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An Investigation of Thermomechanical Behavior of Tunisian Luffa Sponges' Fibers

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Abstract

This work is realized in the context of valorizing natural and local resources, in particular, luffa plant fruit (luffa sponge). The raw fibers of the luffa sponge have a short lifetime. Hence, when they are chemically treated, it constitutes a solution is prepared to limit their degradation in the long term and to improve their mechanical characteristics. Therefore, this paper studies the effect of the chemical treatment on the mechanical properties of the luffa sponge's fibers (fibers of luffa Sponge). The chemical process consists of dipping a brunch of luffa in various concentrations of sodium hydroxide (NaOH) at different time intervals and at different temperature conditions. The luffa sponge's fibers were mechanical. Characterized before and after the treatment, mechanically (micro traction test). It has been shown that an optimum of 61% increase in mechanical properties (tensile strength) has been reached in the following conditions: treatment with 1% concentration for 90 min at 50˚C.

Keywords

Luffa Sponge's Fibers, Chemical Treatment, Thermogravimetric Analysis (TGA), X-Ray Diffraction Spectrum, Infrared Spectroscopy (IR), Mechanical Characteristics, Tensile Strength

1. Introduction

Luffa is a fast-growing plant in many regions of the world as well in the north of Tunisia. The luffa sponger has a cylindrical shape and a ligneous network system in which the stringy cords are disposed of in a multidirectional systematized row forming a natural mat. This stringy vessel is composed of fibrils fused together

with natural (containing a sticky) accoutrements of towels. In Tunisia, the luffa sponge has limited uses, only in the traditional way, for example, in baths for scratching dead skin. Yet, considering its resistance, when luffa sponge is used in bathrooms treated thermally to harsh conditions such as for example temperature, humidity, and friction, it seems to have good physical and mechanical properties; further, it can get a satisfactory lifetime for its raw material. According to the work of [1], the luffa sponge was composed of 30% hemicellulose, 10% lignin and 60% cellulose. It is important in our case to remove hemicellulose in order to improve the qualification of sponges' fibers. Since it is among the main objectives of this study, in literature, some solutions have been proposed to remove hemicellulose as it is reported in [1] [2].

Nowadays, a clean environment is the aim of the world community. Accordingly, the researchers tried to use vegetal fibers in compound accoutrements as reinforcement for replacing well other types of fibers like carbon fibers and glass fibers. However, the decline in the natural fibers' such as bagasse fibers, kenaf fibers, coir filaments, Alfa fibers, sisal fibers, bamboo fibers, and win fibers, remains a weakness that reduces its mechanical properties and attacks (made up of different effects) fibers performances.

There are several types of treatment, which can be classified into two kinds; chemical treatment like acetylation, peroxide treatment, alkaline treatment, and physical treatment like heat treatment, tube treatment and ultraviolet irradiation. The most used strategy is the synthetic treatment and even more exactly the treatment with NaOH. The authors [3] [4] have studied the alkaline treatment on diverse plant fibers. Each work consistently looks for the leading framework to exhibit the finest results for these diverse procedures for treatment.

It has been shown in [5] if the fibers are immersed in (1%, 3%, 5% and 7%) NaOH for (2, 4 and 6 h); the tensile properties of the treated luffa sponge's fibers are improved significantly, to reach the ideal level in case of concentration 5% NaOH for a specific time 4 hours.

In the present study, we propose a chemical treatment by alkalization of luffa's sponge fibers. The process of alkalization is done under different conditions specially by varying the soaking time, the concentration of NaOH, and the temperature. In each case, we estimate the moisture content. In addition, the mechanical and thermal analysis is done by a micro traction test apparatus and Thermogravimetric Analysis (TGA), respectively.

2. Experimental Apparatus and Procedure

The raw material subject of our study is shown in **Figure 1**. The Luffa sponge (**Figure 1(b)**) is ripened and dried. It is formed by a net grid of fibers and bunions in a porous medium (**Figure 1(c)**). It has an elastic property and it can be compacted.

2.1. Apparatus

To determine the components of luffa sponge's fibers, Infrared spectroscopy was

Figure 1. (a) green luffa sponge and ((b), (c)) dry luffa sponge.

used. For other measurements such as the dimension of the fiber, mechanical properties, thermal properties, we use X-ray spectroscopy, Delteronic DH 400, and ATG, respectively.

