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POLYESTER COMPOSITES REINFORCED WITH MALEIC ANHYDRIDE-TREATED FILAMENTS FROM MAUVE

MESQUITA, R. G. A.; CESAR, A. A. S.; MENDES, L. M.; MARCONCINI, J. M.; TONOLI, G. H. D. Polyester composites reinforced with maleic anhydride-treated filaments from mauve. **CERNE**, v. 24, n. 1, p. 1-8, 2018.

HIGHLIGHTS

The untreated mauve-reinforced composites had the superior mechanical performance.

Chemical treatments acted positively for just water absorption.

The mauve filaments showed a high potential to be used in composites, some times with values better than fiberglass composites.

ABSTRACT

The objective of this study was to evaluate the polyester-based fiber composites reinforced with filaments/fibers of mauve treated with NaOH and maleic anhydride. The chemical treatment of the mauve filament/fibers (3 cm of length) was carried out, first with NaOH, followed by maleic anhydride. The resin used was ortho unsaturated polyester with addition of catalyst peroxide methyl ethyl ketone. The composites were prepared with 15% filaments (w/w) and tested for water absorption, three-point bending, tensile and impact strength. Fiberglass composites were produced using the same methodology for comparison purpose. The results demonstrate the potential use of mauve filaments. For some properties, the mauve composites showed better mechanical properties than fiberglass composites (three-point bending and tensile). The treatment of the filaments had varied effect and did not improve the mechanical properties. However, for the water absorption was observed a reduction of 35% in composites with treated mauve filaments.

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INTRODUCTION

Composites can be defined as products made by the combination of two or more materials, which after mixing can still be clearly identified in its mass and additionally show superior properties than their isolated constituents. The increasing interest for composites by various segments is due to their good synergy in the interaction of their components, which offer better structural characteristics for certain applications in comparison to their single constituents, besides the application of fillers can reduce the production costs (Callister Jr, 2002; Raghu et al., 2010).

Vegetable fibers as reinforcement in polymer composites have attracted great interest in developing countries, due to factors such as their low cost, availability, energy savings, interesting mechanical properties, low abrasiveness in the processing equipment, low density, besides being renewable, non-toxic and non-pollutant (Macvicar et al., 1999; Soykeabkaew et al., 2009).

However, the major drawbacks in the use of those fibers in polymer composites are related to the polar and hydrophilic nature of the vegetable fibers, as well as their susceptibility to attack by fungi and bacteria. The high moisture absorption of vegetable fibers is a major obstacle, because it can result in the swelling of the fibers and it affects the dimensional stability of the composite. However, this problem may be minimized by coating the fibers with chemical species. The interaction or interface of the vegetable fibers with the polymer matrix is typically an important issue. However, some incompatibility problems can be mitigated by either modifying the fiber surface or adding compatibilizers (Benítez et al., 2013; Garkhail et al., 2000; Mesquita et al., 2017; Pasquini et al., 2006; Sawpan et al., 2011). Improving the compatibility by changing one or both of the components can develop stronger and more durable bonds (Rowell, 2000). This is a paper that contributes to this fast moving field with new and useful information about the application of vegetable fibers toward products with improved performance, and to their changes promoted in the microstructure of the fiber-reinforced plastics. Furthermore, the effect of mauve fiber morphologies and chemistry on the performance of the unsaturated polyester based composites was rarely reported in the literature. Margem et al. (2015) investigated the effect of mauve incorporation (up 30%) in an epoxy matrix. But only the flexural behavior was investigated. According those authors the composites modulus of elasticity (MOE) increased according the mauve incorporation up 30%. This was due the MOE of the mauve fibers (8.8 GPa) higher than the that of the epoxy matrix (2 GPa).

Since Brazil is a country characterized by agro-based economy and produces different fibers, the present study sought to contribute for the need of obtaining biomaterials that can replace synthetic materials commercially used in several industrial sectors. The development of composites reinforced with these vegetable fibers can contribute to the sustainable development of the plastic composite sector, besides to contribute for the social development, since a lot of fibers are produced by family farms. Those issues were also raise by Leão et al. (2006) as well the availability of mauve in Brazil. Therefore, the objective of this study was to evaluate the physical and mechanical properties of plastic composites reinforced with mauve fibers.

