Ballistic performance of epoxy composites reinforced with Amazon titica vine fibers

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Abstract

Natural fiber-reinforced matrix polymer composites have shown great potential for engineering applications including ballistic protection. However, The Amazon region is full of natural fibers that have not yet been the fully studied and that might have important properties making them more attractive than synthetic fibers. This work presents a novel composite material consisting of titica vine fibers (Heteropsis flexuosa) reinforcing epoxy resin, for possible application in ballistic armor. Composite plates with different volumetric fractions of titica vine fibers (TVF) were subjected to a ballistic test, which consisted of firing multiple shots using .22 LR caliber ammunition. Through the results, it was possible to observe, with statistical validation by Weibull and ANOVA analyses, as well as the Tukey test, that a greater absorption of energy occurred for the conditions of 0 to 20 vol%, but with considerable loss of integrity. On the other hand, the 30 and 40 vol% samples have shown better integrity. In addition, the V_L calculated for the samples with the highest percentage of fibers was lower than those found for the composites with 10 and 20 vol%. Thus, the higher the volumetric fraction of TVF, the lower the impact energy absorption capacity. This behavior corroborates the analysis by scanning electron microscopy (SEM) of the fracture surfaces of the composites after the ballistic test. For composites with less than 20 vol% TVF, brittle fractures evidences were observed, responsible for absorbing more impact energy. On the other hand, for other compositions, there was a predominance of complex mechanisms characteristic of ductile fracture, responsible for absorbing less energy, but still maintaining the integrity of the protection.

Keywords: Natural fibers; Titica vine fibers; Composites; Ballistic armor.

1 Introduction

Natural lignocellulosic (NLFs) fibers reinforced polymer matrix composites are well-known as biodegradable materials, fully or partially recyclable and renewable they are increasingly being disseminated both in scientific research and industrial applications. The great interest in NLFs as a reinforcing material lies in their ease of use, low cost, and good specific properties. Those facts make them an outstanding ecologically correct option for the production of composites [1].

Brazil is one of the greatest manufacturers of NLFs, as it is located in a tropical region, with extensive forests and a favorable climate for the development of plants [2]. Especially, in the Amazonian region NLFs, such as buriti [3], guaruman [4] and caranan [5] have recently been studied researches have shown that NLF reinforced composites present high strength limits, good mechanical properties and satisfactory ballistic performance if employed in multilayered armor systems (MASs) [6-8].

Among the NLFs, the Titica vine fiber (TVF), extracted from the aerial roots of *Heteropsis flexuosa* (Figure 1), is a representative species of the *Araceae* family [9], which is considered as a typical Amazonian plant, with occurrence in areas where there are no floods [10]. TVFs are part of non-timber forest products, which exhibit versatility and variability in terms of employment. These roots are highly appreciated for having long, resistant fibers, easy to work with, and cost-effective for manufacture of baskets, furniture, brooms, rugs and others [11,12]. According to its attractive and little explored scientifically features, TVF shows up as a potential reinforcement material for engineering applications, specifically in ballistic armor. Regarding the mechanical properties of the TVF composites and the single fibers, Table 1 presents some previously studied results for impact resistance.

According to Table 1, interesting properties of TVFs are observed for applications in composites. The apparent

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density of these fibers is one of the lowest reported, being suitable for applications that require lightweight materials [13]. Furthermore, Cunha et al. [13] determined a total porosity of 69%, with potential use in the acoustic insulation industry. Although single TVFs present intermediate mechanical properties [13], when incorporated into an epoxy matrix, a tendency towards an increase in the impact energy absorbed with the fiber fraction was observed in both Izod and Charpy tests [14].

Numerous studies have been developed with the aim of investigating the properties of composites reinforced with NLFs in multilayered armor systems (MASs) for personal protection [15-18]. A MAS is composed of three layers. The front layer is formed by a ceramic material whose purpose is to wear down the projectile tip and absorb most of the impact energy [18]. In the subsequent layer, it is common to use high-performance polymers such as ultra-high molecular weight polyethylene and polyaramid, responsible for absorbing the residual impact energy of the projectile and shrapnel from the projectile and the ceramic itself [18]. However, recently the literature has pointed to the possibility of using composites reinforced with NLFs in this second layer. Finally, the third layer is formed by metallic materials or high tenacity polymeric fabrics [18,19].

