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To cite this article: A Udhayakumar *et al* 2023 *Mater. Res. Express* **10** 075508

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Materials Research Express

PAPER

Effect of chemical treatment on physico-chemical properties of a novel extracted cellulosic *Cryptostegia grandiflora* fiber[*]

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Abstract

The increasing global need to achieve sustainability in product development demands the use of biodegradable materials from renewable resources in many engineering applications. Accordingly, various natural fibers were explored as suitable reinforcement in polymer matrixes due to their low density and biodegradability. Hence, in this present work, a novel fiber reinforcement was extracted from the stem of the *Cryptostegia grandiflora* (CG) plant through a retting process and manual intervention. The extracted *Cryptostegia grandiflora* fibers (CGFs) were chemically treated using NaOH and silane. Various properties like crystal structure, chemical composition, surface morphology, and thermal degradation were studied using x-ray diffraction (XRD, Fourier transform infrared spectroscopy (FTIR) Scanning electron Microscopy (SEM) and Thermogravimetric analysis (TGA). The increasing cellulose content and the removal of hemicellulose after the chemical treatment indicate the potential for this CG fiber as a better reinforcement element in polymers. The increasing trend of tensile strength was observed for the CG fiber in the following order: silane > NaOH > untreated conditions. Two-stage thermal degradation was observed in all the cases where the maximum thermal degradation was found at the silane-treated CG fibers. Based on their performance, the chemically treated CG fibres can be made into composites and used for structural applications.

1. Introduction

Nowadays, due to the increasing demand for petroleum resources, leading to rapid depletion and its adverse effects, the need for eco-friendly materials is indispensable. Hence, researchers, scientists and industrialists are increasingly using natural fibers as potential reinforcements in various polymer matrix composites [1]. The advantages of natural fibers include, but are not limited to, lightweight, nontoxic and biodegradability. Development of several biocomposite materials for construction, automotive, marine and other sectors is increasing, after being inspired by the need for ecologically accepted materials as well as awareness of environmental pollution, global warming and energy crisis [2–4]. Natural fibers are called plant fibers, which can be obtained from the stems, fruits, leaves and seeds of the plants. The plant fibers are composed of cellulose, lignin, hemicellulose, pectin and wax. Natural fibers mainly consist of cellulose, which provides structural

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[*] Some of the images used in figure 1 of this paper were previously published in another work by the same authors, but correct attribution was not provided by the authors in the version of this paper that was originally published.

stability, stiffness and strength. The crystallinity of natural fibers is based on their cellulose molecules [5]. In addition, the interfacial bonding between the matrix and the fibers makes a major contribution to the mechanical properties of the composites. The presence of hydroxyl groups (OH) in natural fibers reduces the effectiveness of load transfer between the matrix and the fibers and obstructs proper bonding with the hydrophobic polymer [6].

Besides, the high moisture absorption, low reactivity, hydrophilic nature and compatibility with a polymeric matrix are characteristics of lignocellulose fibers. These characteristics can be improved by applying chemical treatments, such as acetylation, alkalization and silane [7]. The treatments clean surfaces of natural cellulose fiber by removing impurities and non-cellulosic materials and activating the hydroxyl groups in their surfaces [8].

Both silane and alkali treatments have been reported to be the most efficient and extensively utilized treatments for improving the properties of fibers. Both surface treatments modify the microstructure of the natural fiber and increase the interfacial compatibility among the fiber and matrix [9, 10].

The alkali treatment is the most affordable method for reducing amorphous materials, including hemicellulose and lignin. It also improves the chemical composition, surface morphology, interfacial bonding, thermal stability, crystallinity index and mechanical properties of fibers [11]. It has been reported that treatment of natural fibers with 5% of NaOH improved fiber property changes [12]. The silane treatment is the most efficient method for reducing the volume of hydroxyl with the help of silane coupling agents. The presence of moisture in the fiber helps to form the silanols from the hydrolysable alkoxy group, and these react with the hydroxyl group to produce stable covalent bonds on the fiber surface. The hydrocarbon chains created by the silanols limit the swelling of the fiber [13].

