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MECHANICAL AND PHYSICAL CHARACTERIZATION OF THE NATURAL FIBER *LUFFA CYLINDRICA* FOR ITS POSSIBLE USE IN CONTACT SPORTS EQUIPMENT: 1ST STAGE

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ABSTRACT
Natural fibers are of general interest nowadays due to their promising physical and mechanical properties. Many industries make use of these, for example as protective equipment in the sports industry. Natural fibers like sugar cane, wood, hemp, flax or coir are easy to acquire and process, with cost benefits. This work aims to characterize *Luffa cylindrica* dog-bone test-pieces as received, with and without NaOH treatment, by tensile testing in order to evaluate its elongation by means of ANOVA and simple linear regression models, with the purpose of creating a composite with a recyclable thermoplastic copolymer in future stages.

INTRODUCTION
In everyday activities, objects are used with several purposes. These can be made from components or parts of various materials. Such materials come from four different sources: metallic, ceramic, polymeric and composites [1,2]. Depending on the function and requirement for its service or use, an object can be composed by any type of these materials processed under varied conditions which can provide unique characteristics [1,2]. Many of these characteristics are mechanical properties such as rigidity, toughness, hardness, brittleness, energy absorption, wettability, etc., and physical properties like rugosity, geometry, spatial distribution, etc. Thus, numerous mechanical behaviors may be present and can be explained by their atomic structure, micro and macro conformations, or other principles [3,4]. When creating an object as a composite, the possible conformations involve combinations of the three types of materials with one being the reinforcement and the other the matrix. This type of material makes it possible to have metal-metal, metal-polymer, natural-polymer, or even ceramics as matrices, that harness the best properties of both components [3,4].

The use of composites is widely known in the industry [5]. Nowadays, productive fields such as automotive, aerospace, sports, among others look to improve many parts of their products by focusing on better mechanical performance [6]. Much of this can be attained from different types of composites that differ in manufacturing methods for their obtention, and the type of materials that innovate their composition, but can be similar in desired behaviors [7,8]. Composites are mainly manufactured using synthetic materials [9], and despite their great properties, environmental concerns arise regarding the disposal of these when they finish their usefulness. Therefore, biocomposites are in need for replacing synthetic materials used in the aforementioned industrial fields. One of the solutions to this problem is the use of biodegradable material sources that contaminate less the environment. In this way, a natural material becomes a feasible option due their easy processing and cheap acquisition [9].

The natural fiber used in this work is the sponge gourd *Luffa cylindrica* or vulgarly known as scourer, also called “estropajo” or “pepinillo de esponja”; “esponja”; “estropajo”; “paste”, “quimgombo”; “buchados paulistas”; or “loofah”; in Colombia, Argentina, Mexico, Costa Rica, Venezuela, Brasil, and the United States, respectively. It is believed that it originated in Asia and was introduced to China in the year 6,000 BC, and years after to Egypt in the Middle Age. It can also be found in Africa and India. It is eaten as a gourd in Japan -when cultivated commercially since the 1890s or so-, and can be transformed into an exfoliative product in baths [10-13]. The natural fiber loofah comes from the family *Cucurbitaceae*, grows as a creeper, and can be seen nowadays being sown in tropical areas with high humidity and temperature such as coastal or riversides. In Colombia, and specifically in the department of Antioquia, its cultivation takes place on the countryside in farms where adequate conditions of soil and weather exist for making it a productive crop in a vivarium. Some articles have been written in Colombia about the economic aspects of this natural fiber [14,15].

Loofah fibers are multidirectional and interwoven, forming a mat or a mesh-like structure which can have a great variability in its width and compactivity [14]. Depending on the conditions where it is cultivated, they can be thinner and more compact when grown from 0 -300 meters above sea level, or thicker and less compact if cropped 300 meters above sea level [14]. Most of the studies aim to characterize chemically its composition [16, 20-22], and its mechanical characterization has based mostly in compression properties [17] or individual fibers [20-22]. Other studies focus on its promising use as a medicinal ointment or substance [18] or as a contaminant removal device [19]. There are no studies focusing on characterizing bundles of many fibers’ dumbbell test-pieces by tensile measurements.