- Thermogravimetric Analysis (GTA) is a technique used to characterize thermal degradation. An ATG thermal analyzer "SETSYS Evolution 1750-SETARAM" was used to measure in nitrogen atmosphere at temperature varying, in an interval [30˚C - 900˚C], by an increment of 10 (˚C/min).
- X-ray diffraction analysis is a method of mineralogical analysis of high-performance materials. It can determine atoms' interatomic distances and their arrangement in crystal lattices.
- Infrared spectroscopy (IR) is a method used to determine the nature of chemical bonds in a molecule of each material.
- Moisture is a crucial parameter that should be determined, so the fibers were dried in an oven for 24 h at 105˚C to guarantee mass stabilization. The moisture content is given by Equation (1):

$$\text{Moisturecontent}(\%) = \frac{M_{\text{west}} - M_{\text{dry}}}{M_{\text{west}}} \times 100 \quad (1)$$

where M_{wet} is the initial mass of the sample and M_{dry} is the sample's mass after drying in the oven.

- The fibers' diameters were measured using Deltronic DH 400 (see **Figure 2**), used for contactless manufactured parts measurements, with which the projected image on the screen is magnified 20 times.

2.2. Experimental Procedure

2.2.1. Chemical Treatment

NaOH treatment is done at temperatures (25˚C, 50˚C, and 75˚C). The fibers were cleaned up with hot distilled water to eliminate dust and salinity (see **Figure 3**), then manually disassembled and unbundled into five groups. The fibers were soaking in NaOH solutions at different concentrations (0.5%, 1%, 2%, 3%, and 5%), at various times intervals (1/2 h, 1 h, 3/2 h, 2 h, and 3 h). In total, we obtained 75 samples to be tested differently. Respecting the order of the steps,

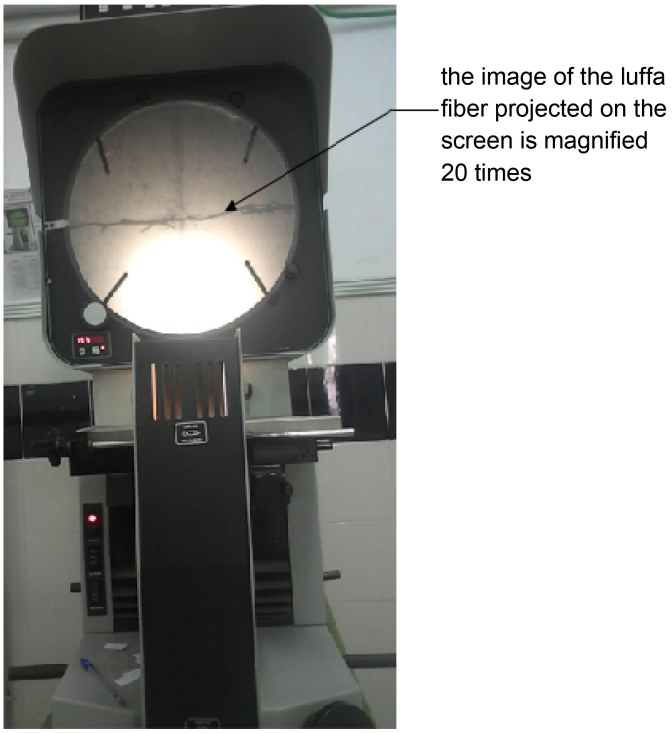

Figure 2. Deltronic DH 400.

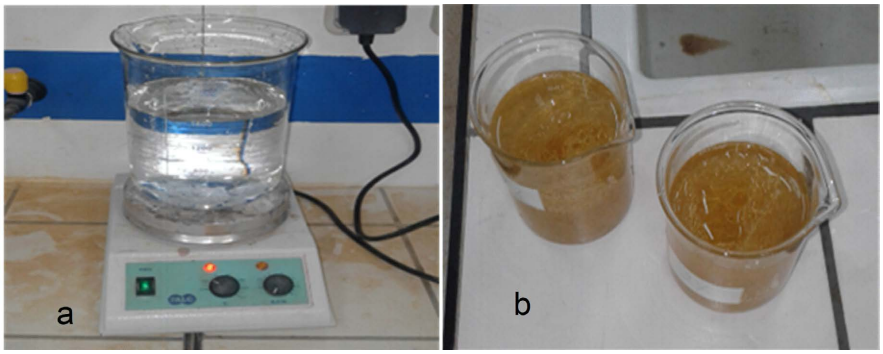

Figure 3. Steps of cleaning fibers in a laboratory: (a) water heating and (b) cleaning fibers with hot distilled water.

the production method, and the treatment values (concentration, temperature, and time), the treatment was done over a series of ordering steps under laboratory conditions.