There are many natural fibers extracted from different parts of plants and transformed into low-density polymer composites for various applications [14–18]. Most importantly, the performance of the composites is based on the functional properties of the reinforcement element, interfacial bonding between fiber and polymer, and the type of polymer matrix used for the composite fabrication. Hence, the need of the hour in the field of natural fiber-reinforced polymer composites is to explore and characterize natural fibers from various new plants. Therefore, a novel natural fiber from *Cryptostegia grandiflora* (CG) belonging to the *Apocynaceae* family has been discovered in this research. Its common name is rubber vine, which is native to southwest Madagascar. When it grows as a shrub and a vine, a rubber vine can reach a height of 2 and 30 meters, respectively [19–21]. Considering the aforementioned report on relevance of silane and alkali treatment to composite structures, surface morphology, thermal stability, chemical and mechanical properties of untreated as well as alkali and silane solution treated *Cryptostegia grandiflora* fibers (CGFs) were investigated in this present study. The properties of the treated and untreated fibers were compared, as obtained through the following methods: x-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, Scanning electron microscope (SEM), a universal testing machine, and thermogravimetric (TGA) analysis.

2. Materials and methods

2.1. Materials and extraction
CG plant stems were collected from Virudhunagar District, Tamil Nadu, India. The fiber extraction process was carried out manually by a manual peeling process, after soaking the fiber in water for ten days at a room temperature. To eliminate excess wetness and safeguard the fiber from biological attacks, the fibers were washed with running water after separation and exposed to Sunlight for two days. Afterwards, various analyses were performed on the dried fibers. Figure 1 shows the process flow of fiber extracted from CG plant stem to fiber treatment.

2.2. Chemical treatments
2.2.1. Alkali treatment
To reduce the non-cellulosic and other contaminants present on surfaces of the CGFs, the dried CGFs were immersed in a 5% of NaOH solution for four hours at a room temperature. To prepare the fibers for further analysis, they were cleaned in running water and dried for three days [22].

2.2.2. Silane treatment
To prepare a silane solution, 2% of 3-aminopropyltrimethoxysilane was added to a mixture of ethanol and water in a ratio of 80:20. Acetic acid was used to adjust the pH value of the silane solution and maintained between 4 and 5. The fibers were then washed and dried for three days at a room temperature after being immersed in the silane solution for four hours [23].
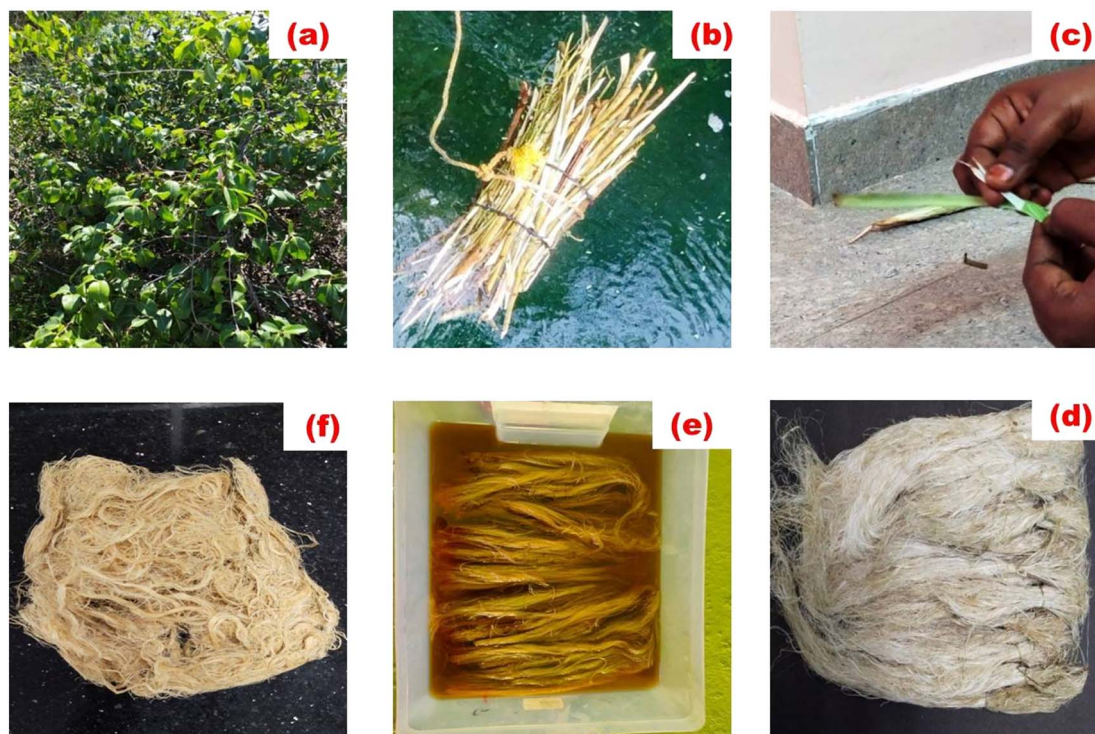
Figure 1. Fiber extraction process: (a) CG plant, (b) retting process, (c) CGF extraction, (d) extracted CGFs, (e) chemical treatment of CGFs and (f) treated CGFs. Reproduced from [1]. CC BY 4.0.

