtained for Charpy and Izod impact test by the Rogers & Plumtree approach [38] for fique fabric composites.](image)

![Fig. 6 – SEM micrograph of the fracture for a 50 vol% fique fabric composite with (a) 400× and (b) 200× of magnification.](image)

![Fig. 7 – SEM micrograph of the fracture for a 40 vol% fique fabric composite with (a) 400× and (b) 1,500× of magnification.](image)
tions. Moreover, a linear trend of increase could be suggested. Yet, for the 50 vol% condition it was observed a change from this linear trend. This behavior could be associated with a maximum amount of fiber in the composition of the composite, which for contents above 40 vol% would result in defects due to the poor wettability of the fabric by the epoxy matrix.

Fig. 6 shows SEM fractographs of epoxy composites with the largest possible amount of 50 vol% fique fabric investigated. For this amount, a relatively weak wettability of fique fibers, in the fabric, the epoxy matrix causes comparatively more structural damage upon the impact. Fracture along the fibers, Fig. 6 (a), reveals delamination at the matrix interface as well as fibers pullout, Fig. 6 (b), in the transverse direction. The fibers, however, still act as obstacles to crack propagation. These structural conditions associated with limited performance in both Izod and Charpy notch toughness, Fig. 4, reverts the linear increase expected by the Rogers and Plumtree approach [38] shown in Fig. 5.

Fig. 7 shows SEM fractographs of 40 vol% fique fabric reinforced epoxy composites. A better wettability between fibers and the epoxy matrix is illustrated in Fig. 7 (a). Moreover, evidence of interface fiber/matrix cohesion is attested in Fig. 7 (b). For this amount of fique fabric the highest Izod and Charpy performance, Fig. 3, was attained still maintaining an expected linear increase [38] depicted in Fig. 5.

Caprino et al. [45] and Alimuhammad et al. [46] reported that typical damage modes observed in composites materials after impact tests include matrix cracking, delamination, fiber breakage, and fiber pullout. Indeed, those damages mechanisms were all observed in Fig. 6 (a) and (b). In Fig. 6 (a) one may notice the delamination between fiber/matrix and longitudinal fiber rupture pointed by arrows. While in Fig. 6 (b) fibers pullout is the main mechanism observed. It should also be observed in Fig. 6 (a) that each fique fiber composing the fabric acts as a barrier to cracks that propagate inside the epoxy matrix.

4. Conclusions

- Charpy and Izod impact energies increase with the volume fraction of fique fabrics incorporated into the epoxy matrix up to 40 vol%. Among the specimens evaluated by the Tukey test with 95% confidence level, the composite with 40 and 50 vol% of fique fabrics were those that exhibited the best results 485 and 222 J/m in terms of Charpy and Izod impact energy absorption, respectively.

- Predominantly brittle fracture occurs in composites with volume fractions of 15 and 30 vol% fique fabrics. These composites exhibit significantly lower Charpy and Izod impact energy than composites reinforced with 40 and 50 vol% of fique fabrics. This latter was the largest possible amount incorporated into the epoxy matrix.

- A modeling for comparison of Izod and Charpy results is suggested based on Rogers and Plumtree approach. Moreover, a linear trend up to 40 vol% of fique fabric was associated with the variation of the amount of energy absorbed and the amount of fiber reinforcement.

- For 50 vol% of fique fabric, the relatively weaker wettability between fiber and epoxy matrix reverts the expected linear increase in notch toughness performance.

- The damage mechanisms observed in this investigation were the same as reported for other polymer matrix reinforced with natural fibers composites and includes fabric delamination, fiber breakage and fiber pullout from the epoxy matrix.

Acknowledgements

The authors thank the support to this investigation by the Brazilian agencies: CNPq, FAPERJ and CAPES.

References