Mercerizing is the main processing step, which is done by dipping the fibers in a NaOH solution and controlling the concentration, time, and temperature (see **Figure 4**). The reaction between the luffa sponge fiber and NaOH given by Equation (2) is similar to one given in [6]:

$$\text{Luffa fiber-OH} + \text{NaOH} \rightarrow \text{Luffa fiber-O-Na}^{+} + 2\text{OH}^{-} + \text{H}_2\text{O}^{+} \qquad (2)$$

Once again, the fibers are washed with hot distilled water after the NaOH treatment to remove any leftover NaOH residues; then the fibers are dried at the chosen temperature, as it is shown in **Figure 5(c)**.

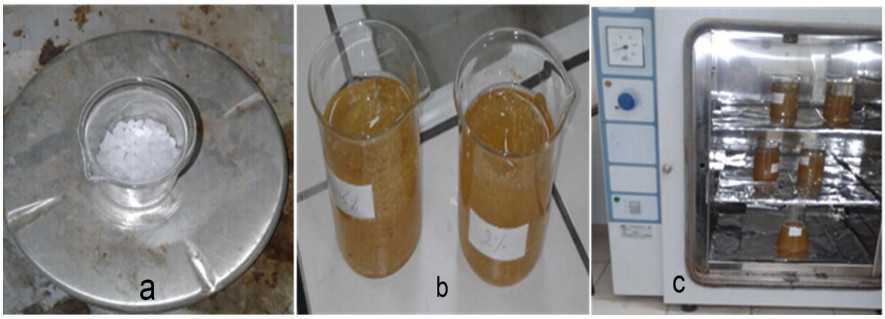

Figure 4. Step of laboratory treatment: (a) NaOH solid, (b) NaOH solution and (c) fiber soaking in NaOH solution in oven.

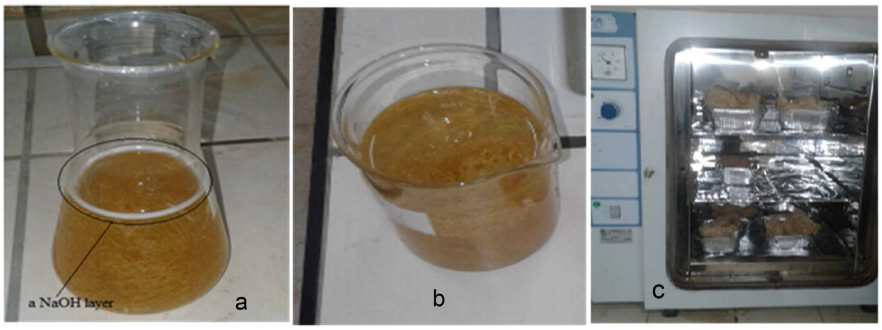

Figure 5. Steps of (a) a layer of NaOH after treatment, (b) the Washing of treated fibers with hot distilled water and (c) fiber drying in the oven.

2.2.2. Mechanical Tests

The sponge fibers of Luffa were at a gauge length of 40 mm and the diameter varied between 0.190 mm and 0.495 mm. The mechanical properties were determined using a traction machine type Shimadzu Auto Graghe with a 20 KN cell (see **Figure 6**) by, in conformity with ASTM D 3822-07. Micro traction tests were performed at a constant speed of 2 (mm/min) to find the Tensile Strength (R_m), which was computed using Equation (3):

$$R_m = \frac{F_{\max}}{S} \tag{3}$$

where F is the maximum breaking load, measured in Newton.

S is the section of the fiber, measured in mm^2.

However, the graph is used to determine the strain (ε) and Young's modulus (E).

3. Results and Discussion

3.1. Physical and Chemical Properties

The measures of moisture content before and after treatment are given in **Table 1** as well as those given in [7].

From this result, we can conclude that the moisture in treated fibers is less than one's untreated ones by a rate of 16.42%. The results are similar to those measured by Y. Chena *et al.* [7].

Figure 6. (a) Example of test pieces of luffa sponge fiber and (b) Traction machine.

Table 1. Moisture content of raw and treated luffa sponge fiber.

