Agave lechuguilla Torrey Fiber as Reinforcement of Polyester Resin

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This study was aimed to evaluate the application of lechuguilla fiber (*Agave lechuguilla* Torrey), with no treatment, as reinforcement of polyester resin. Mechanical assays and pull-out tests were performed on specimens of one fiber and strands of 10 fibers and on lechuguilla fiber/polyester composites and polyester matrix alone. The results indicated that there was no important improvement in composite rigidity, as the Young's modulus in both the fiber and the polyester resin were similar; however, an important improvement in composite resistance, in respect to the matrix alone, attributed to a good interfacial adherence fiber-matrix, was observed. POLYM. COMPOS., 32:1601–1606, 2011.

INTRODUCTION

For years, natural fibers have attracted strong attention as an ecological option for the reinforcement of thermoplastic or thermoset polymers. Species like sisal, henequen, or jute were integrated into polymer matrixes to produce composite materials with notable results [1–5]. An outstanding advantage of natural fibers over synthetic fibers is that they are usually byproducts of some industrial process, what makes them low cost inputs. Natural fibers are also recyclable, biorenewable, and biodegradable resources; they are low density and low abrasiveness materials, and unlike glass fiber or asbestos, for instance, they are not as shocking to human health. However, owing to its hydrophilic nature (high polarity), natural fibers are poorly compatible with low polarity polymers; thus, chemical modification or some other strategy must be taken to improve compatibility [6–10]. It is worth mentioning that the usage of natural fibers in the automotive industry has increased considerably, as they are lightweight and display the mechanical properties that this industry requires for certain applications [11, 12].

At present, important efforts are invested in research aimed both to look for natural fibers to be used as reinforcers of polymer matrixes and to study adherence of natural fibers with polymer matrixes [13–17]. Lechuguilla (*agave lechuguilla* Torrey), for instance, is a plant that belongs to the family of *agavaceae* and grows in the arid and semiarid regions of Northcentral Mexico (*zona ixtlera*). Lechuguilla fiber has been used for centuries and represents the financial support of several communities in these regions [18, 19]. Nowadays, the plant of lechuguilla is mainly used in the handcraft confection of ropes and textiles (*ixtle*). However, literature shows applications with much higher scientific and technologic value, i.e., in obtaining cellulose derivatives, extraction of saponins, as reinforcing material or as absorbent of heavy metals [20–23]. Programs for the preservation of this specie have also been reported [19]. In this article, we report the study of interfacial compatibility between lechuguilla fiber and polyester resin. We intend to provide basic information about the use and performance of this fiber as the reinforcing material in a thermoset composite, to help in giving an optional application with high technological value to this plant.

EXPERIMENTAL

Materials

The lechuguilla fiber used in this research is native to Tamaulipas State, Mexico. The fiber was acquired either in the form of fiber strands or as a textile. Polyester resin industrial degree (catalyzed with cobalt naftate) was used as the matrix.

Methodology

Fiber microstructure was analyzed using a scanning electron microscope (SEM) Jeol JSM5800. The samples

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were prepared, first, by encapsulating individual fibers into epoxy resin and second, by subtly polishing the resin tablet to obtain a perpendicular sharp image of the fiber.

Fiber morphology (macrostructure) was also an important aspect to be considered during experimentation, because it will provide information about the scattering in dimensions and in geometry. Measurements were performed by using the software Image-Pro Plus 4.1 and an Olympus SZH10 microscope. Certain amount of fiber samples of 1.0 cm long was cut from fibers with similar diameters. From this bunch, 150 of them were selected randomly. The diameters at both ends and at the center were registered, and a statistical analysis was performed. The 450 registered data (150 $\times$ 3) were ordered in growing form; each measurement was divided by 450 and the result of every division was the value of the F function for each sample. A plot of cumulative distribution of the average diameters versus diameter was built. This study helped to know the probability $F(x)$ to select randomly a fiber with diameter lower than x.