2.3. Optical microscope

The optical microscope model Leica DM750 M was used to measure the single CGF diameter at a room temperature. An accurate diameter of a natural fiber is difficult to obtain, because it is irregular in shape and non-uniform in thickness [24]. Therefore, the diameter at various locations of 10 samples had been measured and its average value had been reported.

2.4. Chemical analysis

The weight percentage (wt%) of the CGF cellulose was determined, using Kurshner and Hoffer's method, while the NFT 12-008 standard was used to measure the percentage of hemicellulose. The wax content was calculated by the Conrad method, and an electronic analyzer was utilized to estimate the moisture content. The density of CGF was calculated according to the ASTM D 2320-98 (2003) standard, while the ash content was measured, using the IS 199 method. The proportions of density, cellulose, hemicellulose, wax, lignin, pectin and ash of the CGF were similarly determined within the scope of this study. Five CGF samples were taken into consideration for each test, and the average results were reported.

2.5. X-ray diffraction (XRD) analysis

To study the crystallinity of the CGF, x-ray diffraction experiments were performed on both treated and untreated CGFs. The X-pert Pro diffractometer recorded data from $10°$ to $80°$ by generating monochromatic $CuK\alpha$ radiation with a wavelength of 0.154 nm. In addition, the crystallinity index (CrI) was computed, using equation (1).

$$CrI = \frac{H_{22.6} - H_{16.61}}{H_{22.6}} \tag{1}$$

Where $H_{22.6}$ and $H_{16.61}$ represent the height of the peaks at $2\theta = 22.6$ and $2\theta = 16.61$ [25].

2.6. Fourier transform infrared spectroscopy analysis

FTIR can be applied to discover the type of chemical bonds in the fiber specimen. The fine crystal of fiber was mixed with potassium bromide to make granules for testing through a Shimadzu spectrometer. A scanning rate of 32 scans per minute was used to record the spectrum at a resolution of 2 cm^{-1}, from 400 cm^{-1} to 4000 cm^{-1}.
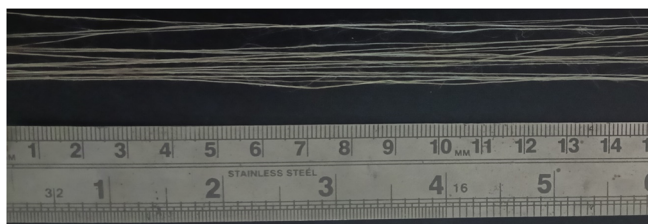
Figure 2. CGF **s**amples used for tensile test.

Table 1. Chemical composition of treated and untreated CGFs.

CGF	Density (g/c.c.)	Cellulose (%)	Hemi cellulose (%)	Pectin (%)	Lignin (%)	Wax (%)	Ash (%)	Moisture content (%)
Untreated	1.02	79.20	17.20	3.60	2.50	0.54	3.20	10.20
NaOH treated	1.47	82.60	12.10	0.09	1.50	0.28	2.40	11.70
Silane treated	1.30	87.40	11.80	1.50	1.00	0.31	1.30	8.30

2.7. Tensile testing

According to ASTM D 3822-07 standards, the tensile strength of the CGF was obtained, using a universal testing machine (Zwick Roell) with a cross head speed of 5 mm min^{-1}, the ambient temperature of 28 °C and the relative humidity of 65%. The total length of 150 mm fiber length was used for the testing as shown in figure 2. A scriber was used to mark the gauge length of 50 mm to determine the maximum stress and strain. In order to ensure the accuracy of the data, the CGF sample was replicated 25 times, and average values were reported.

