PAPER

Evaluation of surface pre-treatments on the surface characteristics modifications in sponge gourd (Luffa Cylindrica) fibers

To cite this article: G Kalusuraman et al 2019 Mater. Res. Express 6 095106

View the article online for updates and enhancements.

Recent citations

- <u>Characterization of Surface Modified</u> <u>Phoenix sp. Fibers for Composite</u> Reinforcement
- G. Rajeshkumar
- Dynamic fracture toughness index: a new integrated methodology for mode-I fracture behaviour of polymer composite under impact loading Vinod Kushvaha et al



IOP ebooks™

Start exploring the collection - download the first chapter of every title for free.

Materials Research Express



RECEIVED 15 May 2019

REVISED 20 June 2019

ACCEPTED FOR PUBLICATION
15 July 2019

15 July 2019

PUBLISHED 24 July 2019

PAPER

Evaluation of surface pre-treatments on the surface characteristics modifications in sponge gourd (*Luffa Cylindrica*) fibers

G Kalusuraman^{1,2}, I Siva^{1,5}, Mustafa Aslan², S Anandkumar³ and K G Satyanarayana⁴

- Department of Mechanical Engineering, Kalasalingam Academy of Research and Education, Anand Nagar-Krishnankoil-626 190, Tamil Nadu, India
- ² Faculty of Engineering, Department of Metallurgical and Materials Engineering, Karadeniz Technical University, Trabzon, Turkey
- ³ Department of Mechanical Engineering, Indian Institute of Technology Jammu, J&K, India
- Poornaprajna Institute of Scientific Research (PPISR), Sy. No. 167, Poornaprajnapura, Bidalur Post, Devanahalli, Bengaluru-562 110, Karnataka, India
- 5 Author to whom any correspondence should be addressed.

E-mail: isiva@klu.ac.in, maslan@ktu.edu.tr, anand.subramaniyan@ iitjammu. ac.in and gundsat42@hotmail.com

Keywords: Luffa Cylindrica fibers, surface pre-treatments, surface roughness, fractogrphy

Abstract

Owing to unique characteristics such as lower weight, biodegradability, etc, of natural fibers and as well as increasing attention being given towards renewable material sources demands the investigations particularly for the growth of polymer-based composites. *Luffa Cylindrica* fiber is one such fiber, whose complete characterizations are required to increase its applications such as in tribological application. This study presents the physical and tensile properties along with its chemical and morphological aspects of *Luffa* fibers with and without chemical surface pre-treatments. Chemical surface pre-treatments employed in this work are viz sodium hydroxide, silane and calcium hydroxide. The results shows that the chemical pre-treatments improved the physical (diameter and surface roughness) and tensile properties as well as morphology and chemical composition of the fibers suggesting suitability of these fibers in engineering application.

1. Introduction

Usage of natural fibers as reinforcement is in great demand in engineering applications due to their abundant availability, low cost, eco-friendliness and bio degradability. Recently researchers have been focusing on the manufacturing of green products to save the earth from global warming and as well as recyclability point of view. Further, industries are very keen to produce natural fiber reinforced based polymer products to replace metals, alloys and neat polymers. The natural fibers, as reinforcement, provide affordable strength, better stiffness, and high modulus [1] compared to man-made synthetic fibers. The natural fiber reinforced composites (NFRC) are considered as a most preferred candidate material which offers comparatively better advantages over the synthetic fibers reinforced composites [2, 3].

James Holbery $et\,al\,[4]$ reviewed the applicability of NFRC in automotive applications. Additionally, these authors have reported that the European companies have taken the measures to employ the NFRC to cater structural parts subjected to lighter load in automotive besides starting to support the successful manufacturing by leading manufacturers like BASF (Germany), Dieffenbacher (Germany), and Rieter Automotive (Switzerland). Karus $et\,al\,[5]$ have reported that the quantity of NFRCs used in German automotive production accounted for 30 000 Tons in 2005 and expected to increase in years to come.

Various research works have been carried out to explore the different types of natural fibers such as sun hemp, coir, bamboo, flax, jute, ramie, roselle, kenaf, sugar cane, straw, rice husk, grass, pineapple leaf, raphia and papyrus etc to name a few. Also, it is well established that employing a suitable surface pre-treatment to natural fibers play a key role in deciding the interface bonding with polymer matrix.

Reddy et al [6] investigated the alkali pre-treatment effects on the century fiber. They reported that the mechanical properties and crystallinity index had increased after the alkali pre-treatment. Shanmugasundaram



Figure 1. Photograph of the luffa fiber.

et al [7] characterized raw and alkali-treated mulberry fibers and observed that alkali treatment removed impurities from the fiber and helped to assist good mechanical bonding between binder, matrix and reinforcement.