Unlike NLFs which are low-cost, synthetic fibers such as aramid fibers can represent a disadvantage in the same aspect, because they are expensive. In addition, as there is no ecological destination for materials for war use, NLFs stand out as promising substitutes for conventional use, as they are from renewable, non-toxic, recyclable and biodegradable sources [20]. In this context, the main objective of the present work was to investigate, for the first time, the ballistic behavior of epoxy matrix composites reinforced with 0 to 40 vol% of TVFs against .22 caliber ammunition.

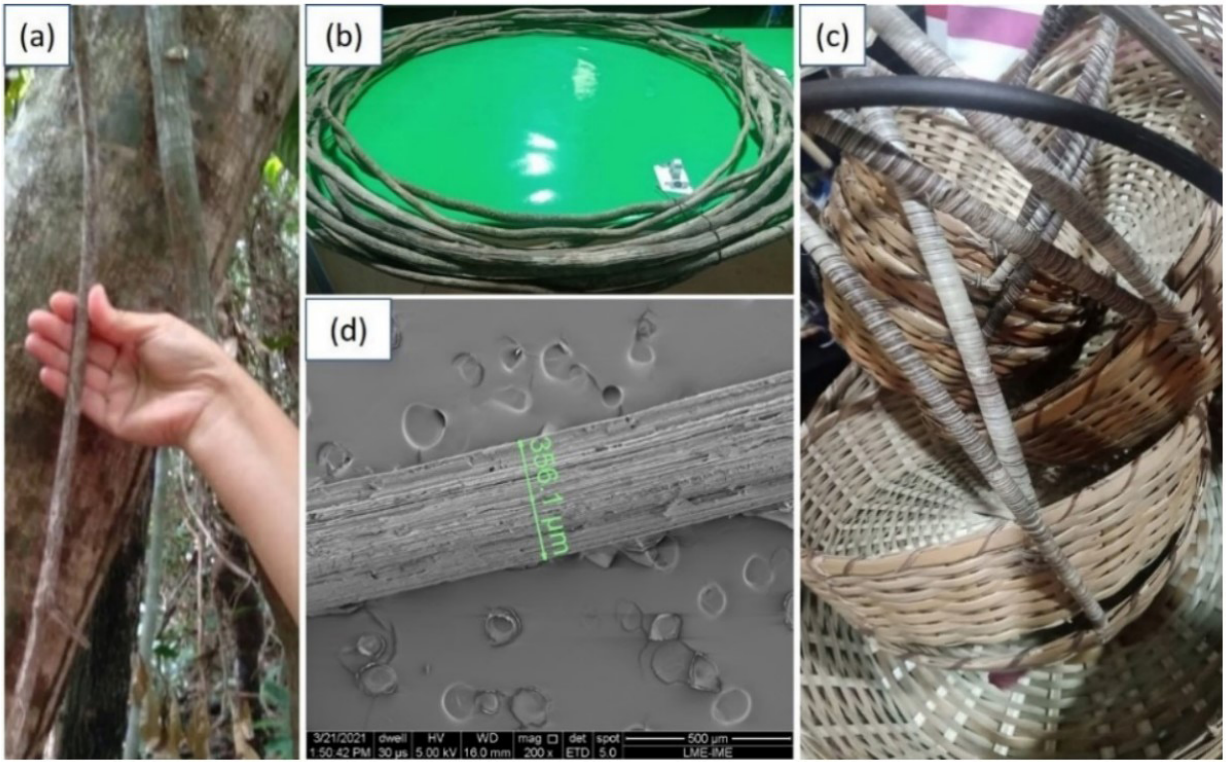

Figure 1. (a, b) Titica vine roots found in the forest; (c) baskets made from TVFs; (d) SEM image of the longitudinal section of the TVF.