2.8. Thermal analysis

The thermogravimetric (TG) technique was conducted as per ASTM E1131 to determine the thermal stability of CGFs. At a temperature between 30 °C and 700 °C and a heating rate of 5 °C min^{-1}, the Perkin Elmer STA 8000 model was employed to scan each sample, weighing about 6 mg. The inert atmosphere was controlled by passing pure nitrogen through the furnace at a rate of 19.8 ml min^{-1}.

2.9. Surface morphology analysis

To evaluate the changes on the CGF surfaces caused by alkali and silane treatments, the morphology of the treated and untreated CGFs were compared, using a CARL ZEISS model scanning electron microscope (SEM) with 1.5 nm resolution.

3. Results and discussion

3.1. Physical and chemical analyses

Table 1 summarizes the chemical composition of untreated, alkali (NaOH) and silane treated CGFs. Methods of fiber extraction from plants, plant age, growing climate and soil characteristics are significant factors that determine the chemical and physical properties of natural fibers [26]. The mechanical characteristics of natural fibers mainly depend on the cellulose content of the fiber. While comparing the cellulose content of the treated CGFs with that of the untreated fibers, the cellulose content of the silane treated CGFs increased approximately by 10%, due to surface modification. Chemical treatments, such as silane and NaOH reduced the hydrophilic behavior of the fibers by reducing the percentages of hemicellulose, pectin and lignin in all the modified CGFs, when compared with the untreated fibers. The percentage of wax in all the modified fibers was lower, when compared with the untreated fibers; it has been utilized in composites to confirm and enhance the bonding strength between matrix and fibers [27]. The decrease in the percentage of ash has a substantial impact on the mechanical properties of the treated fibers. The densities of silane and NaOH treated CGFs increased by 27 and 44%, respectively, when compared with the untreated counterpart. The improvement in density values of chemically treated CGFs was achieved by removing some of the hemicelluloses and lignin from the fibers as well as by the densification of the cell walls [28]. Through optical microscope, diameter of the CGF was determined which is shown in figure 3. When compared with the untreated fibers, the diameters of the chemically treated

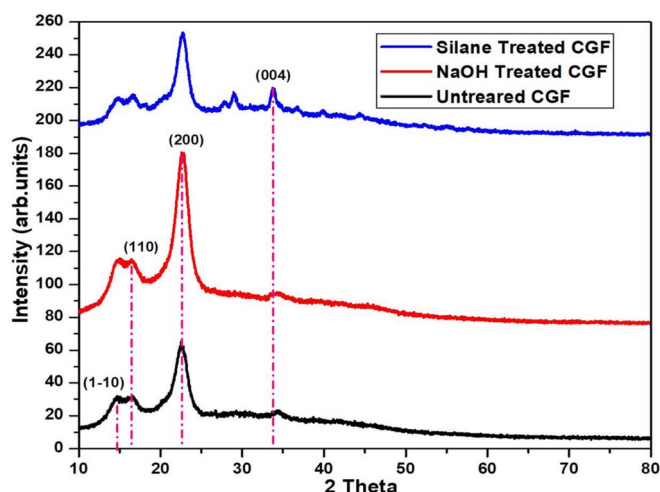**Figure 3.** A CGF diameter, measurement taken with optical microscope.

Figure 4. Treated and untreated CGF XRD—spectra.

fibers were more consistent and smaller. Silane treatment provided better results when compared with the alkaline treatment.

