ISSN: 2277-3754
ISO 9001:2008 Certified
International Journal of Engineering and Innovative Technology (IJEIT)
Volume 4, Issue 8, February 2015

Characterization of Sisal Fibers for use as Reinforcement in Polymer Composites

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Abstract*— In this study, we assessed the physical, mechanical and micro structural properties of sisal fibers. The technical feasibility of the manufacture of polymeric composites reinforced by these fibers, randomly arranged, was analyzed, using the lowest possible level of technological processes in the production stages, aiming to produce a technically feasible composite. It was used a terephthalic polyester matrix pre-accelerated with cobalt naphthenate and cured with peroxide methyl ethyl ketone (MEK) in the ratio of 0.33% volume resin at room temperature. The sisal fibers were obtained in the city market of Belém, Brazil, and hand cut in lengths of 5, 10 and 15 mm, being used the way they were extracted without surface treatment. The composites were made by manual molding without pressure and at room temperature. Test specimens of composites reinforced by sisal fibers were manufactured accordingly to ASTM 638, varying the fibers length (5, 10 and 15 mm) and fixing the mass fraction in 5% of fibers within the composite. The mechanical properties of sisal composites were evaluated by tensile tests and the fracture surfaces generated were analyzed by scanning electron microscopy (SEM) in order to correlate the fracture aspects with the mechanical properties. The results of the sisal fibers and composites characterization were satisfactory; there was an increase in the composites tensile strength with the increasing fiber length within the polymer matrix. The composites with 15 mm length sisal fibers showed the best mechanical performance among the manufactured series. The predominant failure mechanisms were pull out and detachment of the matrix fibers for lower resistance composites; and the disruption of the matrix fibers for composites of higher strength.

***Index Terms*—Reinforcement, sisal fibers, polymeric composites.**

I. INTRODUCTION

In the past years, it can be seen an increasing growth in the number of researches related to the development of materials that combine distinct properties. Composite materials then arose, which are progressively gaining ground in the market. Among the composites that present the best properties are the polymeric composites reinforced with synthetic fibers. However, the use of these fibers in polymeric composites is associated to an increased wear of the equipment, high processing costs and high density. In a world where the climatic changes due to the industrial activity are the major environmental problem, the possibility of using natural fibers as substitutes to the synthetic fibers, such as glass and carbon, is an environmentally correct solution. The natural vegetable fibers, for being plentiful, of low environmental impact when discarded and presenting mechanical, physical and thermal properties suitable for industrial applications, are becoming attractive alternatives from the economic and environmental points of view. In the past, the lack of knowledge about these vegetable fibers structure and properties limited their use. With the research advancements, the vegetable fibers use field has been growing and, thereby, the knowledge of these fibers structure and properties, which will help the production of new products and new applications. In the selection of new vegetable fibers for research, it is necessary to know their composition and structure, as well as the important characteristics to their performance, as length, resistance, color, density, and others [1]. The use of vegetable fibers is not new. It probably dates back to the beginning of our civilizations, when straw or grass was used to reinforce raw bricks-clay. However, the treatment and the way to rationalize their use have changed over the years. Until the 60's decade, the vegetable fibers were quite utilized, mainly in the automotive industry. With the advent of synthetic fibers, they were practically replaced during the 70's and 80's decades. In the past few years, the energetic crisis, the low degree of industrialization and the environmental problems caused by the use of synthetic fibers have again attracted the attention and interest of researchers all over the world for their utilization [2]. It can also be highlighted a natural vegetable fiber that presents great potential, the sisal fiber. In the semi-arid Northeastern Brazil, including the driest arid part, which corresponds to little more than 10% of the region area, practically there are no phytotechnic options, having only the sisal as the basic element of income production and distribution in the countryside [3]. Another important aspect of sisal fiber is its mechanical properties. Currently, numerous works have been developed aiming to incorporate the sisal fibers in polymeric matrices, generating fibrous compounds. The sisal fiber is promising in the development of composite