Mueller *et al* [8] have compared the performance between conventional glass fibers with natural fibers (flax, kenaf and cotton). They reported a significant reduction in total mass with enhanced mechanical properties. In addition, authors extended their recommendation of NFRC towards the automotive structural application. Murali Mohan Rao *et al* [9] and Edeerozey *et al* [10] had investigated the changes in morphological characteristics of natural fibers resulted from the surface treatments. They found the correlation between enhancement in mechanical properties obtained after the fiber surface pre-treatments. Vijayakumar thakur *et al* [11] had treated the natural fibers with silane and reported the improvement in the mechanical properties of NFRC.

Contradictorily, Boynard *et al* [12] had reported that despite change of morphology of untreated luffa fiber after the surface pre-treatment, there was no significant enhancement of mechanical properties. Gor Maria De Rosa *et al* [13] investigation on the chemically pre-treated Okra fibers showed similar results of insignificant improvement in mechanical properties. They attributed this to the fiber deterioration due to the chemical pre-treatment.

Nagaraja Ganesh $et\ al\ [14]$ had studied on the possibility of extraction of the Ligno-cellulosic fibers from $Luffa\ Cylindrica$ plant and reported the possibility of these natural fibers as reinforcement in polymer composite production. Ghali $et\ al\ [15]$ studied the surface pre-treatment of the Luffa fiber with alkali and peroxide. They reported the enhancement in crystallinity index and mechanical properties of composites. Altınışık $et\ al\ [16]$ reported that the $Luffa\ Cylindrica$ fibers could be used as effective adsorbent for the elimination of malachite green from aqueous solution. Kalusuraman $et\ al\ [17]$ studied the friction coefficient of Luffa Cylindrica fibers containing polymer composites by varying the surface treatment. They observed that the significant changes in fiber morphology due to pre-treatments resulting in better tribological performance of the composites.

Considering the above mentioned facts, present work is aimed at the extraction and characterization in respect of physical (including morphlogy), chemical and mechanical properties of luffa fibers with and without surface modifications. These are expected to to find suitable engineering applications for these fibers.

2. Experimental methods

2.1. Materials

In the present work, dried *Luffa* fruits were used in fiber extraction. These fruits were supplied by Shiva fibers, Rajapalayam (Tamil Nadu, India). Figure 1 shows the photograph of the luffa fiber as it appears on along with the fruit.

The chemicals used for the surface treatment of fibers include sodium hydroxide, silane and calcium hydroxide and these were purchased from Ganapathy Scientific, Chennai, Tamil Nadu, India.

2.2. Methods

2.2.1. Extraction of fibers

The dried fruits were first cut longitudinally to crush and to take out the skin. In order to remove the seed from the *Luffa* fruits, the top and bottom ends of the fruits were cut downwards. Further, the fibers in the form of mats

were taken out. These fiber mats were washed by distilled water and were kept in a desiccator at room temperature for about 12 h and subsequently the mats were completely dried.

2.2.2. Surface pre-treatment of Luffa fibers

2.2.2.1. NaOH treatment

The dried *Luffa* fiber mats were immersed in NaOH solution (40 gm of NaOH pellet per litre of water) for an hour. Equation (1) illustrates the surface chemical reaction possible on the *Luffa* fiber mat. The mats were taken out of the solution, washed by distilled water and finally dried in sunlight for minimum 12 h.

2.2.2.2. Silane treatment

The silane treatment of fibers was carried out using trichlorovinylsilane solution (A litre of distilled water would acidify to the pH of 3.5 with acetic acid and 0.3% trichlorovinylsilane was diluted to form the silanol solution). Equation (2) illustrate the surface chemical reaction possible on the *Luffa* fiber mat. NaOH treated fiber mats were then submerged in the prepared silane solution for one hour followed by drying in room temperature for a minimum of 12 h.

2.2.2.3. Calcium hydroxide [Ca(OH)₂] treatment

The calcium hydroxide pre-treatment were performed using $Ca(OH)_2$ solution (prepared by adding 74 gm of $Ca(OH)_2$ in one liter of distilled water). Then the *Luffa* fiber mat was immersed in the solution for 1 h. Then it was taken out, washed with distilled water and finally, dried in sunlight for 12 h. Use of such high volumes of chemicals is already reported [18, 19].

2.3. Physical properties

2.3.1. Morphology and dimensions of Luffa fibers

In order to understand the morphology and to determine the dimensions of the fibers in the mat, about 30 fibers of each of the above varieties (untreated, NaOH treated, Ca(OH)₂ treated and silane treated) were observed under a scanning electron microscope (ZEISS, EVO18, Germany). Measurements were taken at least at 3–4 places along the length of the fibers of each variety.