Force versus displacement assays were performed to study rigidity and mechanical resistance of the single fibers and the fiber strands. To perform this evaluation, on the one hand, specimens of one fiber $\sim$ 40 cm long and similar diameter (visually determined) were selected. On the other hand, fiber strands of 10 fibers were prepared as follows: first, a hook of steel was coupled to a mechanical stirrer and then one end of the strand was tied to it, whereas a knot was done in the other end. All fiber strands were twisted at a speed of 45 rpm to control the number of turns. Specimens with 10, 20, 30, or 40 turns were prepared. It is worth mentioning that cardboard strips were used to protect the ends of the specimens of damage caused by the clamps of the tension machine. The assays were run in a universal tensiometer Instron model 4469.

To evaluate interfacial adhesion fiber/matrix, specimens of one fiber and fiber strands of 10 fibers were prepared. Specimens for pull-out assay were prepared using molds of polyethylene of 12 mm diameter, with a perforation $\sim$ 1 mm in the middle of the base. The perforations were implemented to align the fiber (or strand) in the center of the mold. The polyester matrix was prepared by mixing 200 g of base resin with the amount of catalyst (cabalt naftate) recommended by the deliverer. The mixture was stirred for 2 min and poured into the molds, where the fiber or the fiber strand was previously placed. The specimens were allowed to settle for 2 days at room temperature. After this period, the specimens were recovered from the mold.

To elaborate the fiber/polymer composites, a handmade textile (handcraft product) of lechuguilla fiber was used. Due to the traditional processing procedure, the number of fibers in each strand of the textile varied typically from 12 to 20 fibers. Consequently, the strand diameter was variable too. The textile also presented a lot of empty space, so, to improve reinforcement, strands of 10 fibers of 30 cm long were used to fill in only one direction. Therefore, the reinforced direction of the textile was 2.2 times denser than the transversal direction; that is, the obtained composites contained 38% in volume of fiber in the reinforced direction, whereas only 17% in volume in the transversal direction.

Lechuguilla fiber/polyester composites were prepared in square molds of polyethylene of 15 cm $\times$ 10 cm. The polyester matrix was prepared as indicated previously. First, the textile was placed on the mold base and then the resin was poured until the textile was covered, $\sim$ 0.5 cm depth. After that, with the help of a brush the resin was impregnated on the textile (no extra procedure was applied). The matrix was allowed to cure for 2 days at room temperature; after this time, the composite plaques were recovered from the mold and machined for the elaboration of specimens for mechanical assay.

RESULTS AND DISCUSSION

Morphology

Fiber diameter is an important parameter to be considered in composite design, because composites mechanical behavior could be importantly affected. Lechuguilla fiber morphology, by nature, presents a progressive diameter reduction from one extremity to the other. Thus, in this study, fibers with similar diameter were selected to elaborate the specimens for all the evaluations. Fig. 1a illustrates the