MATERIAL AND METHODS

Materials

The plant used was mauve (*Urena lobata* L.) because of its availability in North region of Brazil. The fiberglass used was the kind E. The mauve filaments and the glass fibers were cut with scissor to 3 cm of length which filament.

The polymer matrix used here was composed of ortho unsaturated polyester resin. Methyl-ethyl-ketone peroxide was used as catalyst (both donated by Fibrasil Industria e Comercio Ltda., Brazil).

Mauve fibers characteristics

The mauve filaments (with 3 cm of length) were macerated to individualize the fiber cells. Measurements (length - L, width – W and lumen diameter - ϕ) of the individual fibers were performed using an Olypus BX41 microscope, with the aid of Wincel Regent PRO software. Each of the morphological features was measured in at least 30 fibers. Anatomical terms describing the fibers followed by the International Association of Wood Anatomists (IAWA, 1989). Some parameters were calculated for individual fibers such as flexibility coefficient and wall fraction (Fonseca et al., 2013; Paula; Alvez, 1989; Paula, 1993). The flexibility coefficient (FC) is given by the ratio between the fiber lumen diameter and the fiber cell diameter, expressed in percentage. The wall fraction (WF) is the ratio between cell wall thickness and cell radius, expressed in percentage.

The chemical analysis (lignin, extractives and ash) were conducted according to the Brazilian standards: extractives according M3/69 (ABTCP, 1974a); Klason lignin (free of extractives) according to M70/71 (ABTCP, 1974c); and ash according to M11/77 (ABTCP, 1974b).

Chemical treatments

The mauve filaments/fibers were treated with a solution of sodium hydroxide (NaOH) at pH = 12 for two hours. This alkaline treatment was carried out to promote two effects on the fiber/filaments: increase the surface roughness resulting in better interfacial adhesion and increase the amount of cellulose uncovered on the fiber surface, thus increasing the number of free hydroxyl (OH) groups exposed as possible reaction sites (Li et al., 2007; Valadez-Gonzales et al., 1999). After that first treatment the filaments were subjected to maleic anhydride as reported in the literature (Cantero et al., 2003). The maleic anhydride/filaments mass proportion was 10%. Maleic anhydride was dissolved in acetone (ratio 25:1 v/m). This mixture was heated in a water bath at 50°C for 24 hours. Subsequently, the material was washed with ethyl alcohol and acetone.

Production of the composites

The composites were prepared by mixing solutions of ortho unsaturated polyester resin with the catalyst (2%) that later were sprawled on the mauve filaments and glass fibers. It was incorporated the maximum mauve filaments proportion in to the composites, that was 15% (w/w). The same proportion was used to glass fibers. This was due to the lower density and high volume of mauve fibers relative to glass fibers. The filaments were poured into steel-framed molds and the resin was sprawled greased with silicone to facilitate de-molding. After curing the specimens were de-molded and conditioned (22±2°C, 65%±5% RH) until constant weight was achieved. The de-molded was carried out after 6 hours. The different steps in preparing the composites with treated and untreated fibers are shown schematically in Figure 1.

Properties and characterization of the composites

An EMIC DL 3000 universal testing machine was used in the tensile and bending tests. Tensile strength was determined following the ASTM D638-01 standard using around eight replicates, a test speed of 5 mm/min and load cell of 3000 kgf (ASTM D638-01, 2003). Bending tests were performed using a three point bending apparatus as recommended by ASTM D790-00 (2000), standard, using test speed of 2 mm/min and load cell of 50 kgf. The modulus of elasticity and the bending strength were calculated.

The Izod impact tests were performed using a Tinius Olsen with a hammer of 2,82 J. The tests were carried out in accordance with ASTM D256-10, (2010), standard using ten replicates.

Water absorption was performed by the immersion of the composites in water, in agreement with ASTM D570-98 (1998), procedure 7.4, long-term immersion (saturated).

The morphologies of the fracture surfaces of the composites after tensile tests were observed in a scanning electron microscope (SEM) Zeiss/DSM 940A t operated at 15 kV. Fracture surfaces were gold coated before SEM visualization.

The physical and mechanical properties were analyzed as a completely randomized design, with the completion of the analysis of variance and when significant the Scott-Knott ($p < 0.05\%$) test average. The results were processed with the aid of SISVAR software.

RESULTS AND DISCUSSION

Fiber characteristics

Table 1 shows the anatomical properties of the individualized fibers from mauve used in the composites.