Nature of fiber	Untreated fibers	Treated fibers	References
Moisture content (%)	7.31 ± 0.21	6.11 ± 0.08	Presentwork
	8.83 ± 0.3	6.27 ± 0.4	Y. Chena *et al.* [7]

3.1.1. X-Ray Diffraction Results

The XRD diffraction diagrams of the treated and untreated luffa sponge's fibers are shown in Figure 7. The peaks corresponding to inter-reticular distance d (5.51, 4.01, 2.67 Å), after chemical treatment, the XRD images' intensity of the luffa sponge fiber at $2\theta = 16.04^{\circ}$ (110) and $2\theta = 22.1^{\circ}$ (200) increased, indicating an increase in crystallinity by dint of the removal of non-cellular materials by NaOH treatment. This is due to the relaxation of cellulose once the amorphous components and pectin have been eliminated from the fiber as in [8] [9]. Similar observations have been reported as in [10] when treating jute fiber with NaOH.

3.1.2. Infrared Spectroscopy (IR)

The infrared spectrum of luffa sponge's fibers is shown in Figure 8, which determines the absorptions observed indicated in Table 2.

We note that untreated fibers (LN) have strong distinctive peaks at 3424 cm^{-1} (O-H stretching band), 2925 cm^{-1} (CH stretching band), 2367 cm^{-1} (C-O stretching of acetyl or Carboxylic acid), 1728 cm^{-1} (CO stretching band), 1647 cm^{-1} (O-H deformation band), 1455 cm^{-1} (Aromatic methyl group stretching (lignin)), 1244 cm^{-1} (C-H deformation) and 1158 - 1045 cm^{-1} characteristic peaks related to cellulose referred to the studies as in [8] [9].

The fibers of Luffa sponge treated by NaOH shows characteristic functional groups of cellulose and lignin, as shown in the curve (LT) Figure 8. By dint of NaOH treatment, the largest change in the infrared spectrum is that the curve (LT) Figure 8 does not have the high absorption of the carboxyl group at 1734 cm^{-1} (C=O) and the band at 1245 cm^{-1} (C-H) are absent. A similar observation has been depicted as in [11] [12]. Probably, it is due to alkaline treatment.

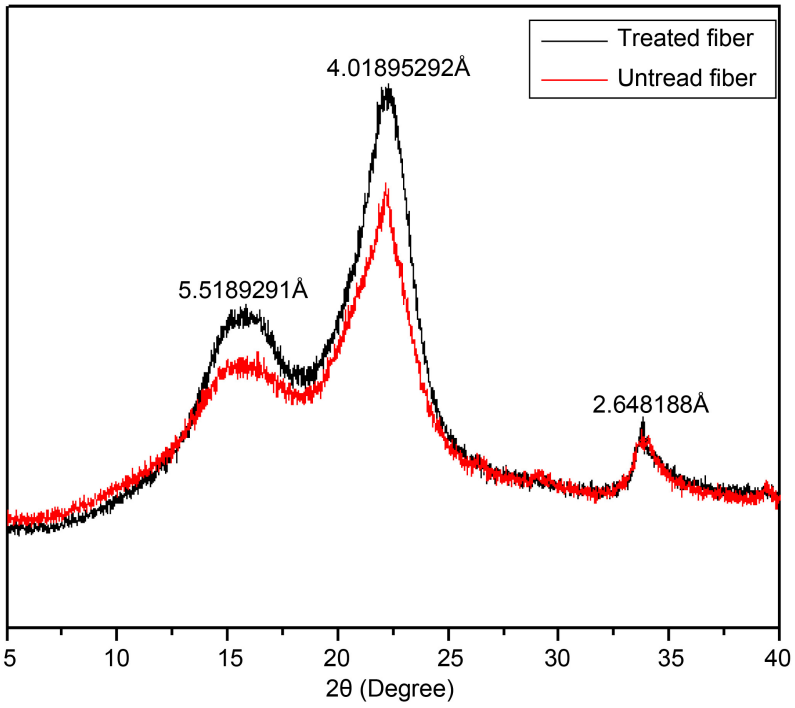

Figure 7. X-ray diffraction spectrum of untreated and treated luffa sponge fiber.