3.2. XRD analysis

Figure 4 shows the x-ray pattern diffraction for the treated and untreated CGFs, which exhibited significant peaks at 2θ angles of approximately 14.72, 16.64, 22.58 and 34.24, respectively. The above angles corresponded to the lattice planes of the cellulose I crystallites, peaks 1 (1–10), 2 (110), 3 (200), and 4 (004), respectively [29, 30]. The prominent peaks 1, 2 and 3 were the cellulose I characteristic peaks [31, 32]. Due to their full width at half-maximum, peaks 1 and 2 seemed to overlap and appear as a single broad peak [33]. According to the XRD analysis, the surface treatments did not seem to impact the transition from cellulose I to II polymorphically. The determined CrIs of silane and NaOH treated and untreated CGFs were 69.95, 66.34 and 62.00%, respectively. The non-crystalline materials, such as hemicellulose or lignin were partially removed by the chemical treatment of the natural fiber. This might allow cellulose fibers to acquire a more crystalline structure [3]. The surface treatment rearranged the cellulose molecules and caused the degradation of amorphous constituents in the natural fiber, which might have been responsible for the increase in the crystallinity of the plant fibers. Finally, the mechanical properties improved by enhancing the crystallinity of the natural fiber [8].
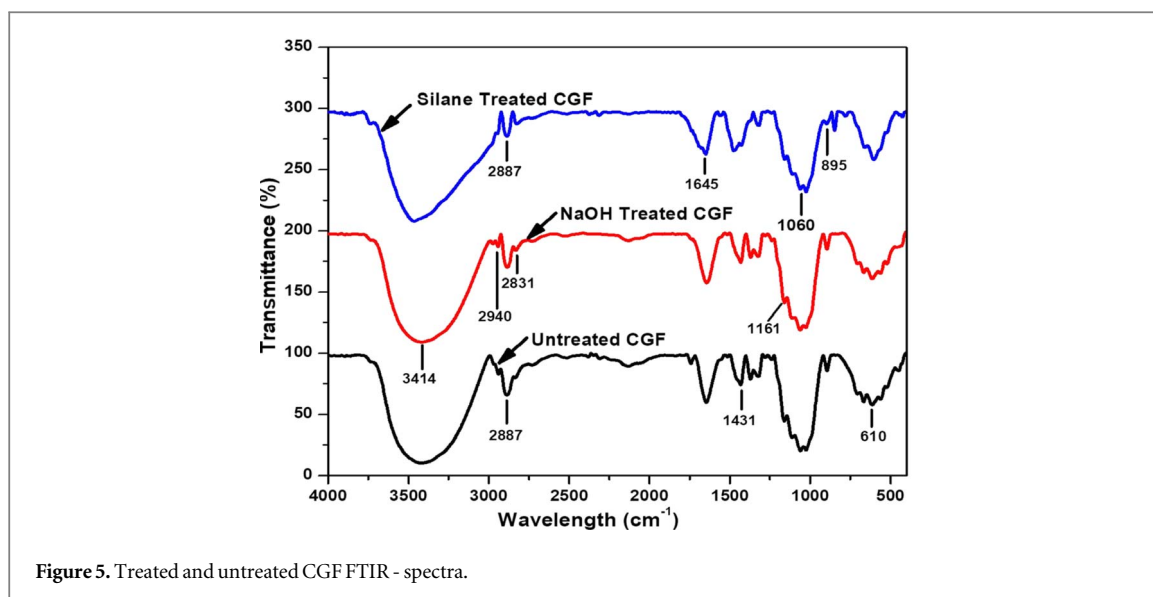
Figure 5. Treated and untreated CGF FTIR - spectra.

3.3. FTIR analysis

The FTIR spectra of both treated and untreated CGFs are shown in figure 5. Both NaOH treated and untreated spectra show a significant absorption band at 3414 cm^{-1}, which can be attributed to the OH stretching vibrations of hydrogen bonded hydroxyl groups from cellulose [30]. The peak concentration of 2940 cm^{-1} caused the CH stretching vibration in cellulose and hemicelluloses, although the intensity was reduced in optimally treated fibers [34]. For untreated and treated fibers, the peak at 2887 cm^{-1} was identified, as associated with the CH$_2$ group of cellulose and hemicellulose, which corresponded to CH stretching [35]. The band of 2831 cm^{-1} represented the bending of the CH$_2$ hemicellulose component of the chemical treated CGFs [36]. At 1645 cm^{-1}, a wide and strong peak caused by C=O stretching vibration in the lignin was observed [37]. CH$_2$ bending vibration in the cellulose was associated with a peak of 1431 cm^{-1} [38]. The C–O–C asymmetrical stretching of cellulose was responsible for the distinctive peaks at 1161 cm^{-1} [39]. The symmetric C–OH stretching in lignin was represented by the noticeable peak at 1060 cm^{-1} [25]. When silane was adsorbed on to the fibers, the peak signals at 1024 cm^{-1} were observed to be the symmetric C–OH stretching of lignin [34]. The silanol groups formed during the hydrolysis of silane were responsible for the observed peak at 895 cm^{-1} with the silane treated sample [40]. The wide absorption band at 610 cm^{-1} was recorded, as caused by the OH bending of the cellulose [41].