2.3.2. Density

Density of both the untreated and various treated *Luffa* fibers was determined using a pycnometer which was previously weighed with toluene as the immersion liquid based on the method suggested by Beakou *et al* [20]. Briefly the method is as follows: The fiber mats were dried for 96 h in desiccator containing silica. Further, they were cut into 5-15 mm long and their initial weight was determined by keeping the fiber mats in the pycnometer at room temperature (\sim 18 °C) and relative humidity of 57% (it was only 3% in the desiccator). Subsequently, the fiber mats were immersed in toluene for 2 h to make the fiber mats free of micro bubbles and again the weight was determined. Based on these, density of the *Luffa* fiber mat was calculated using the following equation (3).

$$\rho = \frac{m_2 - m_1}{(m_3 - m_1)(m_4 - m_2)} \rho t \tag{3}$$

where ρ = the density of the fiber mat (g/cm³),

 $\rho_{\rm t} = {\rm is}$ the density of the toluene (866 kg.cm⁻³),

 m_1 = the mass of the empty pycnometer (kg),

 m_2 = the mass of the pycnometer filled with chopped fibre mats (kg),

 m_3 = the mass of the pycnometer filled with toluene (kg)

 m_4 = the mass of the pycnometer filled with chopped fibres and toluene (kg).

2.3.3. X-ray diffraction studies

X-ray diffraction spectra of both the chemically pre-treated and untreated *Luffa f*iber mats were performed using a Rigaku x-ray diffractometer (Model: D/Max Ultima III) with monochromatic Cu K α radiation and with operating conditions of 40 kV and 30 mA. The scanning range used was $2\theta = 10-80^{\circ}$ by a scanning speed of 5.0 deg min⁻¹. The integrated intensities of the peaks of Bragg in the spectrum of the *luffa* fiber mats were attained, and their crystallinity indices were estimated using the following equation (4) [21]

Crystallinity index(CI) =
$$\frac{I_{am} - I_{002}}{I_{002}}$$
 (4)

where I_{002} is the peak height of the crystalline part and I_{am} is the peak height of the amorphous part.

2.4. Chemistry and stoichiometry studies

To understand the amounts of lignin, cellulose, moisture, wax and ash contents of both the chemically pretreated and untreated *Luffa* fiber mats, their chemical analysis was carried out following the conventional chemical analysis method based on appropriate ASTM standards. Lignin contents were found by using the Klason method [22]. While the wax content in the fibers was determined by the Conrad method [23]; ash content was found as per the standard (ASTM E 1755-61).

Moisture content of the fiber mats was calculated as reported elsewhere [24]. Briefly the method is as follows: A specific quantity of fiber mats were weighed and then they were dried at 104 °C till the constant weight was observed (for about 4 h). Subsequently, difference between the initial and final weights would give the moisture content of the fiber mats. Average values obtained for five samples were taken for the final value with standard deviation.

On the other hand, the chemical structure of the both chemically pre-treated and untreated *luffa* fiber mats were determined by Fourier transform infra-red (FTIR) spectroscopy by the method suggested by Kurschener and Hoffers [25]. First, the fiber mat samples were chopped into tiny particles and pulverized to a fine powder using a mortar and pestle. The fiber powders were then mixed with KBr and pelletized to do the FTIR analysis. Perkin Elmer Spectrum RXI FTIR spectrometer was used at a scanning rate of 32 scans/min with a resolution of 2 cm^{-1} in the wave number range between $400-4000 \text{ cm}^{-1}$.

2.5. Tensile properties

Tensile properties of single fibers were determined as per the ASTM D 3379 standard using an Instron universal testing machine of type 5500 R with fiber samples of 50 mm gauge length [1]. Test conditions used were: applied pressure of 0.4 MPa using pneumatic gripper in a load cell of 1.0 kN and cross head speed of 0.1 mm min $^{-1}$ at ambient temperature (21 °C) and a relative humidity of about 61%. The displacement taking place in the fiber samples was noted using short-stroke transducer having a resolution of approximately 0.1 mm. The tensile tests were performed with the average strain rates in the order of 0.6 s in order to account for the unevenness of natural fibres, 30 number of fiber samples under each conditions were tested and the average values were reported.

2.6. Surface roughness measurement

The surface roughness of all types of fiber mat samples (50 mm long) used in this study was measured along the length of the specimens using a non-contact type 3D profiler (Talysurf CCI MP, UK). Average of 5 measurements on each sample was considered as the final value.

3. Results and discussion

3.1. Physical properties

3.1.1. Density of fibers

Table 1 lists the density values of the treated and untreated Luffa fiber mat. The density of the fiber mat was highest for the fiber mat treated with NaOH followed by that silane treated fibers. On the other hand, the density decreased by the $Ca(OH)_2$ treatment, which indicated lower that of the untreated fiber mat.