For a composite, there are many variables that need to be considered. To join two materials of the same or different type, or even three materials, which are called hybrid composites [23], it is necessary to pay attention to two important aspects: the interface adhesion between the reinforcement and the matrix, and the surface characteristics of both materials. Hence, there are methods for modifying the surface of either the reinforcement or the matrix that are selected depending on their nature [24,25]. Many studies have used loofah as reinforcement with different thermosets or thermoplastics matrices [26,27]. These fibers also have improved cementitious materials [28], and have a great potential to be used in dynamic applications [29, 30].

The present work intends to characterize *Luffa cylindrica* dog-bone bundles in a simple and creative way, to tackle the high variability in its crops to indicate/predict any homogeneity or mechanical parameter. The cutting of loofah samples was based on their natural orientation in longitudinal and transversal ways, and dog-bone dimensions were based on ASTM D-638 for tensile properties of plastics. This approach for bundles, with or without chemical treatment, has not been reported in the literature so far.

The purpose of this work is to contribute with statistical data about tensile properties of luffa bundles, to possibly create polymeric composites that could as well biodegrade to some extent in future stages.

MATERIALS AND METHODS

Luffa Fibers

Samples of loofah cylindrica (also known as luffa) were obtained from the local market in the downtown area of Medellín, Colombia, shown in Figure 1. These come from small growing areas in the countryside, where harvesting of sponge gourds can be possible due to appropriate tropical weather conditions and terrain altitude mostly in the west and northeast of Medellin in the department of Antioquia, Colombia, South America, specifically in San Jerónimo and Bajo Cauca.

The test samples were made using a dog-bone steel mold shape with variable cross-sections of 20mm width and 13mm length approximately, as shown in Figure 2. Sixty (60) dog-bone test-pieces were cut by hand using scissors or bistoury over a red-pen-drawn dog-bone shape onto the loofah mats, Figure 2. The growth of loofah plantings and harvesting was not recorded nor controlled, and is far beyond the scope of this work.

Figure 1. a) *Luffa cylindrica* grown in Antioquia, Colombia and bought in a downtown market in Medellín, b) manual cut of the loofah sponge gourds.

Figure 2. a) Bone-shaped steel molds 20mm width x 13mm length, b) 60 plus hand cut test-pieces using the bone-like steel mold.

<u>Mechanical test</u>

<u>*Tensile strength testing.*</u> The tests were done using a Shimadzu SLBL traction machine with a 5kN load cell. Test-pieces were mounted using clamps crafted by the research group, shown in Figure 3. Speeds were 1mm/min and 50mm/min. Width and thickness for the cross-section area were measured without extensometer but with a manual and digital calibrator to all the test-pieces by the technician operator.

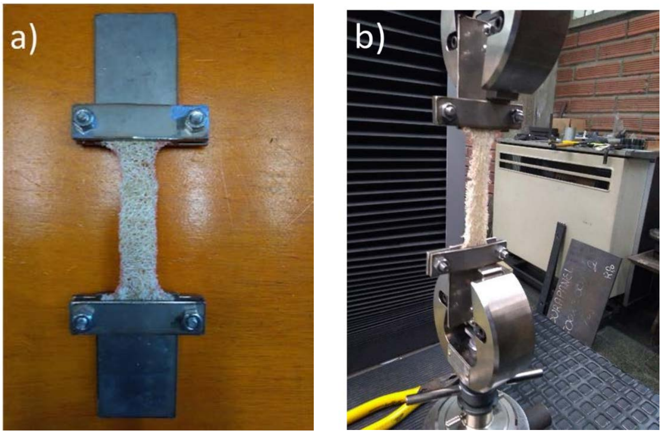

Figure 3. a) Steel clamps with loofah test-pieces, b) mounting of the configuration were rupture of the test-piece is seen.