3.4. Tensile testing

The tensile strength of the treated and untreated CGF are shown in figure 6 and presented table 2, respectively.

The tensile strengths of the untreated, alkali and silane treated CGFs were 791, 853 and 994 MPa, respectively, which showed that chemically treated CGFs treated significantly exhibited an increased mechanical property. The chemical treatments reduced the compositions, including hemicellulose, lignin and pectin, which in turn improved the amount of cellulose, thereby improving the mechanical properties and crystallinity of CGFs [42]. The proper removal of non-cellulose constituents might increase elastic modulus and tensile strength of the natural fibers [12]. The stress transmission from matrix to natural fibers and significant improvement in interfacial adhesion were achieved by treating the fiber surface [43]. The reduction of impurities and more non-cellulosic composition caused by higher chemical composition triggered the degradation and breakage of CGFs by decreasing elongation [44]. This analysis showed that CGF strands are suitable reinforcing elements in various biocomposite structures.

3.5. Thermal analysis

Figure 7 depicts the TGA curve for the treated and untreated CGFs. Fiber degradation was described in three stages. The initial weight loss was observed during the first stage between 30 °C and 140 °C for both treated and untreated fibers. A weight loss of approximately 5.6% was estimated, due to surface water evaporation. It was observed on the untreated fiber at about 120 °C [3, 4]. In this first stage, the weight loss of silane treated CGF was 55.89% lower than the untreated and alkali treated CGFs, but alkali treated fiber recorded a weight loss that was mostly equal to the untreated fibers. It was evident that silane treated fibers became less hydrophilic. The second stage established the degradation of hemicellulose, lignin and cellulose at temperatures between 180 °C and 370 °C. During this second stage, the hemicellulose began to degrade before the cellulose, due to a lower degree
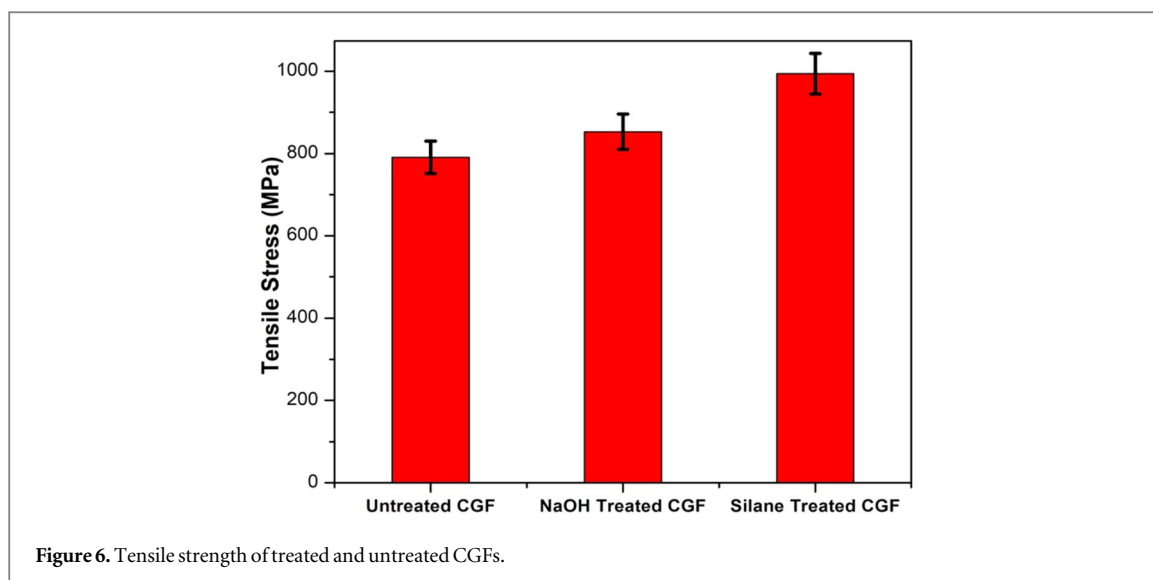
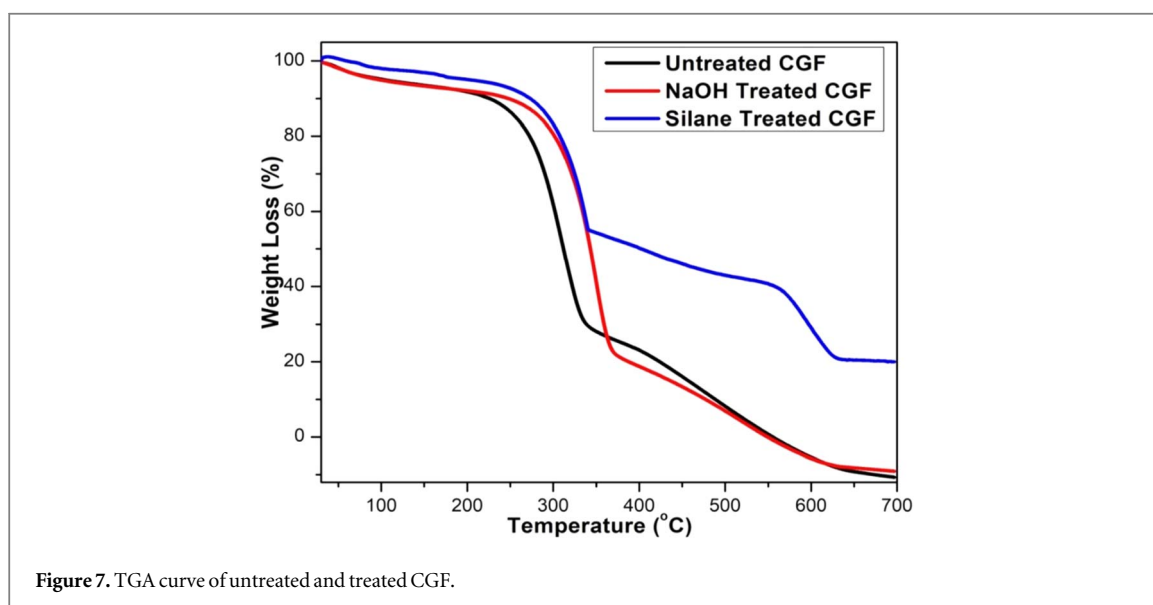**Figure 6.** Tensile strength of treated and untreated CGFs.