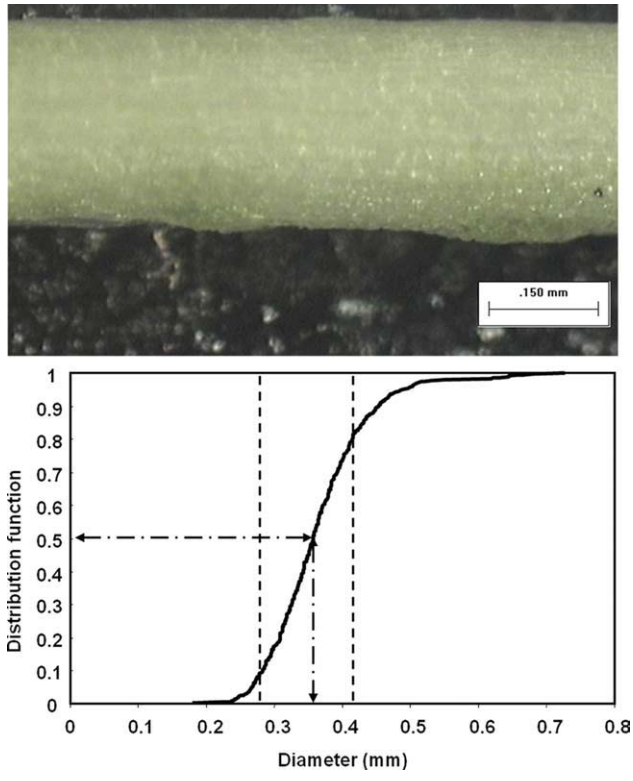

FIG. 1. (a) Morphology of a lechuguilla fiber and (b) cumulative distribution of the average diameters versus diameter [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com].

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morphology of a lechuguilla fiber; as can be seen, the diameter in transversal direction is not uniform, and its surface is rough.

The curve of average diameters versus diameter obtained from the statistical study is illustrated in Fig. 1b. This study indicated that (a) the diameters were found into an interval from 0.180 to 0.727 mm, with most of the diameters between 0.28 and 0.42; (b) the average value was 0.36 mm, with a standard deviation of 0.074 mm, and (c) the diameter distribution displayed a sigmoidal function ("S" shape). The average value was in agreement with the diameters reported for other natural fibers [24, 25].

Fiber microstructure was analyzed by SEM. Fig. 2a depicts a SEM photograph of the transversal section of a lechuguilla fiber. As it is observed, the fiber is elliptical with a distinctive longitudinal split. An enlargement of the image is illustrated in Fig. 2b; this image indicates that each fiber is, in turn, constituted by polygonal microfibers with three to eight sides clearly observed.

Mechanical Assays on Fibers and Streaks

Fig. 3 shows the plot of force versus displacement for a specimen of one fiber. The motif of using force in this

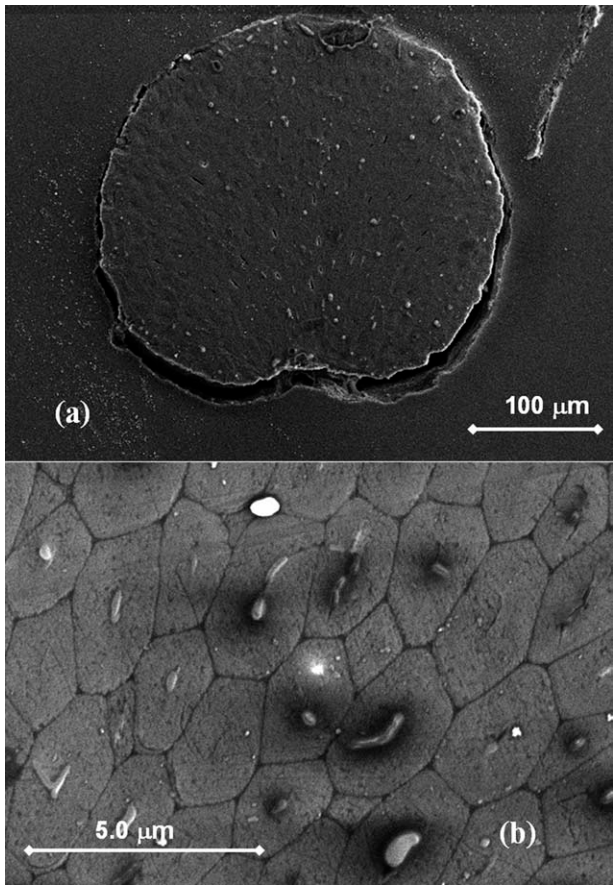

FIG. 2. SEM micrograph of (a) the transversal section of a lechuguilla fiber and (b) the microstructure of a lechuguilla fiber.