Table 1. Mechanical properties of single TVF and its composites

Conditions	Density (g/cm^3)	Diameter (μm)	σ_{max} (MPa)	E (GPa)	ε (%)	References
Single TVF	0.50 ± 0.07	650.1 ± 175.7	25.92 ± 6.69	1.02 ± 0.22	7.36 ± 2.05	[13]
	Izod Impact Energy (J/m)			**Charpy Impact Energy (J/m)**		
10TVF	24.20 ± 6.10			66.44 ± 8.72		[14]
20TVF	25.43 ± 6.58			50.54 ± 5.73		[14]
30TVF	41.01 ± 4.37			62.13 ± 5.22		[14]
40TVF	58.65 ± 9.26			75.05 ±7.32		[14]

As well as to identify the fracture mechanisms acting on composite samples after the shooting.

2 Experimental procedures

2.1 Materials

The titica vine roots were acquired in the local market of the city of Boa Vista, located in the state of Roraima, Brazil. Splints measuring approximately 15 cm were immersed in water for 24 h to facilitate the extraction of fibers with a blade.

The polymer used as the matrix was an epoxy resin type diglycidyl ether bisphenol A (DGEBA), hardened with triethylenetetramine (TETA), associated with a stoichiometric ratio of 13 parts hardener to 100 parts of resin. Both resin components were supplied by Epoxy Fiber, Rio de Janeiro, Brazil.

2.2 Composite processing

For the manufacture of composites, a metallic mold, with an internal volume of 214.2 cm^3 (dimensions 15 x 12 x 1.19 cm) was used. The resin/hardener mixture was poured into the mold at a previously calculated fiber/resin ratio. For these calculations, the fiber was measured by the geometric linear density method, which consists of weighing about 100 fibers and measuring their length and diameter using an optical microscope. The fiber density was obtained by dividing the weight by its calculated volume, with an average value of 0.50 g/cm^3 being found. The epoxy resin density value of 1.11 g/cm^3 was taken from the literature [21]. The TVFs were washed in running water and dried in an air oven at 60°C for 24 h. Then, they were deposited in the mold in the preferably aligned direction. Samples of 0 – 40 vol% of TVFs were produced.

Finally, the composite plates were pressed in a hydraulic press with a load of 5 tons for 24 h, until it was completely cured. The density of the 10 - 40 vol% plates could be estimated by the mixture rule [21] and ranged from 1.04 - 0.86 g/cm^3.

2.3 Ballistic tests

For the ballistic tests, one composite plate was produced for each condition (0, 10, 20, 30 and 40 vol% fibers), with 5 shootings being fired on each plate. A Gunpower SSS (4000 psi) compressed air rifle was used to perform the test, as well as commercial ammunition of 0.22 LR of 3.3 g of weight. The tests were carried out in the laboratory of the Military Institute of Engineering (IME), located in Rio de Janeiro - Brazil.

Regarding the measurements of projectile impact (V_i) and residual (V_R) velocities, a model MK3 Air Chrony gun chronograph, with a precision of 0.15 m/s, and a model Pal ProChrono gun chronograph, with a precision of 0.31 m/s, were placed, respectively, 10 cm before and after the target. The shooting was performed perpendicularly to the target at a distance of 5 m.

In order to compare the materials tested, the energy absorbed by the target (E_{abs}) was estimated by applying the difference in kinetic energy before and after impact, as shown by Equation 1, where “m” is the mass of the projectile [22].

$$E_{abs} = \frac{m\left(V_i^2 - V_R^2\right)}{2} \tag{1}$$

From the values of E_{abs}, the limit velocity (V_L) can be estimated, following Equation 2, which is associated with the maximum velocity that the target would resist.

$$V_L = \frac{\sqrt{2E_{abs}}}{m} \tag{2}$$

2.4 Statistical validation

In order to statistically validate the level of reliability and significance of the results obtained in the ballistic test, Weibull analysis and analysis of variance (ANOVA) were performed together with the Tukey test. The Weibull parameters β and θ in the frequency distribution function are associated, as shown in Equation 3.

$$f\left(x\right) = \exp\left[\left(\frac{x}{\theta}\right)^{\beta}\right] \tag{3}$$

These parameters, with the R^2 of precision, contribute to assess the level of reliability of the data. The ANOVA and the Tukey test make it possible to reveal the existence of differences between the mean and standard deviation values of the energy absorbed by the target, with a confidence level of 95%.