3.1.2. Fiber diameter

Figure 2 shows scanning electron micrograph of the surface of treated and untreated *Luffa* fibers, which were used for the measurement of diameters. Table 2 lists the average of measurement of the 35 fibers of the each

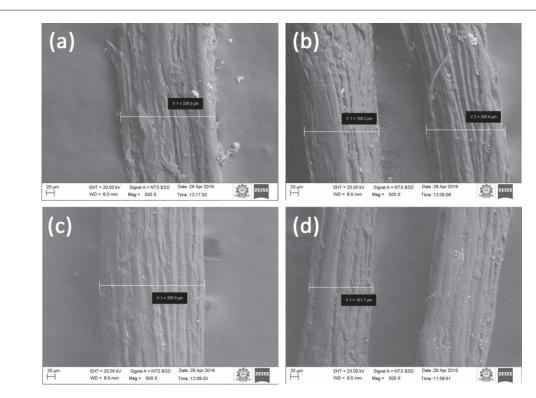


Figure 2. Scanning electron micrographs showing surfaces of *Luffa* fibers with and without surface pre-treatments: (a)-Untreated; (b)-NaOH treated; (c)-Silane treated and (d)-Ca (OH)₂ treated.

Table 1. Density of untreated and treated Luffa fibers at room temperature.

Sl no	Types of fiber	Density Kgm ⁻³
1.	Untreated <i>luffa</i>	1247
2.	NaOH treated <i>luffa</i>	1337
3.	Silane treated <i>luffa</i>	1310
4.	Ca(OH) ₂ treated <i>luffa</i>	1145

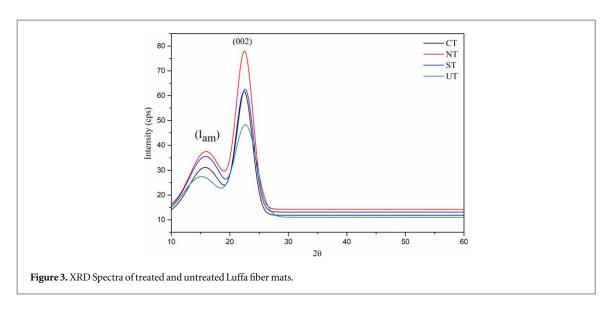
Table 2. Diameter values of untreated and treated *Luffa* fibers

Sl. No.	Treatment	Diameter (mm)
1.	Untreated	0.46
2	NaOH	0.36
3	Silane	0.34
4	$Ca(OH)_2$ treated	0.47

variety. It can be seen from the table 2 that the $Ca(OH)_2$ treated *Luffa* fibers showed the highest value of diameter among other treated fibers and which was almost equal to that of the untreated fibers. On the other hand, surface treatments of fiber mats with NaOH and silane slashed the fiber diameter values compared to untreated fibers. $Ca(OH)_2$ may provide a coating on the surface of the fibers increasing the diameter of the fibers, while other two types of treatments may erode the surface of the fibers resulting fiber diameter reduction. This is in agreement with the observation of earlier reports [6, 11] about effect of treatment of natural fibers with NaOH and Silane solutions.

3.1.3. X-ray Diffraction (XRD) Studies

Figure 3 shows the XRD spectra of treated and untreated *Luffa* fiber mats revealing characteristic of lignocellulsosic fibers with two peaks indicating the amorphous and crystalline parts.



As can be seen from the figure that these peaks are noted at $2\theta = 15^{\circ}$ and $2\theta = 22^{\circ}$ and also, they can be attributed to [002] and [110] crystallographic planes respectively [21].

Values of crystallinity index obtained for untreated and treated fibers were found to be 33.01%, 29.13%, 29.84% and 29.61% respectively for untreated, NaOH treated, silane treated and Ca (OH)₂ treated *Luffa* fiber mats. These values clearly point out that the highest value of crystallinity index is for untreated fibers indicating more ordered structure in these fiber mats, while the treatment by different chemicals lowered the ordered structure with possibility of higher amount of amorphous parts, similar to the observations for other natural fibers [14].

3.1.4. Surface roughness characterization

It is well known that the roughness values of fiber play a vital role in the compatibility of the fiber and matrix during the preparation of composites. Figures 4(a)–(d) show the surface texture of the untreated and chemically pre-treated *luffa* fibers. It can be seen from these 3D diagrams that fiber surfaces are irregular as revealed by different colors. These irregularities might be due to the presence of voids and some impurities.

Figures 4(a)–(d) shows the 2D line diagram of surface pattern of the untreated and surface treated luffa fibers. Roughness values (Ra) were obtained from these figures, which indicated highest value of 4.86 μ m for untreated fibers. In the case of chemically pre-treated fibers, roughness values were found to be in the decreasing order starting with those for NaOH treated followed by that of silane treated and the lowest for Ca (OH)₂ treated fibers.