Ultimate Tensile Strength (UTS) was obtained for all the test-pieces from their correspondent force vs. elongation/ stress vs. strain graphs organized in excel to undergo a proper analysis of the desired mechanical behaviors and how these can be explained.

<u>**Chemical treatment**</u>

The method applied to the longitudinal and transversal loofah test-pieces was chosen based on the most common use seen in the literature, and due economic issues because of scarce resources.

<u>NaOH treatment.</u> For this step, sodium hydroxide (NaOH) scales were bought from the local chemistry store Químicos JM. Thus, 20gr of NaOH were diluted in 1 liter of tap water in an attempt to copy industry conditions -this water is treated by the regional public services company EPM, and by the date of its use -according to the July report of 2018 in its webpage [32]- it followed all the global regulations for its human consumption. Then, loofah test-pieces were immersed in the 2%NaOH solution and left at room temperature for 2 hours. The treated samples were extracted with latex laundry gloves and a breathing mask to avoid the corrosiveness of NaOH and were washed immediately with DI H20, bought at the local chemistry store Químicos JM, by simply pouring neatly the DI water while rubbing each sample gently for about 10mins; no pH meter was used. After this, the test-pieces were left to dry at room temperature for 48 hours and stored in plastic bags for further mechanical tests.

Figure 4. a) Loofah test-pieces soaked in 2%NaOH for 2hours, b) subsequent DI H2O rinsing, c) drying at room temperature for 48hours.

Statistical analysis

All the data collected from the measurements of loofah test-pieces and tensile testing of untreated and treated loofah test-pieces was analyzed using the free-source statistics software R by applying descriptive analysis, ANOVA, and a simple linear regression model. The hypotheses are if the variables type of cut, speed, force, width, thickness, chemical treatment, and time are different, and therefore significative, for the resultant elongation variable.

RESULTS

Mechanical test

Tensile strength testing. The initial length of the test-pieces when fixed in the clamps was measured to the 20 treated test-pieces and to 20 untreated test-pieces with a measuring tape, to both longitudinal and transversal cuts. The initial length average for these was used for the remaining 20 untreated samples that were not measured when fixed in the clamps due to absentmindedness of both the researcher and the technician when adjusting these. The width and thickness of 18 untreated longitudinal and 18 untreated transversal test-pieces was measured with a manual caliber by the technician, and the remaining 4 untreated longitudinal and transversal plus the 20 treated test-pieces with a digital caliber.

The resultant graphs from the data given by the machine at two different speeds, 1mm/min and 50mm/min, show no uniformity concerning maximum force, elongation, stress, and strain (data not shown). In Figures 5 and 6 maximum elongation results are compared.

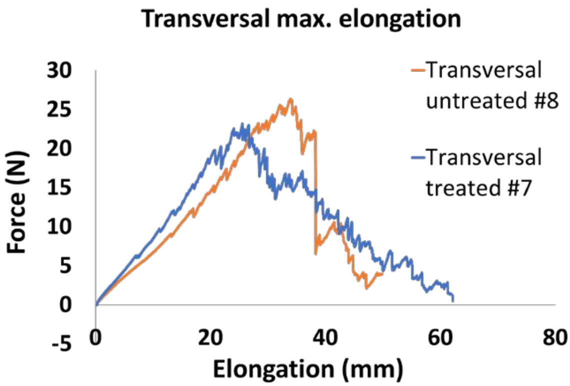

Figure 5. Comparison of the maximum elongation among the 30samples, treated and untreated, of transversal test-pieces of *Luffa cylindrica*. Testing speeds of 1mm/min for 6samples and 50mm/min for 14 samples.