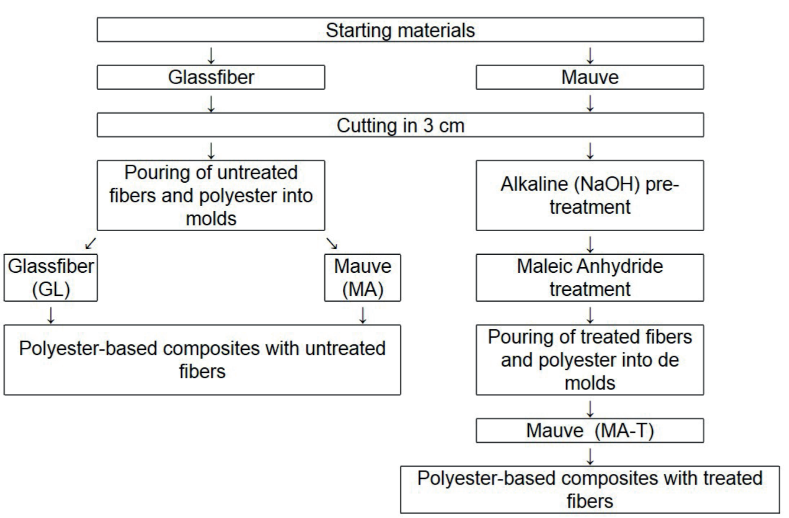

FIGURE 1 Illustrative scheme of the treatments and fiber-reinforced composites.

TABLE 1 Morphological characteristics of the individualized fibers.

Fiber	L (µm)	ϕ (µm)	T (µm)	W (µm)	FC (%)	WF (%)
Mauve	3238.1	5.1	5.8	16.7	30.6	69.4

Where: L = fiber cell length, ϕ = lumen diameter, T = cell wall thickness, W = fiber cell width, FC = flexibility coefficient, and WF = wall fraction

When working with polymer composites, the cells width (W) determination is important to calculate the aspect ratio, which is associated with the definition of the critical fiber length. Long and narrow fibers are preferable because they confer higher mechanical properties to the composites (Guimarães Jr et al., 2010). Mauve fibers have higher length and lower width compare to others natural fibers, also tested in polyester composites, such as pine, *Eucalyptus* and sugarcane bagasse (Mesquita et al., 2017).

The wall fraction (WF) is related to the rigidity of the fiber cell wall and values above 60% are normally related to stiffer fibers (Guimarães Jr et al., 2010). Mauve fibers have higher WF (69.4%) compare to others natural fibers, also tested in polyester composites, such as pine (26.7%), *Eucalyptus* (55.9%) and sugarcane bagasse (47.3%) (Mesquita et al., 2017).

The Table 2 presents the chemical mauve fibers characteristics used. Mauve present low content of extractives, lignin and ash, thus the holocellulose content is higher and, since the results of the composites are highly dependent on the chemical fibers properties, which explains the high values of mechanical properties for the mauve composites presented in the next section. However, a high amount of holocellulose also contributes to increase the water absorption due the amount of hydroxyl groups present in cellulose and hemicelluloses (Li et al., 2011b).

TABLE 2 Chemical composition of the mauve fibers.

Material	Extractives*	Lignin**	Ash*
		%	
Mauve	0.91	12.67	0.95

*based on total fiber mass; **based on extractives free mass

Physical-mechanical properties of the composites

The Figures 2 to 4 present the mechanical properties of the composite formulations. The composites reinforced with mauve without treatment were those with the highest values for most properties. The highest mean values of the modulus in the tensile and bending tests of the composites reinforced with mauve are probably related to the wall fraction (WF) of the fibers, since this property is related to the stiffness of the lignocellulosic cells. Another important factor that contributed to the good performance of the composites reinforced with mauve is the amount of cellulose, which is responsible for the strength of the fibers. These fibers presented high holocellulose content. The lowest values of the mechanical properties for fiberglass can be related with the volume that these fibers held. The density of mauve fibers is lower, thus the fibers held the space (volume) better than fiberglass. The better homogeneity and distribution contribute for a better distribution of the efforts during the tests.