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materials due to its low cost, good mechanical properties and availability in the market [4]. The sisal fiber relative price has been about 11% of glass fiber prizes and 0.07% of carbon fiber prizes. It also presents advantages over other vegetable fibers if comparing mechanical performance and unit cost [5]. Among the polymeric matrices used for the composites production, the polyester matrix has been excelled due to its mechanical properties, low viscosity, high wet ability and low cost. Unsaturated polyesters have diversified properties and extremely versatile uses, and have been used as a fixed popular term for polymeric matrix in composites. The polyester is widely industrially produced and presents a lot of advantages if compared with other thermosetting resins, including the ability of cure at room temperature, good mechanical properties and transparence [6]. In this context, this work proposed to characterize the sisal fibers and use them as reinforcement in the development of polymeric composites, characterizing the tensile strength and micro structural analysis of the generated fractures. It also aimed to verify the possibilities of applications of these materials as liners, partition walls, lining cars and other applications of this kind.

II. MATERIALS AND METHODS

- ***MATERIALS***

✓ ***Polyester resin***

The polymer used in this work development was unsaturated terephthalic polyester resin in the pre-accelerated form, produced by Royal Polímeros under the commercial name of Denverpoly 754. The cure agent was MEK peroxide (Butanox M-50), in the ratio of 0.33% (v/v). The resin was obtained already pre-accelerated with cobalt naphthenate (CoNap), in the mass ratio of 0.15%.

✓ ***Sisal fibers***

The sisal fibers from the Agave sisalana specie were obtained in the city market of Belém, Brazil, and cut in three (3) different lengths: 5 mm, 10 mm and 15 mm. The fibers were utilized the way they were obtained, without surface treatment and under room conditions. The desired lengths were achieved by hand cutting (with scissors) from the fiber bundles. A hundred (100) fibers were removed, without surface treatment, as samples to perform the experimental procedures.

- ***METHODS***

✓ ***Sisal fibers characterization***

The sisal fibers tensile strength, length, diameter, elongation, superficial aspect, specific mass and moisture content were characterized. The fibers tensile strength assays were performed on a KRATOS Universal Testing Machine, model IKCL3, with data acquisition system with a load cell of 5 kN, adopting speed of 5 mm/min and measuring useful length between clamps of 15 mm. A hundred sisal fibers samples were tested, in order to give credibility to the results. The sisal fibers samples were prepared with KRAFT paper supports, called TAB in the literature. The TABs are used to uniformly distribute the charge applied on the fiber that is being tested and also to protect the fiber from the damages for occasional positioning of the clamps in the testing machine. The KRAFT paper TABs (basis weight of 200 g/m²) with dimensions of 25 mm X 65 mm were glued with cyanoacrylate (Super Bonder from Loctite) on the fibers useful length ends, according to ASTM D 3822 [7]. Complementarily to the tensile test, was determined the fibers length and average diameter, by optical microscopy, admitting the fibers with circular cross-section, and three measurements were carried out over the longitudinal direction of the hundred sisal samples. The fibers microstructure was analyzed using scanning electron microscopy (SEM), where was verified its superficial aspect and cross-section from the tensile tested samples and samples embedded in acrylic matrix and prepared according to the metallographic procedures. For the determination of the fibers specific mass was utilized the pycnometer method using water as non-solvent, so that the material was immersed in water and the displaced volume observed, according to DNER-ME 084/95 [8]. The determined average value for the sisal fibers specific mass was 1.42 g/cm³. The fibers moisture content was determined by drying the fibers in a Quimis stove, model MD 1.4. It was determined the fibers moisture content on wet basis of three samples with initial mass of 3 grams. The fibers were initially weighed and dried until reach a constant mass, determining the moisture content by the equation (1).

$$T\,\% = \frac{M_1 - M_2}{M_1} * 100 \quad (1)$$

Where (T%) correspond to the percentage of moisture content, (M_1) to the initial mass before drying, and (M_2) to the final mass after drying.