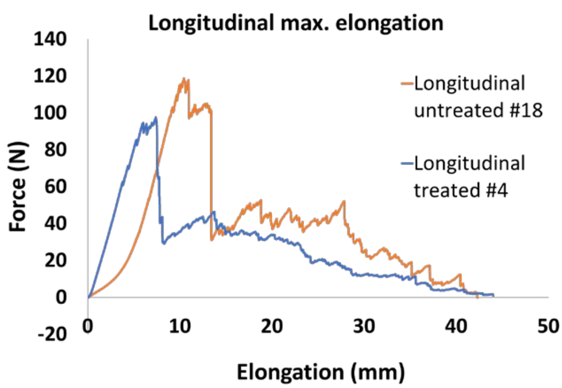

Figure 6. Comparison of the maximum elongation among the 30samples, treated and untreated, of longitudinal test-pieces of *Luffa cylindrica*. Testing speeds 1mm/min for 6 samples, and 50mm/min for 14samples.

<u>Statistical analysis</u>

A descriptive analysis is shown as boxplots in Figure 13, where it can be seen that the means are different.

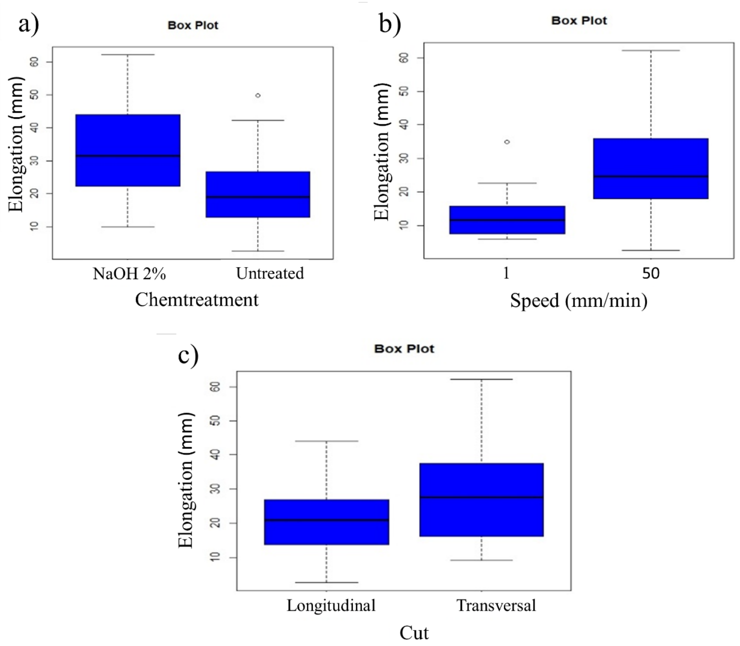

Figure 7. All 60 samples boxplots with bold horizontal lines as means: a) Chemtreatment (chemical treatment) results for the averaged 40 untreated and 20 treated samples; b) Cut (type of cut) results for 30 longitudinal and 30 transversal loofah samples (treated and untreated); c) averages for 12 samples evaluated at 1mm/min and 48 samples (untreated plus treated) evaluated at 50mm/min;

In order to conclude about the significative difference of the numerical (quantitative) and categorical (qualitative) variables for the dependent variable elongation, an ANOVA was applied to confirm such hypotheses. This methodology aims to explain variability of data. It bases on the F-value, which is the division of the explained variability (controlled treatments) over the unexplained variability (chance or uncontrolled elements proper to nature) [33]. The results are shown in Tables 1 and 2: the variable force was not significative as its F-value is below 1, indicating a higher variability within treatments than that among treatments; the variables cut (type of cut), chemtreatment (chemical treatment), speed, thickness, width, and time were significative as their F-value was above way above 1, indicating a lower variability within treatments than that among treatments-the initial length (numerical variable) was not included because of the lack of variability in its obtention when assuming an average equal to all 40 longitudinal and transversal untreated test-pieces instead of different values obtained for these with a measuring tape.

Table 1. ANOVA showing the numerical (speed, force, thickness, width, time) and categorical (cut, chemtreatment) variables for the response elongation with their respective significance asterisks.