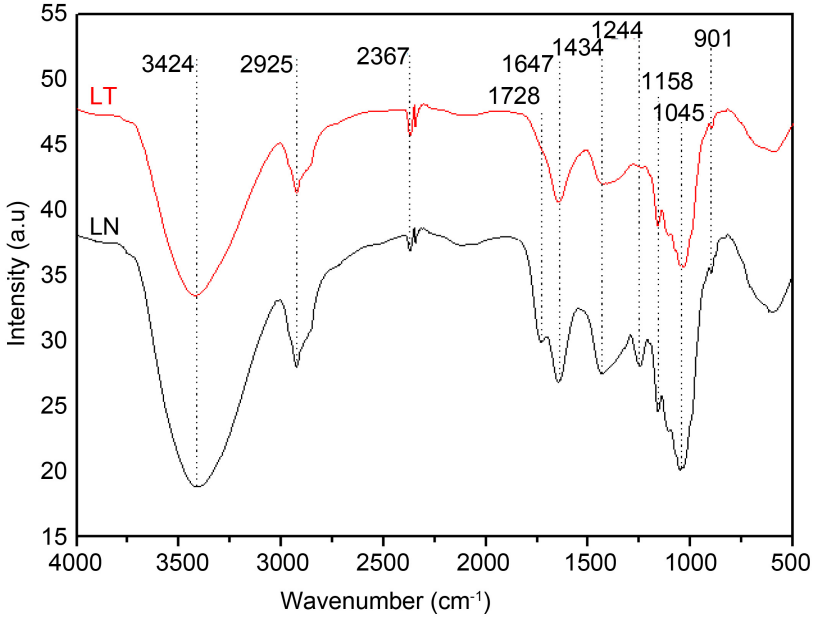

Figure 8. Infrared spectrum of untreated (LN) and treated luffa sponge's fibers (LT).

3.2. Mechanical Properties

The fiber traction curve shown in **Figure 9** proves the fragility of the fibers; also, the plastic zone is absent, only the zone of the elastic behavior is present; similar observation has been reported in [14].

Figure 10 and **Figure 11** show the variation of mechanical properties in different

Table 2. Infrared spectra of treated and untreated luffa sponge's fibers.

Wavenumbers (cm^{-1}) references				Assignments
Present work		Y. Wang *et al.* [13]		
LN	LT	LN	LT	
3424	3424	3430	3370	OH stretching
2925	2925	2920	2925	Saturated C-H stretching
2367	2367	2360	2360	C-O stretching of acetyl or Carboxylic acid
1733		1734		C=O stretching
1647	1647	1636	1636	OH deforming
1422	1434	1450	1455	Aromatic methyl group stretching (lignin)
1244		1383	1383	C-H in deforming
1164	1158	1160	1160	Antisym. bridge C-OR-C stretching (cellulose)
1045	1045	1062	1062	C-OR stretching (cellulose)
901	901	854	854	CH_2 deforming

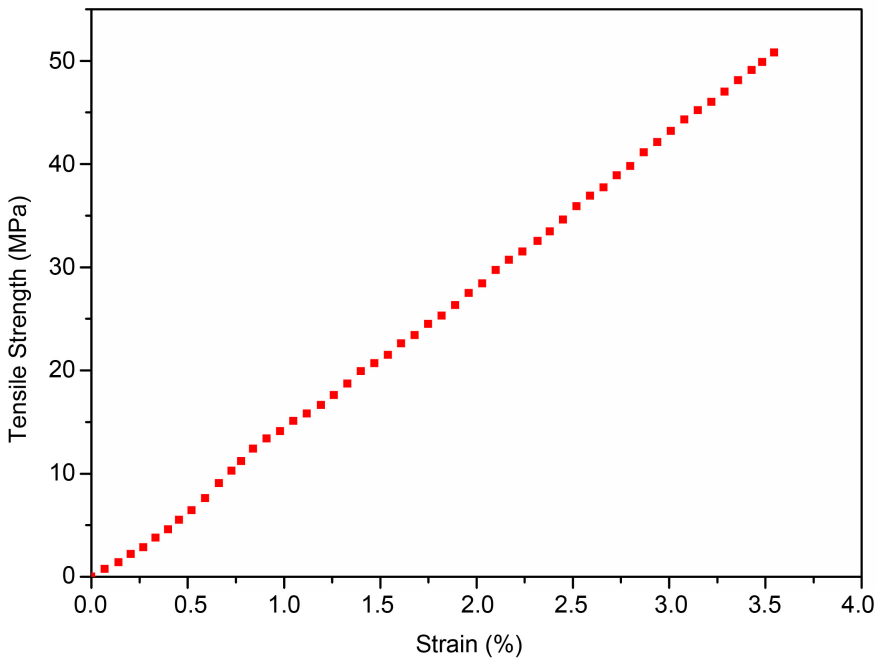

Figure 9. Luffa sponge's fibers typical tensile strength-strain curve.

cases, namely tensile strength (the maximum stress a material can resist before breaking) and the Young module of treated fibers for different concentrations, different durations, and temperatures: (a) treated for 25˚C, (b) treated for 50˚C and (c) treated for 75˚C, with a time interval of 1/2 h, 1 h, 3/2 h, 2 h and 3 h.