2.5 Scanning electron microscopy (SEM)

SEM analysis of the composite's ballistic fractured surface was performed by in a model Quanta FEG 250 FEI microscope operating with secondary electrons accelerated at 2 KV. For the preparation of the samples, they were extracted from the cross-section in relation to the shot surface of the target. Then they were gold-coated for observation with secondary electrons.

3 Results and discussion

3.1 Ballistic tests

The composite plates with 0, 10, 20, 30 and 40 vol% of TVFs were subjected to ballistic testing, using 0.22 LR caliber ammunition, in order to estimate the ballistic efficiency of the intermediate layer. Through this test, it was possible to estimate the absorbed energy (E_{abs}) and the limit velocity (V_L) of each tested condition. Table 2 are presented the values of variables such as projectile mass (M_p), average impact velocity (V_i), average residual velocity (V_r) and energy absorbed (E_{abs}) for each of the conditions tested.

From the data presented in Table 2, as the amount of TVF is increased there is a tendency of increasing the residual velocity of the projectile. As a result, the 10TVF composite exhibited higher energy absorption capacity (98.12 ± 8.99 J) compared to all TVFs composites. This can be explained by the predominance of the brittle behavior of the epoxy matrix [23,24]. However, due to such behavior, for ballistic applications, plain epoxy is completely fragmented and therefore not suitable for personal protection considering multiple shootings. Figure 2 shows the post-impact aspects of the composite plates tested.

Table 2. Projectile mass (M_p), average impact velocity (V_i), average residual velocity (V_r) and absorbed energy (E_{abs}) values for samples of 0 – 40 vol% of TVF

Samples	M_p (g)	V_i (m/s)	V_r (m/s)	E_{abs} (J)
Epoxy (0TVF)	3.34 ± 0.06	270.48 ± 5.13	93.33 ± 28.96	105.54 ± 7.23
10TVF	3.35 ± 0.03	269.99 ± 6.77	104.06 ± 59.31	98.12 ± 8.99
20TVF	3.29 ± 0.09	270.91 ± 6.49	121.19 ± 15.49	95.26 ± 5.14
30TVF	3.32 ± 0.02	269.81 ± 4.97	144.41 ± 8.05	85.06 ± 4.48
40TVF	3.34 ± 0.08	270.11 ± 4.99	153.86 ± 8.61	81.02 ± 5.87

Figure 2. Samples after residual velocity test. (a) 10TVF; (b) 20TVF; (c) 30TVF; (d) 40TVF.

Regarding the post-impact aspects of the tested samples, i.e, the physical integrity, after five shootings, the 40TVF composites presented the best dimensional stability and integrity among them. On the other hand, plain epoxy resin was completely shattered, similarly to 10TVF. According to Monteiro et al. [25], the integrity criterion is of great importance for ballistic applications.

From the data provided in the ballistic test, the limit velocity values (V_L) for the groups tested were determined. Table 3 presents the results obtained from this research in comparison with recent works from the literature.

The limit velocity (V_L) understood as the velocity from which the projectile can pierce the target and below this the target is barred [27] was estimated from the measurements of energy absorbed by the composite plates.

Despite the downward trend in absorbed energy, it is noticeable that the V_L values for composite TVFs plates are comparable to other NLF composites recently used for the same purpose, incorporated in epoxy matrix. Samples of 30 vol% TVFs showed a V_L slightly lower than those of hemp fibers and with superior results than those with junco fibers, as shown in Table 3.

3.2 Statistical validation

From the Table 4, Weibull's statistical analysis was performed, with the purpose of determining characteristics and reliability trends of the samples tested. Table 4 presents the Weibull distribution for the energy absorbed from the composite for all configurations.

Evaluating Table 4, one could notice that this statistical treatment represented, in general, a homogeneous characteristic of the samples individually. For better understanding, Figure 3 presents the graphs plotted through the parameters obtained in this analysis for each percentage of TVFs studied.