Analysis of Variance Table

Response: elongation

	Df	Sum Sq	Mean Sq	F value	Pr(>F)
cut	1	890.83	890.83	16.9931	0.0001353 ***
chemtreatment	1	2111.89	2111.89	40.2856	5.427e-08 ***
speed	1	836.44	836.44	15.9555	0.0002049 ***
force	1	6.00	6.00	0.1145	0.7363995
thickness	1	864.39	864.39	16.4887	0.0001654 ***
width	1	753.80	753.80	14.3792	0.0003907 ***
time	1	2422.71	2422.71	46.2146	1.039e-08 ***
Residuals	52	2726.00	52.42		

Signif. codes: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1

Table 2. General simple linear regression model result showing the corresponding standard estimate coefficients for all the numerical and categorical variables considered for the elongation with their significance asterisks.

Coefficients:

	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	-18.51750	17.81511	-1.039	0.30342
cuttransversal	8.75390	3.32624	2.632	0.01115 *
chemtreatmentuntreated	-8.17575	2.91723	-2.803	0.00711 **
speed	0.63801	0.07654	8.335	3.77e-11 ***
force	0.02534	0.03422	0.740	0.46241
thickness	2.74534	1.28258	2.140	0.03703 *
width	1.22918	0.72990	1.684	0.09817 .
time	-117.25231	17.24773	-6.798	1.04e-08 ***

Signif. codes: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1

Residual standard error: 7.24 on 52 degrees of freedom

Multiple R-squared: 0.7431, Adjusted R-squared: 0.7085

F-statistic: 21.49 on 7 and 52 DF, **p-value: 2.696e-13**

An R^2 value of 74.31% corresponds to the linear model of elongation in equation (1):

$$\text{Elongation} = -18.51750 + 8.75390*\text{cuttransversal} - 8.17575*\text{chemtreatmentuntreated} + 0.63801*\text{speed} + 0.02534*\text{force} + 2.74534*\text{thickness} + 1.22918*\text{width} - 117.25231*\text{time} \quad (1)$$

DISCUSSION

Appropriate formulae for the maximum tensile strength, ductility, tear strength, elastic modulus, and resilience calculations of the graphs in Figures 5 to 12 will be made using mechanics of materials theory accordingly to data and graphs, and correspondent models to detect any tendency in the literature may be approached in further analyses.

The developing of a linear model may include all the variables, but the ones that are more significant may be left in it. This means that force and width may be removed from the equation as they do not vary sufficiently to model the elongation behavior. The use of these natural fibers is beneficial because of their availability in Antioquia, Colombia, as well as their biodegradability, thus being a potential resource for biodegradable composites. Further research is to be conducted in optimizing the manufacturing of environmentally friendly composites using additional natural fibers also from Colombia [29-31].

CONCLUSIONS

a. The curves for the four types of loofah test-pieces do not have a predictable pattern, and the resemblance of one another cannot be completely understood by looking only at the graphs.
b. The means for the qualitative variable "cut" showed a slight difference in their means according to the boxplot descriptive analysis and ANOVA results.
c. The means for the qualitative variable "chemtreatment" showed a clear difference in their means according to the boxplot descriptive analysis and ANOVA results.
d. The means for the quantitative variable "speed" showed a notable difference in their means according to the boxplot descriptive analyses and ANOVA results.
e. The most appropriate speed for the purposes of tensile measurement is 50mm/min as the rupture times (data not shown) fell into the range of the ASTM D-638 designations for Speed of Testing in plastics.
f. Elongation may be modeled linearly with such geometry and dimensions, direction of fibers, chemical used, and selected rate of traction for the loofah natural fibers obtained in Medellín.
g. The absence of the quantitative variable "initial length" suggests that if included, it could increase the fit of the linear model as most of the variables are significative, despite not following any ASTM norm exact specification.
h. The solids content of tap water was not considered as a variable to be studied in the analyses.

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