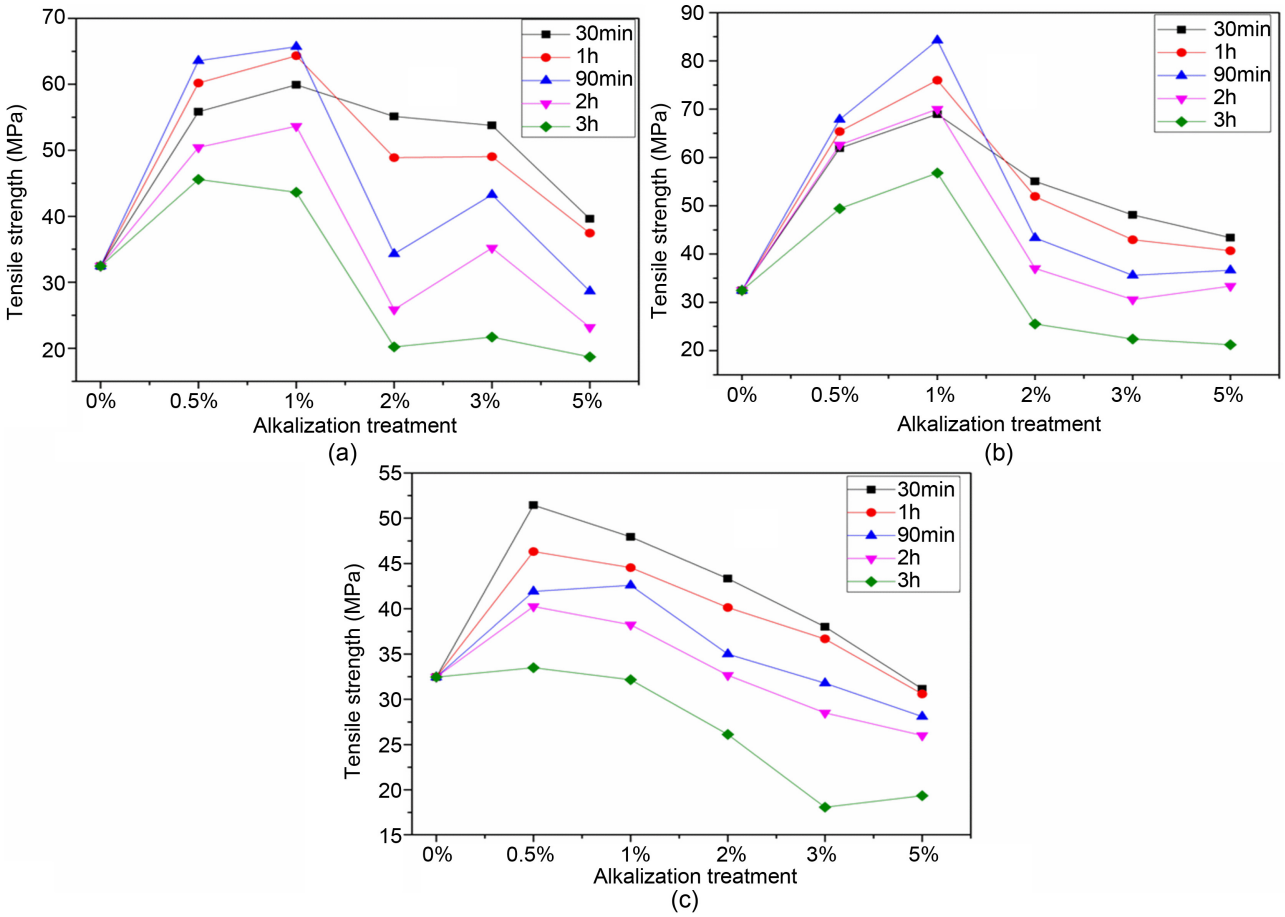

Figure 10. The effect of NaOH on the tensile strength of luffa sponge's fibers treated with different concentrations and duration (a) for 25˚C, (b) for 50˚C and (c) for 75˚C.

Five tests were conducted for each specimen type support.

Table 3 gives the effect of NaOH on the tensile strength and Young's Modulus values of raw and treated luffa sponge's fibers obtained and quoted in the literature.