✓ ***Preparation of test specimens***

The tests specimens were prepared by manual molding using silicone molds, without release agent or pressure. The composites were produced from the optimized matrix according to the procedure performed by [9], who made particulate composites. Pre-weighed amounts of resin, cure agent and, in this work case, sisal fibers, were mixed in a becker, being homogenized for about five minutes and leaked at room temperature in the molds. The mass fraction of each type of reinforcement used in the production of the test specimens in this research was defined by the mold volumetric capacity to accommodate the reinforcement without pressure or compression, and without the matrix. The mass fractions were fixed in 5% for the composites with fibers of 5 mm, 10 mm and 15 mm length. The fibers were randomly placed inside the molds.

✓ ***Tensile tests***

The test specimens were produced from the silicon mold in the minimum number of ten for the tensile tests for matrices reinforced with sisal fibers. The composite tensile tests were performed on a KRATOS Universal Testing Machine, model IKCL3, with data acquisition system with a load cell of 5 kN, adopting speed of 5 mm/min and measuring useful length

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ISO 9001:2008 Certified
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between clamps of 60 mm. The tensile tests were performed accordingly to ASTM D 638M [10].

III. RESULTS AND DISCUSSION

✓ ***Sisal fibers characterization***

The sisal fibers tensile strength, elongation, length, diameter, specific mass, moisture content, superficial aspect and microstructure were characterized. The results are shown in Table I.

Table I. Non-treated sisal fibers characterization

Material	Tensile Strength (MPa)	Elongation (%)	Diameter (mm)	Specific Mass (g/cm³)	Moisture Content (wet basis) (%)
Sisal Fiber	453.62 (± 91.98)	5.5 (± 2.02)	0.250 (± 0.032)	1.42 (± 0.01)	13.10 (± 0.5)

The results shown in Table I are either on the average or above the results found by other researchers [11]. The sisal fibers tensile properties were 51% higher than the average of the values found by [5]; 61% higher than the values obtained by [4]; 48% higher than the values found by [12]; 40% higher than the values of [3]; 22% higher than the results obtained by [13]; and 32% higher than the values found by [11], evidencing the great variability of the natural fibers mechanical properties. The Table II shows a comparative between the sisal fibers physical and mechanical properties found in this work and the results found by other researchers who used sisal fibers in their works.

Table II. Comparative study between the results of sisal fibers physical and mechanical properties found in this work and the results found by other researchers who used sisal fibers

Author	Diameter (mm)	Specific Mass (g/cm³)	Tensile Strength (MPa)	Elongation (%)
THIS WORK	**0.250 (± 0.032)**	**1.42 (± 0.01)**	**453.62 (± 91.98)**	**5.5 (± 2.02)**
[5]	0.486	1.591	218.3 (± 51.3)	7.07 (±3.95)
[4]	0.194	-	176 (± 63.36)	2.2 (±0.04)
[12]	0.223	-	234.30 (± 75.10)	-
[3]	0.223	-	268.11	2.08
[13]	0.205 (± 4.3)	1.41	350 (±7)	6 – 7
[11]	0.260 (± 0.49)	1.48	304.55 (± 115.73)	5.80 (±1.97)

The sisal fibers micro structural characterization was performed using scanning electron microscopy (SEM), showing the micro structural peculiarities of the fibers outer and inner surfaces. Fig. 1 presents the results.