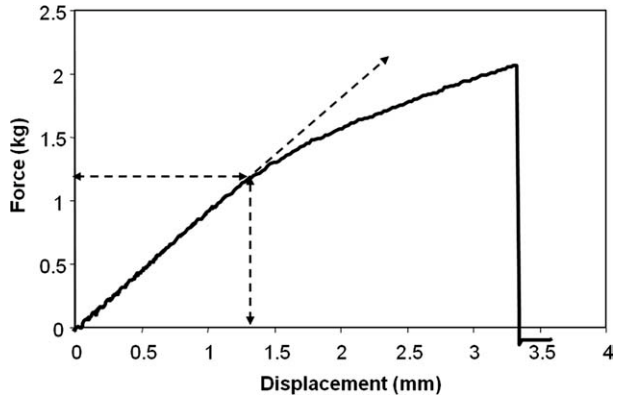

FIG. 3. Plot of force versus displacement (tension test) for a specimen of one fiber.

plots was based on the fact that lechuguilla fibers, like most natural fibers, are irregular filaments, that is, their diameter reduces (or increases depending on the view point) longitudinally from one end to the other. Therefore, the concept of stress (σ = force/area) appeared not to be completely valid, as stress is normally applied for probes with regular dimensions. The term displacement [D = (final length − initial length) × 100], even though it was calculated in a similar sense as strain {γ = [(final length − initial length/final length)] × 100}, was used to avoid confusion with the typical stress–strain plots. The curve shows an elastic behavior at small displacement ~ 1.26%, and immediately after, a nonlinear section likely due to viscous phenomena. Young's modulus and material resistance are commonly obtained from these assays; however, due to fiber irregularity, as mentioned above, it was considered that rigidity and maximum force were more representative parameters of the fiber; nevertheless, the values of Young's modulus and resistance were also reported. It is worth mentioning that all evaluated specimens produced curves with similar form as the shown in the figure.

Rigidity, K, of a fiber was determined from the data of force, F, and displacement, D, in the elastic region by means of *Eq. 1*:

$$K = \frac{F}{D} \tag{1}$$

As fiber rigidity is inversely proportional to its length, and the evaluated fibers had different lengths, the global rigidity per length unit of fiber, K_m, for every fiber was calculated from *Eq. 2*:

$$K_m = \frac{F}{\text{strain}} = \frac{F}{\frac{D}{L}} = K * L \tag{2}$$

where L is the length of the fiber.

The average values and standard deviation of K_m, and maximum stress, F^{max}, for the 98 performed measurements are reported in Table 1. To get an idea of the

TABLE 1. Physical properties of lechuguilla fiber.

Property	Average	Standard deviation
Strength (kg)	1.97	0.56
Young's modulus (GPa)	7.39	2.22
Deformation (%)	6.84	0.031
Tension resistance (MPa)	184.26	52.43
Global rigidity (N/mm) × mm	772.14	233.76
Moisture absorption[a] (%)	55.16	–
Length (mm)	129.02	8.58
Density[b] (g/cm^3)	1.79	–
Diameter (mm)	0.36	0.07

[a] Samples of fibers were dried for 24 h at 110°C, later on the fibers were immersed in water at room temperature for 24 h, then the fibers were retired from the recipient, and the excess of water was removed. The fibers were weighed, and the reported value is the average of 10 measurements.

[b] Density was determined by using an Ultrapicnometer 1000 Quantachrome instruments. The reported value is the average of 15 measurements.

magnitude of the "apparent Young's modulus" and the "apparent resistance," *Eqs. 3* and *4* were used:

$$E = \frac{K_m}{\frac{\pi d^2}{4}} \tag{3}$$

$$\sigma^u = \frac{\frac{F^{max}}{\pi d^2}}{4} \tag{4}$$

where d is the average diameter of the fiber, E is the Young modulus and σ^u is the maximum stress.

Results of fibers physical characterization are shown also in Table 1. It was found that lechuguilla fiber tension resistance (184.26 MPa) was lower than the resistances reported for other fibers like sisal (511–635 MPa), hemp (345–1035 MPa), or ramie (400–938 MPa). Similarly, lechuguilla's Young's modulus (7.39 MPs) was also lower than that of other fibers like jute (26.5 GPs), hemp (27.6 GPs), or sisal (9.4–22.0 GPs). However, the maximum deformation, 6.8 (%), was higher than the reported for other natural fibers [1, 25].