The treatment of the filaments with NaOH and maleic anhydride decreased average property values of impact strength, modulus of elasticity, bending strength and tensile modulus. Then the chemical treatment acted negatively to these properties and the most significant reduction was in bending strength. The bending strength of the interaction depends not only on the fiber/matrix cohesion, but also of the properties of the fibers/filaments. The chemical treatments possibly degraded the fibers and decreased the average values of their properties (Sinha and Panigrahi, 2009). This effect was also observed by Hundahausen et al. (2015) that modified fibers with Maleic anhydride. Those authors modified fibers using MA to produce medium density fiberboards (MDF). Because of the hydrolysis of polyssacharides and cross-linking of cell wall polymers, they observed a reduction in elastic modulus and bending strength in the MDF.

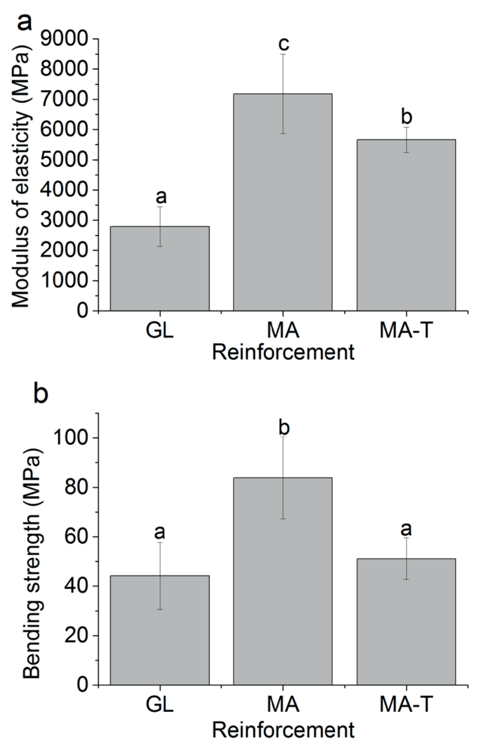

FIGURE 2 (a) Modulus of elasticity under bending and (b) bending strength of the composites reinforced with the different materials. Averages followed by the same letter do not differ by Scott-Knott test (α=0.05). Fiberglass composites (GL); untreated mauve composites (MA); and treated mauve composites (MA-T).

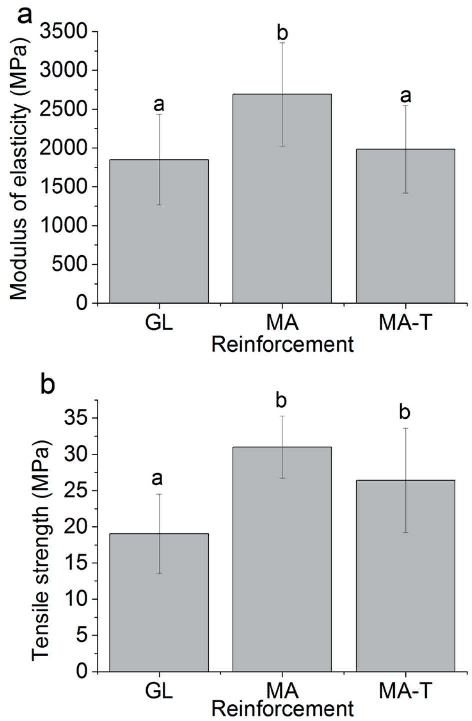

FIGURE 3 (a) Modulus of elasticity under tensile and (b) tensile strength of the composites reinforced with the different materials. Averages followed by the same letter do not differ by Scott-Knott test (α=0.05). Fiberglass composites (GL); untreated mauve composites (MA); and treated mauve composites (MA-T).

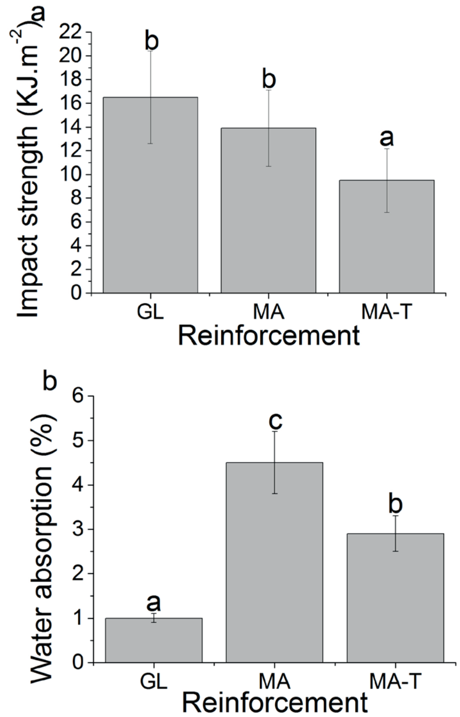

FIGURE 4 (a) Impact strength and (b) water absorption of the composites reinforced with the different materials. Averages followed by the same letter do not differ by Scott-Knott test (α=0.05). Fiberglass composites (GL); untreated mauve composites (MA); and treated mauve composites (MA-T).