From Figure 3, one could verify that the points are only a little distant from the adjustment line. In addition, it is important to note that the characteristic value (θ) is similar to that found by the average energy absorbed by the composites. In order to verify the occurrence of a significant difference between the results of absorbed energy in the ballistic test by the composite plates, ANOVA was applied to the results, the data obtained are shown in Table 5.

Table 3. Limit velocity (VL) values from the present work and other authors

Samples	V_L (m/s)	References
10TVF	243.66 ± 10.93	PW
20TVF	240.2 ± 6.53	PW
30TVF	226.99 ± 6.08	PW
40TVF	221.46 ± 8.10	PW
Epoxy/Hemp - 10%	238.4 ± 14.1	[26]
Epoxy/Hemp - 20%	246.9 ± 7.2	[26]
Epoxy/Hemp - 30%	256.3 ± 10.5	[26]
Epoxy/Junco - 10%	221.5 ± 5.5	[16]
Epoxy/Junco - 20%	215.2 ± 15.9	[16]
Epoxy/Junco - 30%	212.5 ± 15.2	[16]

PW – Present work.

Table 4. Weibull distribution for absorbed energy by the composite for all in natura compositions tested

Samples	β	ϴ	R^2
Epoxy (0TVF)	14.16	109.00	0.9005
10TVF	10.00	102.70	0.7153
20TVF	18.55	97.69	0.9589
30TVF	16.77	87.49	0.7743
40TVF	13.59	83.81	0.9348

Table 5. ANOVA of the absorbed energy by the composites from 0 – 40 vol% in the ballistic test

Variation Causes	DF	SS	MS	F_{calc}	$F_{critical}$
Treatment	4	1,975.33	493.83	11.53	2.87
Residue	20	856.19	42.81		
Total	24	2,831.52			

DF = Degrees of Freedom; SS= Sum of Squares;MS=Middle Square.