3.2. Chemical analysis

3.2.1. Chemical composition

The chemical composition of lignocellulosic fibers influences the strength properties of fibers as well as their polymer composites [20]. Table 3 shows amounts of chemical constituents of untreated and treated *Luffa* fiber mats. It can be seen that NaOH treated fibers show the highest cellulose content followed by those of silane treated. On the other hand, $Ca(OH)_2$ treated fiber exhibited the lowest cellulose even lower than the untreated fibers. The cellulose content in fiber plays a substantial role in the tensile strength of fiber. These observed cellulose content of dissimilar types of fibers is supported by the values of crystallinity index obtained by x-ray diffraction studies as seen earlier.

On the other hand, lignin content decreased drastically in all types of treated fibers compared to that of untreated fiber. This suggests chemical treatment of the fiber with different chemicals leads to removal of lignin along with some impurities of the fiber as observed by other researchers [4]. Similarly, wax content also decreases by the chemical pre-treatment of the fibers, in agreement with earlier reports [16].

It is well known that wax on the surface of the fiber which affects the interfacial bonding between the fiber and matrix in the preparation of the composites. Therefore, lower wax content leads to have the better interlocking between the fibers and the polymer matrix. This would be beneficial in getting better adhesion, which is in agreement with earlier reports [26] and thus result in improving strength of polymer composites reinforced by these fibers. In fact, tensile properties of *Luffa* fibers themselves showed improvement over those of untreated fibers as will be discussed later in the paper.

During chemical analysis, silanized fibers were found to produce lowest ash when compared to other variety of fibers (62% lower than that obtained in the untreated fibers and 56% lower than that obtained by other surface

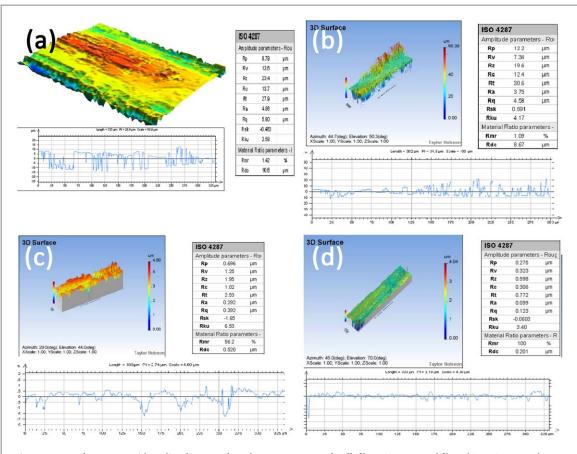


Figure 4. 3D surface texture with 2D line diagram of roughness parameters of Luffa fiber -(a): untreated fiber; (b): NaOH treated; (c): Silane treated and (d): $Ca(OH)_2$ treated.

Table 3. Amounts of chemical constituents of untreated and treated fibers.

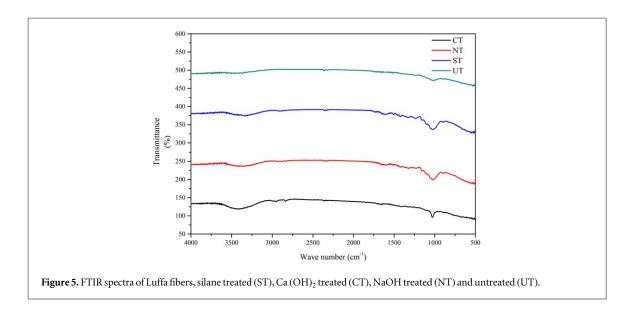
Property (%)	Untreated	NaOH treated	Silane treated	Ca(OH) ₂ treated
Cellulose content	73.92	83.69	81.73	60.19
Ash content	4.74	4.07	1.79	3.81
Moisture content	9.75	9.82	9.78	10.45
Lignin content	21.85	11.39	15.55	10.37
Wax content	0.48	0.37	0.24	0.10

treatments used in this study). This suggests suitability of the silanized fibers for fire resistance applications of composites.

3.2.2. FTIR studies

Figure 5 shows the FTIR spectra of untreated and chemically pre-treated [NaOH, Ca(OH)₂, and silane] *Luffa* fibers. It can be seen from the figure that spectrum for the untreated fibers is similar to those observed for other lignocellulosic fibers [25, 26], exhibiting an absorption bands between 2800 cm $^{-1}$ to 2900 cm $^{-1}$. These are due to aliphatic C–H stretching vibrations indicating the presence of alkyl chains. The band appeared at 1737 cm $^{-1}$ is due to absorption acid carbonyl. The bands in the range 1000–1200 cm $^{-1}$ confirms the C–O–C stretching of polyesters of all the chemically treated and untreated fibers. The appeared band within the range 3200–3400 cm $^{-1}$ might be due to O–H stretching vibrations, which indicates the presence of hydroxyl groups in the polyesters.