Sinha and Panigrahi (2009) also observed a decrease of mean values for bending strength in polyester composites when the jute fibers were exposed to plasma 5 and 15 minutes. For the tensile strength property the composites with mauve treated and untreated do not showed difference. However the values of these composites were higher than the fiberglass composites.

Mishra et al. (2000) have studied the inclusion of sisal and hemp in Novalc resin (40 to 60% of fibers). The fibers were treated with maleic anhydride too. They observed that the maleic anhydride did not increase the properties, except the impact strength. Joseph et al. (1999) researched the inclusion of sisal (20%) with 3.5 cm of length in unsaturated polyester composites. They founded values of 31.8 MPa for tensile strength and 1950 MPa for modulus of elasticity (tensile), which are very similar to the ones observed in this study. The authors also observed that the values of these properties increase with the increase of fiber amount, but just until 50% of reinforcement.

The impact strength was affected negatively by the modification with maleic anhydride, however the mauve composites without treatment were statistically equal to those made of fiberglass composites. Wittawat et al. (2014) found different results using maleic anhydride to improve the interaction between sisal and rubber composites. They observed that the mechanical properties of these composites were improved with the treatment with maleic anhydride. However, in these cases they added the maleic anhydride in the matrix and this could protect the sisal from the degradation.

Water absorption (WA) by the resin is almost null, since it has a hydrophobic character. Then the hydrophilic vegetable fibers are largely responsible for the water absorption of the composite. The water absorption was the only property that was improved by treatment of the fibers. The treatment resulted in a reduction of 35% in water absorption. The composites of untreated filaments showed air bubbles and this helps to explain the higher water absorption. Likely, the treatment improves the interaction between matrix and fibers/filaments which explain the decrease in water absorption but at the same time the treatment degraded the fibers which explain the decrease in the mechanical properties. Hundhausen et al. (2015) modified fibers with MA to produce MDF, and they observed that the thickness swelling decreased in MDFs with modified fibers, however, the water absorption did not changed. They also observed that the internal bond was enhanced and the better performance of thickness swelling was attributed to the better gluing of the boards and the increase of surface area, but not to the hydrophobization of the fibers.

The pre-treatment with NaOH also helps to explain the decreased in WA. The mauve fibers/filaments showed a high amount of holocellulose (Cellulose and hemicelluloses). Hemicelluloses have lower molecular mass, are branched, are amorphous and have a lot of hydroxyl groups, but they are soluble in alkali (D'almeida, 1988). Probably part of those hemicelluloses were degraded and contributed to decrease the WA. The water absorption in the fiberglass composites is due the formation of air bubbles and cracks.

Microstructure of the composites

The SEM images showed a very weak interaction between filaments and polymer matrix (Figures 5 to 7). It was observed that the filaments were not fractured. Voids were observed in the interfacial region between the matrix and filaments, which is the consequence of a low interaction between them, probably due to the hydrophobic matrix combined to the hydrophilic mauve fibers. The SEM images showed a concentration of fiberglass in some points of the composite and this contributed for the lower performance of this composites (Figure 4).

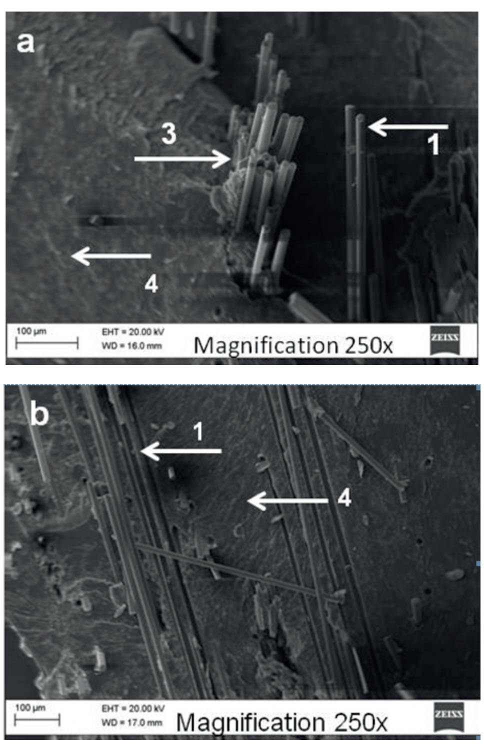

FIGURE 5 SEM micrographs of the fracture surface of the composites reinforced with fiberglass. (a) 250x magnification (GL); (b) 500x magnification (GL). Arrows indicate: (1) fiberglass, (2) presence of air bubbles, (3) surface left by the pull-out of the fiber (4) resin.