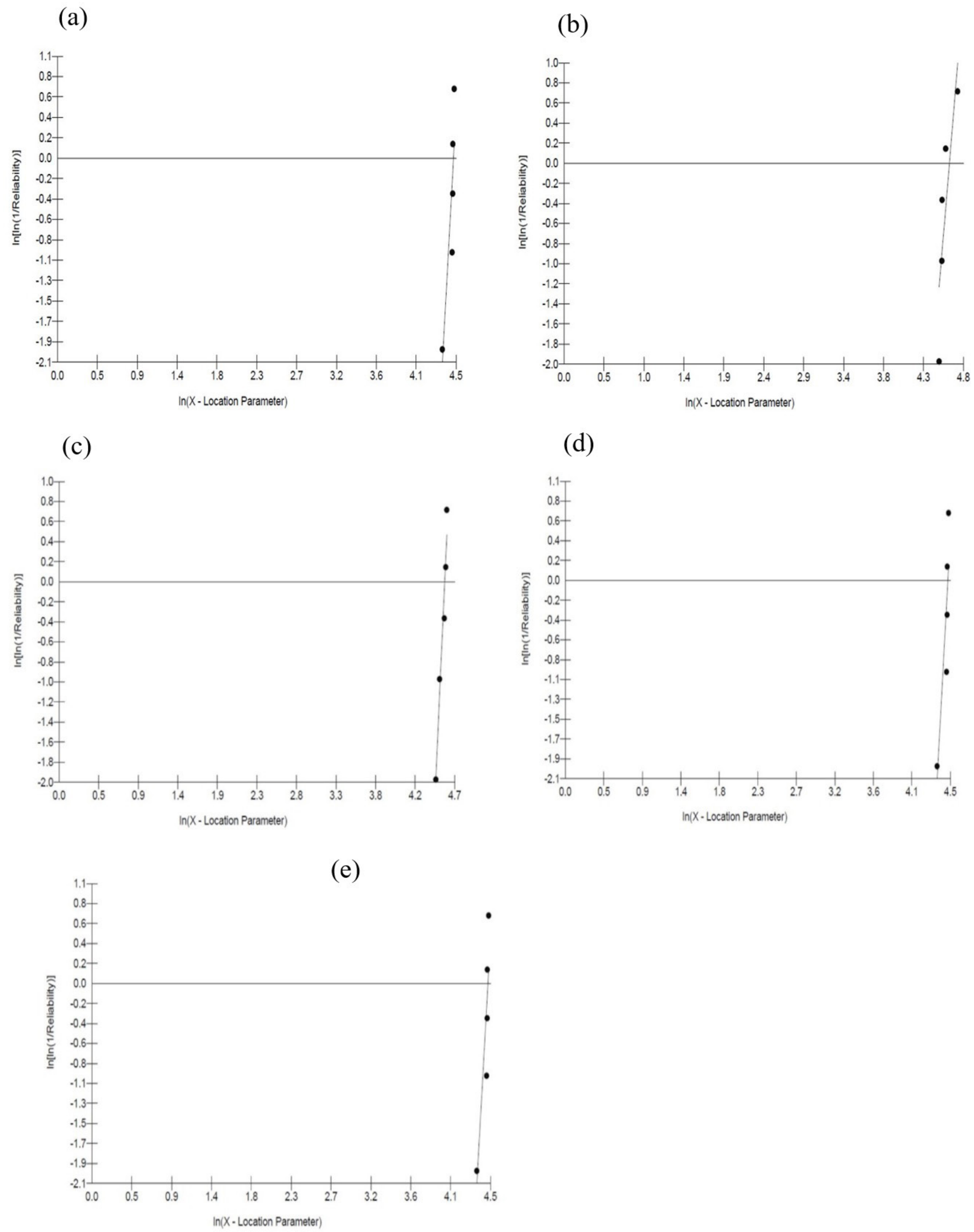

Figure 3. Weibull plot for energy absorbed in the residual velocity test. (a) Epoxy; (b) 10TVF; (c) 20TVF; (d) 30TVF; (e) 40TVF.

According to the values obtained by the ANOVA represented in Table 5, the hypothesis the averages are equal with a confidence level of 95% is rejected, as $F_{calc} = 11.53$ is greater than $F_{critical} = 2.87$. Therefore, the volumetric fraction of TVFs present in the composites had an effect on the absorbed energy during the test. In order to identify which volumetric fraction of fibers actually showed better-absorbed energy results, the Tukey test was applied to compare the means. Table 6 presents the honestly significant difference (HSD) obtained via Tukey's test.

The calculated HSD was 12.38 and differences above this value are considered significant. The values in Table 6 reveal that 10TVF, 20TVF and pure epoxy composites are indistinguishable from each other and 30TVF and 40TVF composites follow the same standard. Thus, it can be inferred that, although the V_L calculated for the 30TVF and 40TVF composites are very close to those previously discussed in the literature (Table 3), the fibers provided a weakening of these samples in relation to the other groups. Among the reasons for this occurrence, the possible low fiber strength, which needs to be better studied, can be attributed to the poor fiber/matrix adhesion, this is often caused by moisture, or even pressing with excessive load during the manufacture of the plate, which can generate microcracks in the material.

3.3 Scanning electron microscopy (SEM)

Figure 4 presents the micrograph of the longitudinal section of the TVF. The SEM image is able to detect microscopic features referring to the surface of lignocellulosic fibers such as roughness and the presence of defects such as micropores of around 10 µm, voids, depressions and cracks.

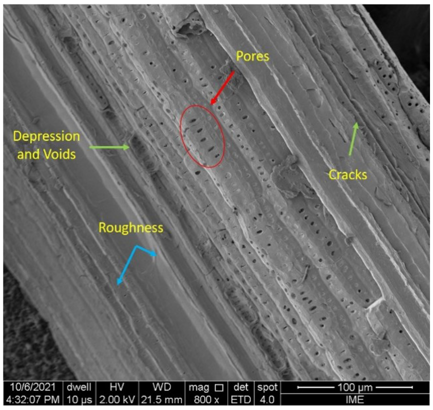

Figure 4. SEM of the longitudinal section of the TVF (800x).

After the ballistic test, the fractured samples were also analyzed by SEM to verify the fracture mechanisms acting during the impacts. Figure 5 shows the fracture surface of composites reinforced with 10 – 40 vol% TVFs.