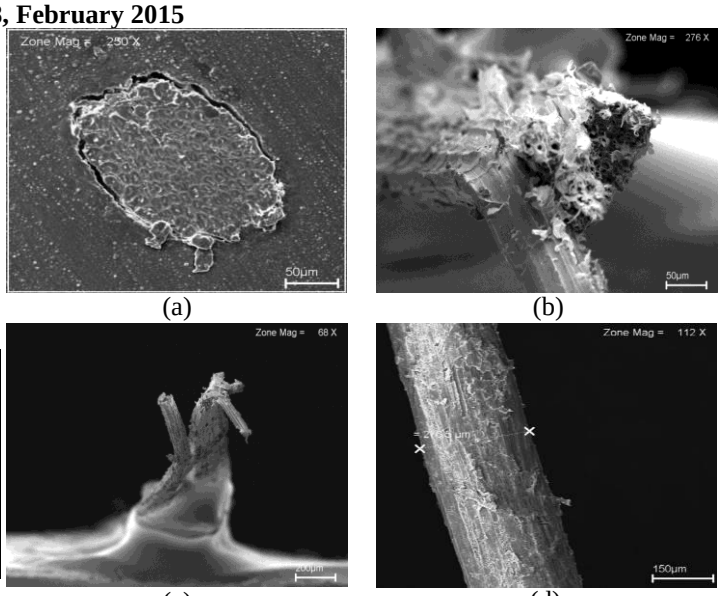

Fig. 1. Scanning electron microscopy: (a) Cross-section of a sisal fiber embedded in acrylic matrix presenting elliptical shape; (b) Sisal fiber presenting with voids between the microfribils bundles; (c) Fracture region aspect of a sisal fiber after tensile assay, illustrating the disruption of the elementary fibers; (d) Sisal fiber surface aspect, showing irregularities and small relief on the surface.

Fig. 1 (a) shows sisal fiber embedded in acrylic matrix presenting elliptical shape, thus proving why the fiber was used in circular shape during the tensile assays, measuring the fiber diameter. Fig. 1 (b) shows the sisal fiber containing voids between the microfribils bundles, that is a vegetable fiber typical feature. Fig. 1 (c) presents the fracture region of a sisal fiber tested after tensile assay, evidencing the disruption of the elementary fibers. Fig. 1 (d) illustrates the sisal fiber superficial aspect, showing irregularities and small relief on the surface. The sisal fibers moisture content showed that the initial moisture was 13.10% in wet basis. The drying curve of the sisal fibers at 105 ± 5° C, shown in Fig. 2, obtained by the drying of 3 grams samples, demonstrates that the sisal fibers have initial moisture within the level of other vegetable fibers, such as curauá composite and jute, already used in polymeric composites [11]. Therefore, the sisal fibers moisture content is good for their use in polymeric composites. The sisal fibers microstructure analyses were carried out at Scanning Electron Microscopy Lab (LAMBEV) from the Geosciences Institute of Universidade Federal do Pará (UFPA). The equipment used was a SEM, model LEO – 1430. The analysis conditions for the secondary electrons images were: electric beam current = 90 µA, constant-acceleration voltage = 10 kV, work distance = 15 – 12 mm. Their superficial aspect and cross-section were verified from samples which had been tensile tested and samples embedded in acrylic matrix and prepared according to the metallographic procedures. Fibers under room conditions, with initial moisture of 13.10% and without drying or surface treatment, were used to produce the composites.

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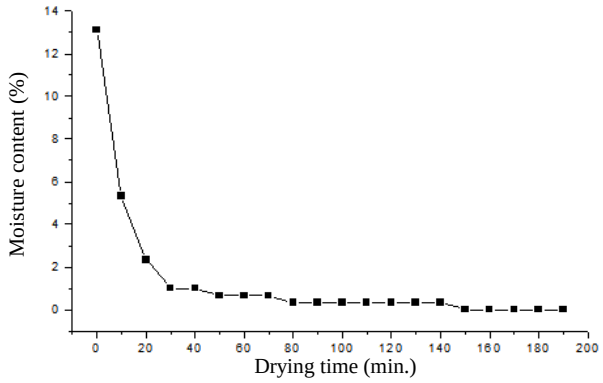

Fig. 2. Drying curve for the sisal fibers at 105 ± 5° C.

✓ ***Composites reinforced with sisal fibers***

Table III shows the results obtained in the tensile tests for the polyester composites reinforced with 5, 10 and 15 mm length sisal fibers with their respective mass fractions of fibers.