TABLE 2. Mechanical properties of strands of 10 fibers as function of the number of turnings.

Specimen turns/30 cm	Global rigidity per unit length, K_m (N/mm) × mm		Strength, F^{max} (kg)	
	Average	Dispersion	Average	Dispersion
10	1227	23	10.51	2.05
20	1695	218	20.34	1.17
30	1207	56	17.09	1.69
40	924	59	14.24	1.07

Mechanical evaluation was achieved on 18 strands of 10 fibers each and with different number of turns. The purpose of this evaluation was to determine the effect of torsion over fiber mechanical performance. Fig. 4 shows the plots of force versus displacement for a strand with 30 turns/30 cm long. At the very beginning, the behavior was similar to the observed at the probe of one fiber. The first transition could be understood as the breakage of the first fiber. After that, the strand continued monotonically resisting load until the second breakage or transition presented. At the third breakage, the maximum strength was reached and immediately after, the load decreased and new breakages appeared as the displacement continued. Load decrement was attributed to the fact that some fibers did not support the load more and broke, which caused untwisting of the broken fibers and rigidity got lost. The mechanical properties determined from the strands were: global rigidity per unit length, K_m, which was calculated by using *Eq. 2* and streak resistance, defined as the maximum force, F^{max}, that the strand resisted. The average results and standard deviation of K_m and F^{max}, for the strands with different turns (10, 20, 30, or 40 turns/30 cm long) are shown in Table 2. The twisting that produced the greatest resistance was 20 turns/30 cm long; thus, this twisting was considered the optimum to prepare the strands. It is important to mention that at higher number of turns, the torsion produced fiber fragility due to excessive deformation and friction.

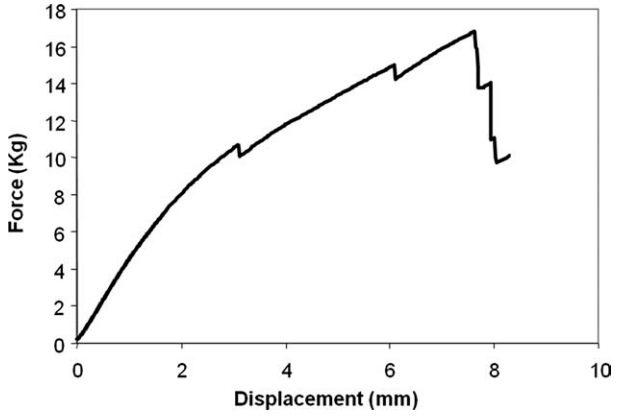

FIG. 4. Plot of force versus displacement (tension test) for a strand of 10 fibers.

FIG. 5. Plot of force versus displacement (pull-out test) for a strand of 10 fibers.

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TABLE 3. Pull-out assays for specimens of one fiber and strands of 10 fibers.

	Interfacial resistance (kg/mm)		Critical length (mm/kg)	
Specimen	Average	Dispersion	Average	Dispersion
1 fiber	0.21	0.06	5.17	1.93
Strand (10 fibers)	2.57	0.69	0.42	0.14

Pull Out Assays on Fibers and Streaks

A curve of pull-out assay for a strand is illustrated in Fig. 5. As can be seen, when the load was applied different stages of slipping occurred until the final fall of the load. Interfacial fiber-matrix resistance corresponds to the relationship between slipping strength and matrix height (in this case, 7.38 mm). The results of this assay are shown in Table 3. The critical length, described as the relationship between specimen height and interfacial resistance, are also included in Table 3. The critical length can be understood as the maximum height that the matrix should have for it not to unstick from the fiber if strength of 1.0 kg was applied.