Analyzing the fracture surface of the 10TVF composite (Figure 5a), a completely brittle type of fracture can be observed, due to the presence of river marks on the surface impacted by the projectile. Furthermore, in this sample, the cracks propagated in a catastrophic way, compromising the integrity of the plates. For the 20TVF composite, it was possible to identify a mechanism not reported in the previous sample (10TVF), which was an intralaminar fracture of the fiber (Figure 5b). In this type of fracture, delamination occurs following the direction of the fibers in the same layer [28].

Although the 30TVF and 40TVF composites showed lower levels of energy absorption, these plates revealed a more integral and dimensionally stable fracture surface, inferring that the fibers acted efficiently as a barrier to the propagation of cracks. In addition, the presence of a distinct fracture surface was observed, exhibiting more complex failure mechanisms such as interfacial detachment and fiber pullout (Figure 5c-5d).

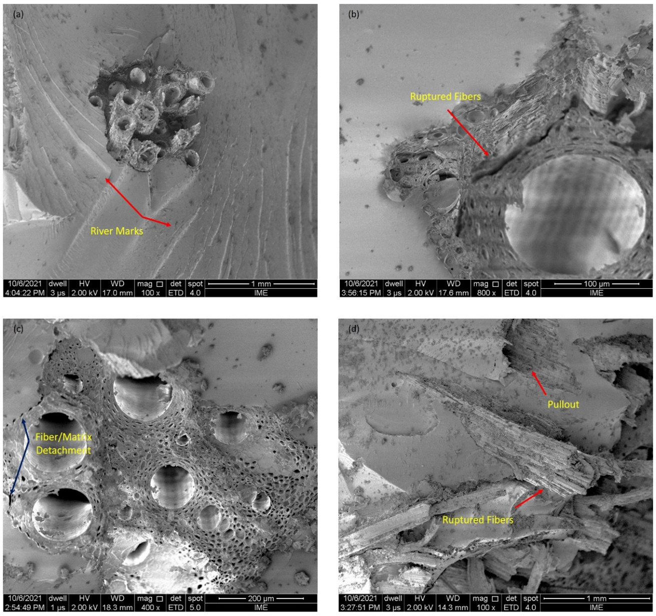

Figure 5. SEM images of fractured samples: (a) 10TVF, 100x; (b) 20TVF, 800x; (c) 30TVF, 400x; (d) 40TVF, 100x.

Table 6. HSD as measured by Tukey test for the absorbed energy of composites with volume different fractions

	Epoxy	10TVF	20TVF	30TVF	40TVF
Epoxy	0	7.41	10.28	20.47	24.52
10TVF	7.41	0	2.87	13.06	17.10
20TVF	10.28	2.87	0	10.19	14.24
30TVF	20.47	13.06	10.19	0	4.048
40TVF	24.52	17.10	14.24	4.048	0

4 Conclusions

- This research studied for the first time the possibility of using titica vine fiber-reinforced polymer matrix composites for ballistic application. The fracture mechanisms, the impact absorption capacity, in addition, the integrity of the composites after ballistic tests against a .22 LR caliber ammunition were investigated.

- The ballistic test showed a drop in energy absorption for composites with 30 and 40 vol% TVF, a fact confirmed by statistical analyses. On the other hand, composite plates with 0 – 20 vol% TVF shattered and/or presented their dimensional integrity easily compromised, which is not suitable for ballistic armor. Despite this, the composites with higher fractions of TVFs obtained limit velocity values close to or even slightly higher than what has already been found in the literature.

- Regarding SEM analyses, it was identified different fracture mechanisms acting on composites with 10 – 40 vol% TVF. It was possible to verify the type of brittle fracture in the 10 vol% samples, associated with the presence of river marks and the propagation of catastrophic cracks. From the 20 vol% composites, the trend changed to ductile fracture, showing the presence of more complex mechanisms such as fiber/matrix detachment, pullout and fiber rupture.

Acknowledgements

The authors would like to thank the Brazilian agencies CAPES, CNPq, FAPERJ and FAPEAM for their support.

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Received: 29 Jun. 2022
Accepted: 27 Jun. 2023