Table III. Tensile tests results for the polyester composites reinforced with 5, 10 and 15 mm length sisal fibers, randomly arranged

Reinforcement	Reinforcement Mass Fraction (F_M) %	Tensile Strength (σ) (MPa) Average (Standard Deviation)
5 mm length sisal	5	16.98 (± 1.37)
10 mm length sisal	5	22.78 (± 1.72)
15 mm length sisal	5	27.05 (± 1.49)

Fig. 3 presents a comparative between the results obtained with increasing length of sisal fibers. The data indicate that the resistance of the composite produced increased with increasing fiber length, confirming what has been reported by other researchers [4]. The composite showed significant improvement of its mechanical properties with the increasing fiber length: the composite reinforced with 15 mm length fibers achieved mechanical performance 38% higher than the composite reinforced with 5 mm length fibers, because, as already known, the greater the fiber length the lower will be the number of these fibers ends, and, thus, lower the possibility of crack nucleation in the composite, acting positively on the composite mechanical properties. It is also worth mentioning that the increasing fiber length improves the fibers alignment effect obtained in the composites. For the manufacturing process, the mixture fiber/matrix is leaked into the silicone mold by gravity and, for the tests specimens dimensions, there is a natural tendency of the fibers arranging in the longitudinal direction of the specimens, which makes that the tensile test be in the same direction of the fibers, improving, therefore, its mechanical strength. The 15 mm length sisal composites results were 40% and 20% higher than the results found for the banana and sisal composites, respectively [14]. The sisal composites results were 19% higher than those found by [15]. The sisal composites achieved similar results to those found by [16].

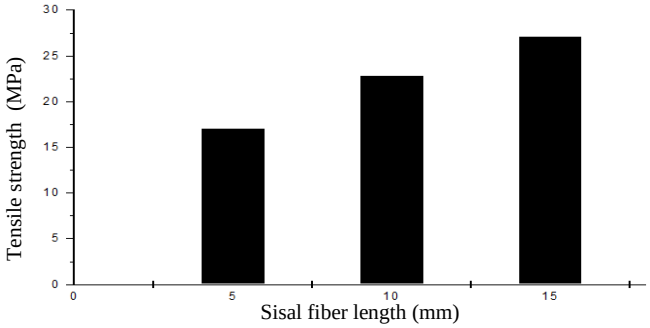

Fig. 3. Comparative results of the tensile strength tests for the composite of polyester matrix reinforced with sisal fibers.

Fig. 4 presents the behavior charge versus displacement of characteristic tests specimens of series of composites reinforced with sisal fibers. It can be seen it that the composites reinforced with 5 and 10 mm length fibers obtained very similar loading and elongation levels; and the composites reinforced with 15 mm length fibers obtained high loading and elongation levels, in the mechanical performance, showing that the composites reinforced with 15 mm length fibers achieved significantly higher results than those obtained with composites reinforced with 5 mm length fibers. This fact is explained by the increased values of charge and deformation using greater fiber length (15 mm).

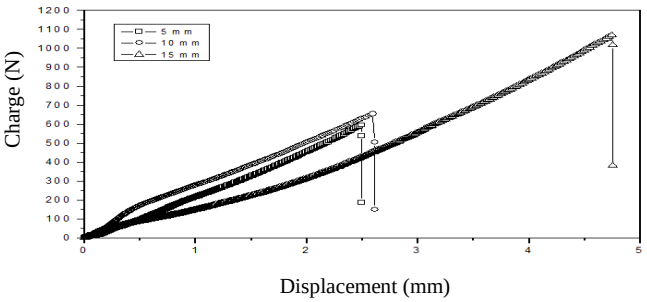

Fig. 4. Curves charge versus displacement, for tests specimens' characteristic of composites reinforced with sisal fibers series.

Fig. 5 shows the micrograph of a 5 mm length sisal fibers composite. It can be observed that pull out (white arrows) is the failure mechanism on the composite fracture surface, showing the weak adhesion between the fiber and the matrix.