Mechanical Assays on Lechuguilla Fiber/Polyester Composites and Polyester Matrix

Curves of stress versus strain for the matrix alone and for lechuguilla fiber/polyester composite are shown in Fig. 6. As observed, the reinforced polyester presented an increase of 50% of resistance in respect to the matrix alone. It is worth saying that the maximum deformation to bring about composite fail was about 2.5%, meanwhile the maximum deformation that the matrix stands was 0.5%, that is, materials flexibility incremented. It should be pointed out that during the evaluation several fibers torn and ruptures occurred, which were attributed to the good fiber-matrix interfacial adherence.

It was clear during mechanical assay that composite resistance was provided by the fibers. If we imagine we have the fibers alone with no matrix, the expected

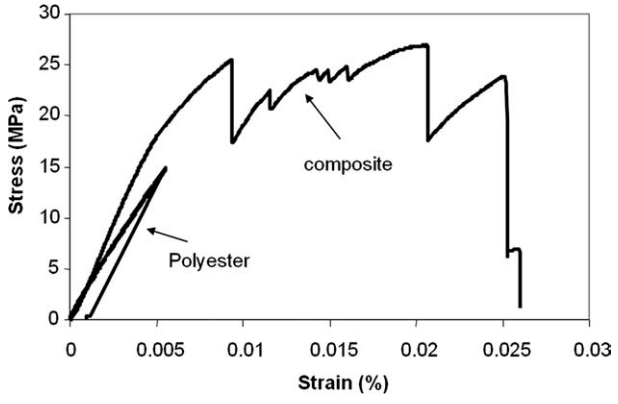

FIG. 6. Plot of stress versus strain for pure polyester resin and lechuguilla fiber/polyester resin composite.

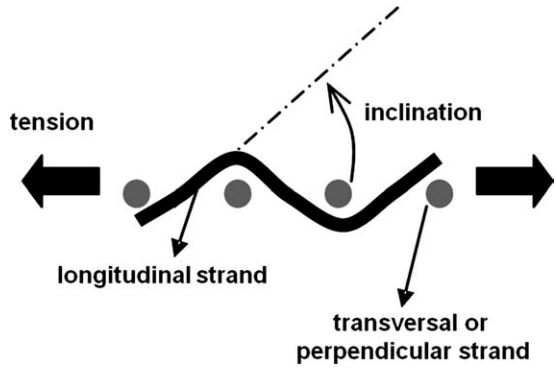

FIG. 7. Physical arrange of the lechuguilla fiber fabric used to reinforce the polyester resin.

composite resistance would be 0.38 $\times$ fiber resistance $\approx$ 70 MPa, as there is in the composite 38% of reinforcement in the assay direction. However, the resistance was only about 30 MPa. This could be due to the high diameter variability of the fiber in the textile; this way, the high amount of thin fibers contributes poorly to the textile resistance. Also, the fibers in the textile were not twisted in the same way as in the strands, which could have influenced friction among the fibers and, as a consequence, textile resistance. Besides, streaks in the textile are not perfectly aligned along specimen tension direction, as they have to incline to pass through the perpendicular strands, as it is illustrated in Fig. 7. To obtain the expected value (70 MPa), a much better control of fiber diameter, twisting tension and lower inclination of the strands in the textile, are needed. The function of perpendicular strands is, on the one hand, to keep together the group of longitudinal fibers and on the other hand, to reinforce the matrix in transversal direction, which will be considered in future study.

CONCLUSION

According to the obtained results, it can be concluded that lechigulla fiber/polyester composites will present very similar rigidity as the polyester resin matrix, because fibers Young's modulus is only 7 GPa, whereas the matrix has 2 GPa. Hence, there will not be matrix reinforcement from the point of view of rigidity. However, from the point of view of resistance, and assuming good interfacial adherence fiber-matrix, there will be resistance reinforcement, as fiber resistance is $\sim$ 9 times higher than matrix resistance. Thus, lechuguilla fiber can be considered as a good candidate for the reinforcement of polymer composites.

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