On the other hand, NaOH, $Ca(OH)_2$ and silane treated fibers show absorption bands in 2800–2900 cm⁻¹ confirming the non-aromatic C-H stretching in all the treated fibers. The strong absorption band at 1737 cm⁻¹ in the untreated fiber is shifted to 1654 cm⁻¹ in NaOH treated fibers. This may be due to the formation of salt of carboxylate group with sodium. But calcium and silane treated fibers did not show any evidence of any variation in this band suggesting no structural changes taking place in the organic part of the *Luffa* fibers after treatment with these two chemicals as observed by others [27].



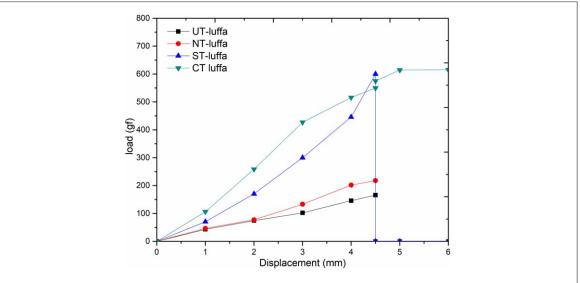


Figure 6. Plots of applied load versus displacement of untreated (UT) and chemically pre-treated [NaOH-NT; Silane-ST and $Ca(OH)_2$ treated] *luffa* fibers.

3.3. Tensile properties

Figure 6 shows load versus displacement curves of untreated and chemically pre-treated *Luffa fibers*. Thirty 30 fibers were tested and average values of load and displacement is plotted. It can be seen from the figure that the untreated fibers show the characteristic slow increase in the applied load with increasing strain. This increase in load is lower compared to those of chemically pre-treated fibers with Ca(OH)₂ treated fibers showing the highest applied load at the highest strain followed by that for silane treated and finally that of NaOH treated *Luffa* fibers.

From the stress-strain curves obtained for all the fibers, values of tensile properties (Young's modulus, yield strength, ultimate strength and % elongation) were determined. These values are tabulated in table 4. It can be seen from the table that untreated fibers exhibit lowest values of all the tensile properties when compared to chemically pre-treated fibers irrespective of the chemical used. This observation is expected and in general agreement with earlier reports [28].

These observations are understandable, since the untreated fibers possess cracks and pores on the surface of the fibers as evident from the morphology studies of the fiber (see next Section). Further, the surface treatments with used chemicals and silanation of fibers seem to sustain the fiber surface roughness to some extent and provide space for polymer anchoring. This is evident from the SEM micrographs of the untreated and surface treated fibers (See next Section). In fact, removal of impurities from the fiber surface due to these treatments may impart greater mechanical interlocking with the polymer matrix. In the case of silane treatment, there may be cross linked network owing to covalent bonding by restraining the swelling of the fiber. This improves the strength properties and the observations are in agreement with earlier observations by others [29, 30].

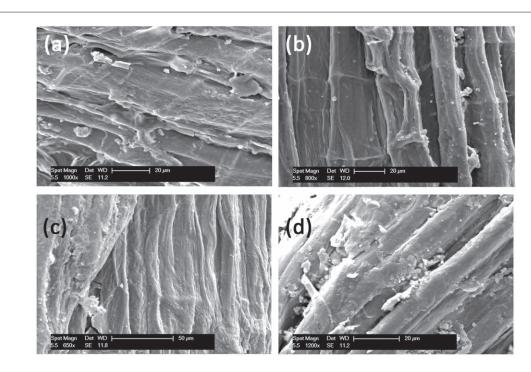


Figure 7. Scanning electron microscopic photographs of surfaces of Luffa fibers-(a): Untreated fiber; (b): NaOH treated fiber; (c): Silane treated fiber and (d): $Ca(OH)_2$ treated fiber.

 ${\bf Table \, 4. \, Values \, of \, tensile \, properties \, of \, chemically \, and \, untreated \, treated \, \it Luffa \, fibers.}$

Fiber	Ultimate tensile strength (MPa)	Elongation (%)
Untreated	309.52 ± 18	5.52
NaOH treated	274.18 ± 16	6.62
Silane treated	524.96 ± 25	4.04
Ca (OH) ₂ treated	719.31 ± 32	5.89

Treatment of Luffa fibers with $Ca(OH)_2$ may provide better coating of the fibers whereby load bearing capacity of the fibers might have increased leading to the highest tensile properties.