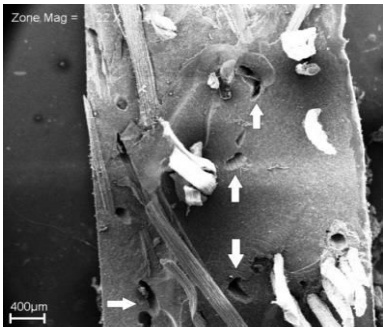

Fig. 5. Fracture surface of the polyester composite reinforced with 5 mm length sisal fibers. The white arrows indicate the fibers which pulled-out from the matrix.

ISSN: 2277-3754
ISO 9001:2008 Certified
International Journal of Engineering and Innovative Technology (IJEIT)
Volume 4, Issue 8, February 2015

Fig. 6 presents the fracture of a 10 mm length sisal fibers composite. It can be seen that the pull out (white arrows) is the main failure mechanism, but it can be noticed the presence of broken fibers (black arrows).

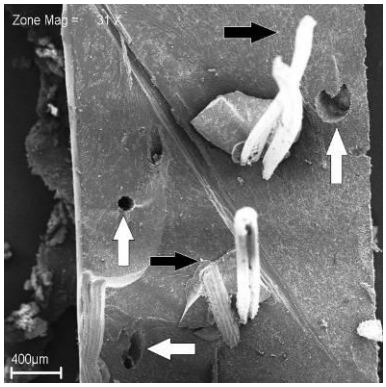

Fig. 6. Fracture of a polyester composite reinforced with 10 mm length sisal fibers. The white arrows indicate the predominance of matrix fibers pull out.

Fig. 7 shows the fracture of a 15 mm length sisal fibers composite. It presents a high predominance of broken fibers (black arrows), what improves the composite strength, proving a better adhesion between the fiber and the matrix.

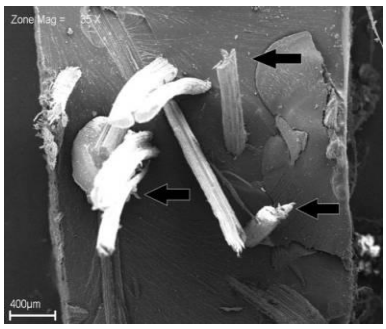

Fig. 7. Fracture of polyester composite reinforced with 15 mm length sisal fibers. The black arrows indicate the broken fibers.

IV. CONCLUSIONS

- The studied sisal fibers presented efficient mechanical, physical and microstructural features and properties, for the results were similar or higher than those found for vegetable fibers traditionally used in composites production.
- The composite reinforced with sisal fibers presented mechanical performance quite satisfying, despite the low mass fraction obtained with the processing technique used, presenting efficient mechanical properties for certain types of applications, such as partition walls, liners, lining cars and other applications of this kind.
- The increasing sisal fibers length improved the fiber composites tensile strength. The sisal composites obtained the best results for the 15 mm length fibers.
- The fractography methodology used to evaluate the mainly failures mechanisms on the composite materials by scanning electron microscopy was appropriate.
- The mainly failure mechanism on the composites with greater strength was fiber breakout (delamination). and on the composites with lower strength the mainly failure mechanisms were the pull out and fiber detachment from the matrix.
- Polymer composites reinforced with vegetable fibers are extremely important for the development of new materials for the various branches of industry such as construction, shipbuilding, automotive and other areas. Hence the importance of researching new vegetable fiber with potential reinforcing of composites that nature. Resulting in a material with good properties, low cost and environmental impact.

V. ACKNOWLEDGMENT

We thank CAPES for the postgraduate scholarship granted, and the Scanning Electron Microscopy Lab (LAMBEV) from the Geosciences Institute of Universidade Federal do Pará (UFPA). We also acknowledge the Postgraduate Program in Engineering of Amazonian Natural Resources (PRODERNA).

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