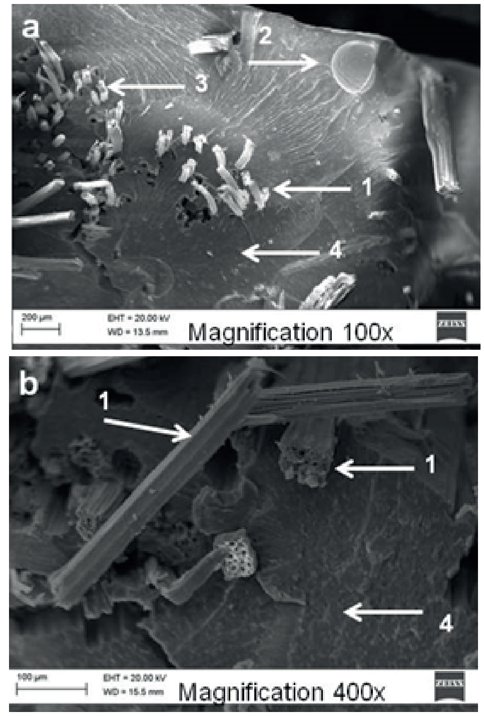

FIGURE 6 SEM micrographs of the fracture surface of the composites reinforced with mauve untreated. (a) 100x magnification (MA); (b) 400x magnification (MA). Arrows indicate: (1) mauve fibers, (2) presence of air bubbles, (3) surface left by the pull-out of the fiber (4) resin.

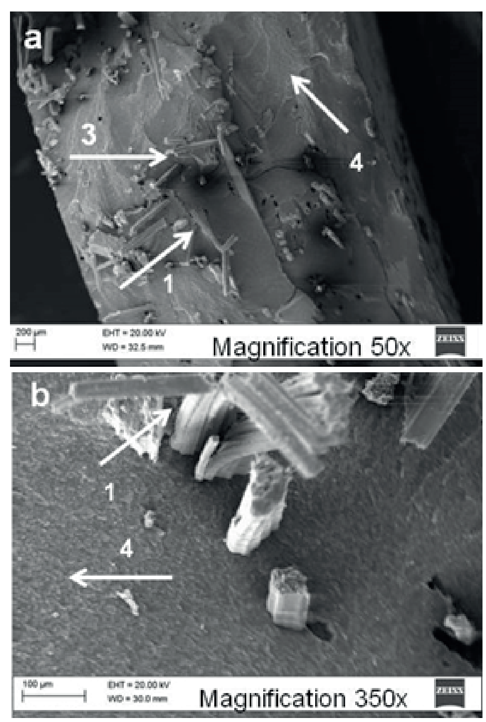

FIGURE 7 SEM micrographs of the fracture surface of the composites reinforced with treated mauve. (a) 50x magnification (MA-T); (b) 350x magnification (MA-T). Arrows indicate: (1) mauve fibers, (2) presence of air bubbles, (3) surface left by the pull-out of the fiber (4) resin.

CONCLUSION

Among the composites analyzed, the untreated mauve-reinforced composites had the superior mechanical performance (bending and tensile). The improved performance is probably due to the strength of the mauve fibers (which contains higher percentage of cellulose and fibers with higher proportion of wall fraction), besides they held better the composite due the lower density and higher volume.

Chemical treatments (NaOH and maleic anhydride) acted positively for just water absorption. Thus, the maleic anhydride treatment should be used when the physical properties of the composites are more important than the mechanical properties.

The mauve filaments showed a high potential to be used in composites, some times with values higher than fiberglass composites. New research should be carried out with a less aggressive maleic anhydride treatment (lower concentration and treatment time).

The present work contributes to the widespread use of the vegetable fibers for development of composites that will be studied for multi-purpose applications.

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