3.4. Morphology studies

Figures 7(a)–(d) show the surface structures of *Luffa* fibers with and without surface pre-treatments using NaOH, silane and Ca $(OH)_2$ solutions. It can be observed that surface of untreated fiber (figure 7(a)) exhibits highly rough surface along with some impurities adhering to the fiber surface. On the other hand, surfaces of chemically pre-treated fibers seem to be smoother with NaOH treated fiber showing slightly rougher surface with almost absence of impurities on the fiber surface (figure 7(b)) compared to surfaces of other two chemical treated ones, viz., silane treated (figure 7(c)) and Ca $(OH)_2$ treated (figure 7(d)) surfaces. It can also be observed that the presence of some cracks on the surface of NaOH treated fiber. The above observations can thus explain the differences in tensile properties obtained in this study.

4. Conclusions

In this investigation, the fibers extracted from the matured *Luffa* fruits are characterized to find the suitability of producing the polymer composite towards low load structural applications. Based on the characterization of *Luffa Cylindrica* fibers in respect of its physical properties (dimensions, density, structure, morphology and surface roughness), chemical properties (chemical composition and structure) and tensile properties, the following conclusions can be drawn:

- Surface pre-treatment of *Luffa* fiber removed majority of the non-crystalline constituents, resulting fiber's overall strength enhancement.
- NaOH treated fibers showed the highest cellulose content (83.69%) followed by those of silane treated fiber (81.73), Ca(OH)₂ treated fiber (60.19%) in comparison to the value of 73.92% for the untreated fibers. On the other hand, lignin content decreased drastically in all the surface treated fibers [11.39%, 15.95% and 10.37% for NaOH, Silane and Ca(OH)₂ respectively] compared to that of untreated fiber (21.85%). Similarly, wax content also decreased for all the surface treated fibers with 0.37%, 0.24% and 0.10% for NaOH, Silane and Ca(OH)₂ respectively compared to 0.48% of untreated fibers.
- Diameter of the Ca(OH)₂ treated fibres is the highest (0.47 mm) (almost similar to that of untreated fiber-0.46 mm) amongst all fibers while NaOH and silane treatments decreased diameters of *Luffa* fibers (0.36 mm and 0.34 mm respectively). This reduction in diameter resulted in high aspect ratio, which would enhance better load transfer in composites.
- Chemical treatments of Luffa fibers decreased the crystallinity index of Luffa fibers as determined by XRD with
 the highest value of about 33 % for untreated fibers. Obtained values of crystallinity index treated fibers were
 found to be 33.01%, 29.13%, 29.84% and 29.61% respectively for NaOH treated, silane treated and Ca (OH)₂
 treated Luffa fiber mats.
- Surface roughness values (Ra) of Luffa fibers were found to be the highest (4.86 μm) for untreated fibers, while
 they decreased for all the surface treated fibers with the decreasing order starting from those for NaOH treated
 followed by that of silane treated and the lowest for Ca (OH)₂ treated fibers. This would promote polymer
 anchoring due to high peak and valley with negligible impurities.
- Ca (OH)₂ treated *Luffa* fibres showed the highest tensile strength (\sim 719 MPa) followed by silane treated (\sim 525 MPa) and NaOH treated (\sim 274 MPa) compared to that of untreated fibers (\sim 309 MPa).
- Based on all the above finally it may be stated that surface treatment of the *Luffa* fiber mats would lead possibility of using these fibers in making their composites with polymer matrices suitable for structural and other engineering applications.

Acknowledgments

This particular research work hasn't received any financial support from any of the external agencies.

ORCID iDs

References

- [1] Joseph P V, Joseph K and Thomas S 1999 Effect of processing variables on the mechanical properties of sisal-fiber-reinforced polypropylene composites *Compos. Sci. Technol.* 59 1625–40
- [2] Geng Z C, Sun J X, Liang S F, Zhang F Y, Zhang Y Y, Xu F and Sun R C 2006 Characterization of water-and alkali-soluble hemicellulosic polymers from sugarcane bagasse *Int. J. Polym. Anal. Charact.* 11 209–26
- [3] Nacher L S, Amoros J C, Moya M S and Martinez J L 2007 Mechanical properties of polyester resins in saline water environments *Int. J. Polym. Anal. Charact.* 12 373–90
- [4] Holbery J and Houston D 2006 Natural-fiber-reinforced polymer composites in automotive applications *JOM Journal of the Minerals*, Metals and Materials Society 58 80–6
- [5] Karus M, Ortmann S, Vogt G D and Kaup M 2003 Use of natural fibres in composites in the German and Austrian automotive industry-market survey 2002: status analysis and trends *J. Ind. Hemp* 8 73–8
- [6] Reddy K O, Reddy K R N, Zhang J, Zhang J and Varada Rajulu A 2013 Effect of alkali treatment on the properties of century fiber *J. Nat. Fibers* 10 282–96
- [7] Shanmugasundaram N and Rajendran I 2016 Characterization of raw and alkali-treated mulberry fibers as potential reinforcement in polymer composites J. Reinf. Plast. Compos. 35 601–14
- [8] Mueller D H and Krobjilowski A 2003 New discovery in the properties of composites reinforced with natural fibers *J. Ind. Text.* 33
- [9] Rao K M M and Rao K M 2007 Extraction and tensile properties of natural fibers: vakka, date and bamboo Compos. Struct. 77 288–95
- [10] Edeerozey A M, Akil H M, Azhar A B and Ariffin M Z 2007 Chemical modification of kenaf fibers Mater. Lett. 61 2023-5
- [11] Thakur V K, Singha A S, Kaur I, Nagarajarao R P and Liping Y 2010 Silane functionalization of Saccaharum cilliare fibers: thermal, morphological, and physicochemical study Int. J. Polym. Anal. Charact. 15 397–414