According to Table 3, the comparison between the studied raw and treated luffa sponge's fibers with others cited in the literature shows that the studied treated fibers have a tensile strength value higher than the raw luffa sponge's fibers, similar results were measured as in [7] [15].

An optimum tensile strength of about 61% rate increase, and for Young's module about 48% increase, is observed for the fibers drenched for about 90 min in an alkaline solution of 1% NaOH at a temperature of 50˚C. This result is very favorable when compared to other fibers, such as coir fibers as in [16], which have a tensile strength of 80 MPa and Young's modulus of 6 MPa. The tensile strength of pineapple leaf fibers is 126.6 MPa, while Young's modulus is 4405 MPa as in [17]. This is the concept behind the valuing of these fibers.

3.3. Thermal Properties

The results of measurements made by the TGA on the treated and untreated

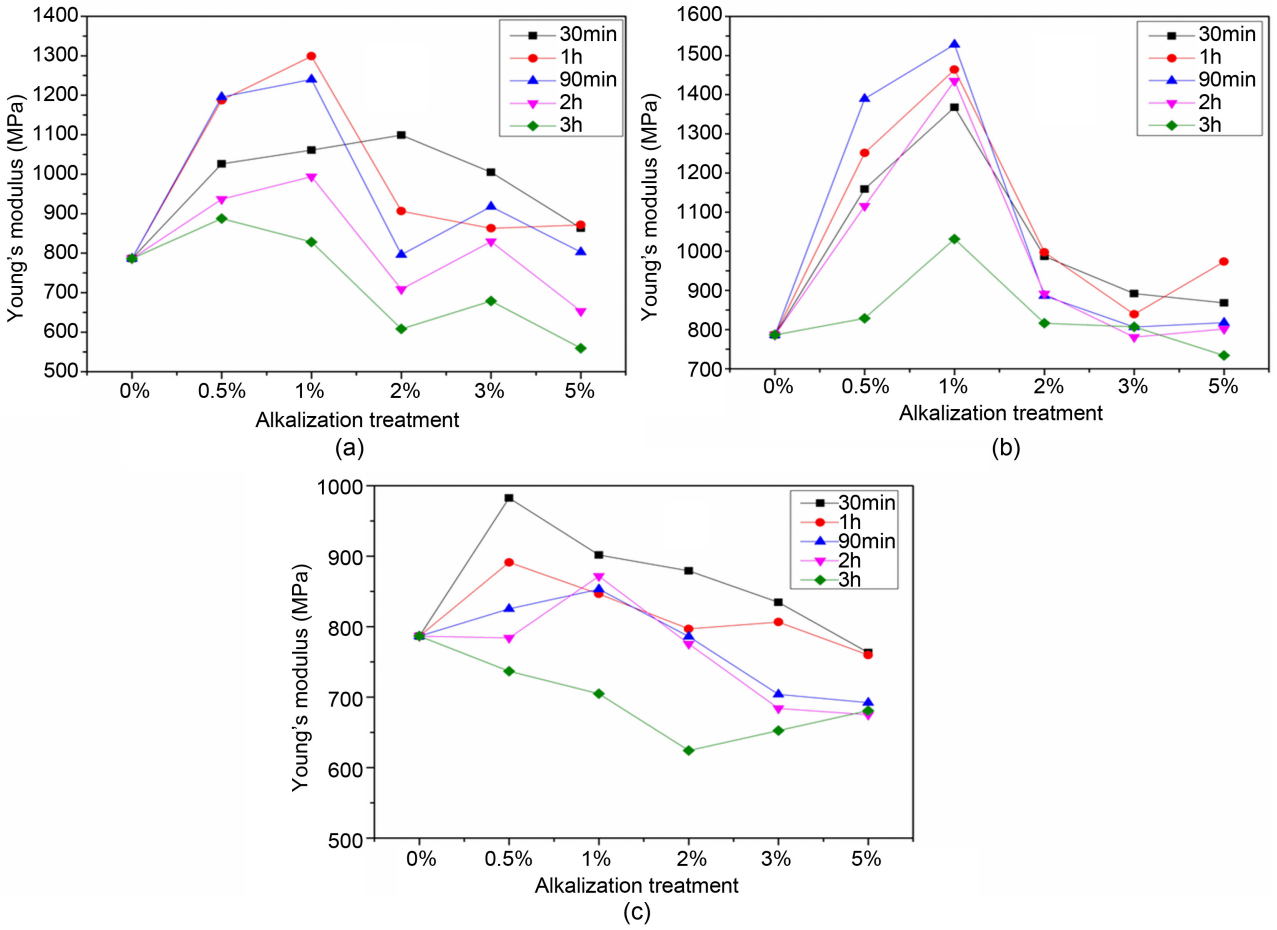

Figure 11. The effect of NaOH on the Young's modulus (E) of luffa sponge's fibers treated with different concentrations and duration (a) for 25˚C, (b) for 50˚C and (c) for 75˚C.