- [12] Boynard C A and d'Almeida J R M 2000 Morphological characterization and mechanical behavior of sponge gourd (Luffa cylindrica)—polyester composite materials Polym.-Plast. Technol. Eng. 39 489–99
- [13] De Rosa I M, Kenny J M, Maniruzzaman M, Moniruzzaman M, Monti M, Puglia D, Santulli C and Sarasini F 2011 Effect of chemical treatments on the mechanical and thermal behaviour of okra (Abelmoschus esculentus) fibres *Compos. Sci. Technol.* 71 246–54
- [14] Nagaraja Ganesh B and Muralikannan R 2016 Extraction and characterization of lignocellulosic fibers from Luffa cylindrica fruit Int. J. Polym. Anal. Charact. 21 259–66
- [15] Ghali L, Msahli S, Zidi M and Sakli F 2009 Effect of pre-treatment of Luffa fibres on the structural properties Materials letter 63 61–3
- [16] Altınışık A, Gür E and Seki Y 2010 A natural sorbent, Luffa cylindrica for the removal of a model basic dye J. Hazard. Mater. 179 658-64
- [17] Kalusuraman G, Siva I, Jappes J T and Kumar S A 2016 Effects of fiber surface modification on the friction coefficient of luffa fiber/polyester composites under dry sliding condition J. Polym. Eng. 36 837–46
- [18] Beakou A, Ntenga R, Lepetit J, Ateba J A and Ayina L O 2008 Physico chemical and microstructural characterisation of rhectophyllum camerunse plant fibre Composite Part A 39 67–74
- [19] D'Almeida J R M, Aquino R C M P and Monteiro S N 2006 Tensile mechanical properties, morphological aspects and chemical characterization of piassava (Attala funifera) fibers *Composite Part* A 37 1473–9
- [20] Pearl I A (ed) 1967 The Chemistry of Lignin (New York: Marcell Dekker) p 339
- [21] Conrad C M 1944 Determination of wax in cotton fiber. A new alcohol extraction method Industrial & Engineering Chemistry
 Analytical Edition 16745–8
- [22] Sreenivasasn V S, Soma Sundaram S and Ravindran D 2011 Microstructural, physico-chemical and mechanical characterization of Sancevieria cylindrica fibers—an exploratory investigation *Mater. Des.* 32 453–61
- [23] Kurschner K and Hoffer A 1933 Cellulose and cellulose derivate Fresenius. Journal of Analytical Chemical 92 145-54
- [24] Seki Y, Sarikanat M, Sever K and Durmuşkahya C 2013 Extraction and properties of Ferula communis (chakshir) fibers as novel reinforcement for composites materials Composites Part B: Engineering 44 517–23
- [25] Reddy K O, Maheswari C U, Dhlamini M S, Mothudi B M, Zhang J, Zhang J, Nagarajan R and Rajulu A V 2017 Preparation and characterization of regenerated cellulose films using borassus fruit fibers and an ionic liquid Carbohydrate Polym. 160 203–11
- [26] Storozheva E N, Sekushin V N and Tsyganenko A A 2006 FTIR spectroscopy evidence for the basicity induced by adsorption Catal. Lett. 107 185–8
- [27] Fiore V, Scalici T and Valenza A 2014 Characterization of a new natural fiber from Arundo donax L. as potential reinforcement of polymer composites Carbohydrate Polym. 106 77–83
- [28] Saravanakumar S S, Kumaravel A, Nagarajan T and Moorthy I G 2014 Effect of chemical treatments on physicochemical properties of Prosopis juliflora fibers Int. J. Polym. Anal. Charact. 19 383–90
- [29] Brígida A I S, Calado V M A, Gonçalves L R B and Coelho M A Z 2010 Effect of chemical treatments on properties of green coconut fiber Carbohydrate Polym. 79 832–8
- [30] Gomes A, Goda K and Ohgi J 2004 Effects of alkali treatment to reinforcement on tensile properties of curaua fiber green composites JSME International Journal Series A Solid Mechanics and Material Engineering 47 541–6