Table 3. Mechanical properties of raw and treated luffa sponge fiber.

Nature of fiber	Tensile strength (MPa)	Young's modulus (MPa)	references
Untreated fiber	32.46	786.52	Presentwork
Fiber treated	84.26	1519.83	
Untreated fiber	33.54	820.24	Y. Chena *et al.* [7]
Fiber treated	74.23	370.03	
Untreated fiber	44	-	T. A. Mobaraka *et al.* [15]
Fiber treated	73	-	

fibers are shown in **Figure 12**. We can observe that the two curves are similar, but the shapes of the two bumps are different. We also notice that the number of fibers is reduced by 100% at 30˚C; for both samples, up to 250˚C, there is a waste of 6% and 11% for treated and untreated fibers, respectively.

This decrease in luffa sponge's fibers is due to the waste of water and moisture under the effect of temperature. Subsequently, the loss of mass accelerates up to 400˚C, where treated fibers lose 33% of their mass while untreated fibers lose

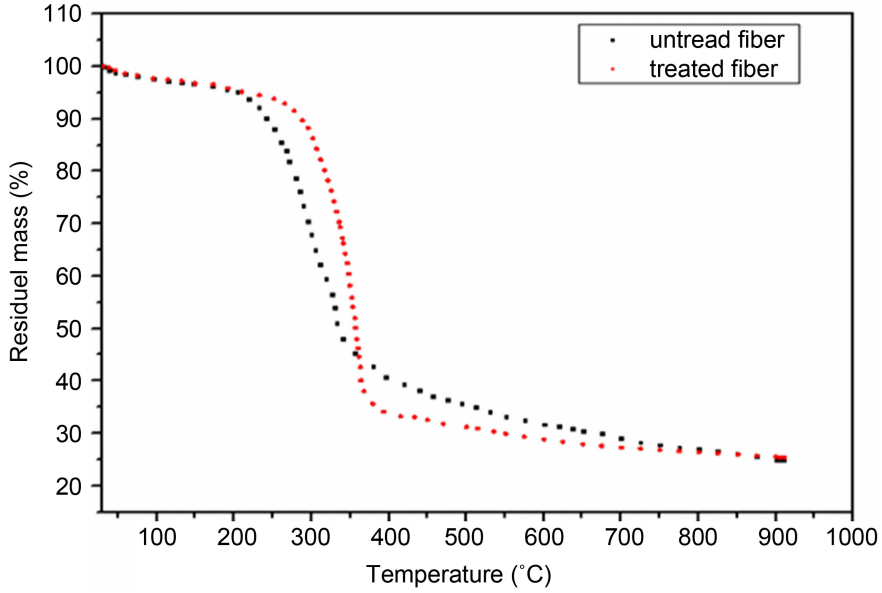

Figure 12. Thermogravimetric analysis of treated and untreated fibers.

40%. This is due to the breakdown of hemicellulose which is more sensitive to thermal degradation.

For 270˚C and 300˚C, the processes of lignin and cellulose decomposition are becoming exothermic. Hemicellulose decomposes at a much lower temperature, than pyrolysis of cellulose and lignin which occurs respectively at 300˚C and 400˚C as it reported in [18].

Izani *et al*. [19] proved that the temperatures vary between 200˚C and 400˚C, corresponding to the decomposition of cellulose and hemicellulose. While lignin's decomposition starts above 400˚C as in [20], and it is the most difficult component to degrade the mass stabilizes at 700˚C or 30% for treated fiber; however, the loss remains stable above 550˚C or 37% for untreated fiber. Therefore, the lost mass of treated fiber is lower than that of untreated fiber, as such as shown in **Figure 12**. Thus, we can conclude thermal stability was improved by fiber treatments.

4. Conclusions

This research proves that chemical treatment remains the main significant method to improve the mechanical properties of luffa sponge's fibers, reaching an increased rate of 61%. These results induce the potential of using treated luffa sponge's fibers in composite materials (MC) for buildings instead of carbon fibers.

Furthermore, this study needs more investigation to estimate the lifetime of luffa sponge fiber in composite materials.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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