Figure 7. TGA curve of untreated and treated CGF.

Table 2. Tensile properties of untreated and treated CGFs.

CGF	Diameter (μm)	Tensile strength (MPa)	Elongation at break (%)
Untreated	117	791	3.1
NaOH treated	108	853	2.9
Silane treated	97	994	2.7

of polymerization [4, 35]. The weight loss of untreated fibers was 66.96%, which was 34.9% more than that of silane treated CGFs.

More also, the third stage occurred between 370 °C and 700 °C, which was related to the combustion of the degraded materials, lignin and wax decomposition. The lignin degradation started at a low temperature of 160 °C and extended to a wide range up to 900 °C [45]. The silanols present on the surface of the silane treated fibers improved the heat resistance and degradation temperature of the fibers, which shifted the peak degradation temperature of cellulose to a higher temperature. The maximum weight loss at 550 °C for silane treated fiber was 59.13% and the weight loss of untreated was 89.91%, while that of NaOH treated fiber was 99.20%, indicating a considerable improvement in the thermal stability, as the silane layer prevented structural damage to the CGFs [40].
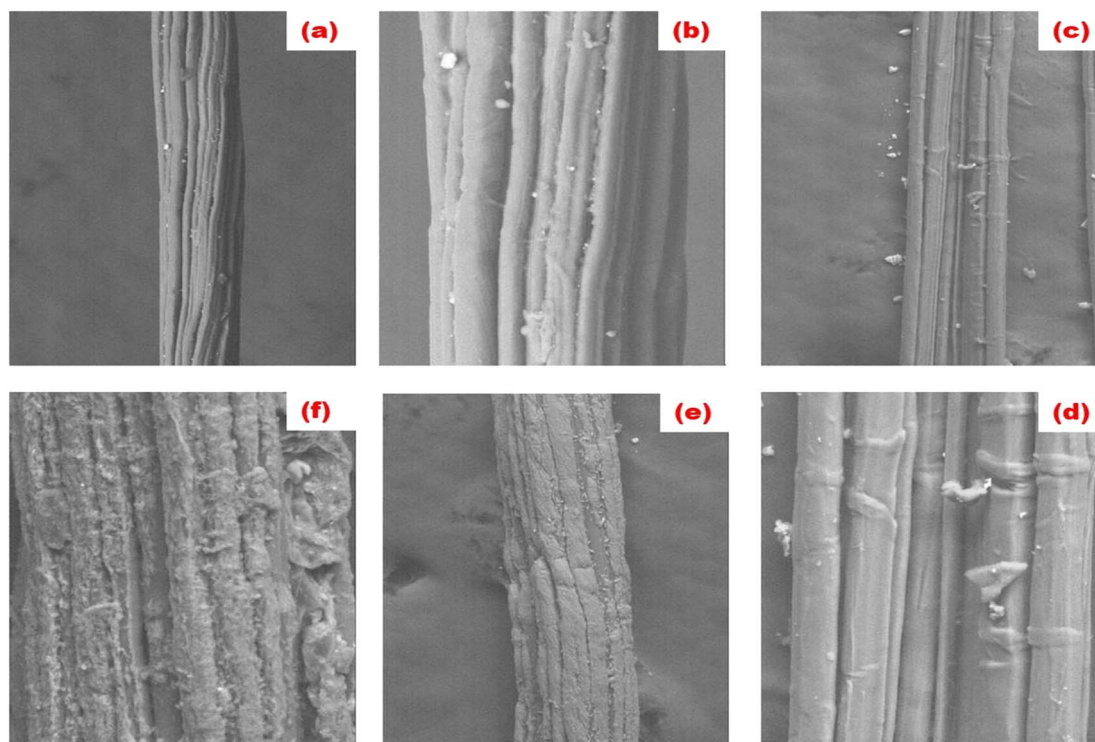
Figure 8. SEM images of: (a), (b) untreated CGF, (c), (d) NaOH treated CGF and (e), (f) silane treated CGF.

3.6. SEM analysis

The surface morphologies of treated and untreated CGFs were compared under SEM analysis. A morphological microscopic examination is best suited to describe the fiber surface structural changes. The fiber surface contributes an important role in increasing the interfacial bonding between the resin and the fibers, which improves mechanical properties of composites [46]. Figures 8(a) and (b) show an untreated fiber surface with many impurities. The surfaces of the fibers could be cleaned, smoothed or roughened through chemical treatments, such as alkali and silane.

When treating the fiber surface with alkali, the fiber surfaces were roughened more than the untreated fibers by reducing the hemicellulose, lignin and ash, as shown in figures 8(c) and (d) [44]. According to the previous study, the interfacial bond or strength of the sisal fibers was enhanced between the fiber and the matrix by surface roughening, aided by alkali treatment [47]. The silane treated CGFs in figures 8(e) and (f) exhibited a rougher and more uncontaminated surface than the alkali treated CGF surfaces. The ethanol/water present in the silane solution reduced the hemicellulose and pectin partially, and the fiber surface was roughened by chemisorbing the silanols. According to the results, the alkali and silane treated fibers recorded better mechanical properties than untreated fibers [3].

4. Conclusions

A novel reinforcement CG fiber was extracted and subjected to various testing and characterizations under alkali and silane-treated conditions. The optimum of 5% was used for the alkali treatment to avoid structural deterioration of the fiber surface. A maximum increase of 10% in cellulose was obtained in the silane-treated condition, which may have happened due to the removal of holocellulose, the waxy layer, and surface impurities. The removal of hemicellulose was clearly visible through the absence of peaks in the FTIR result. The crystallinity index for the CG fiber was found in the following decreasing order: silane (69%) < NaOH (66%) < untreated (62%). The removal of weak amorphous constituents leads to the exposure of crystalline structure in the form of fibrils. The tensile strength of silane and NaOH-treated fibers exhibited a 25% and 7% increase, respectively, in comparison with the untreated CG fiber. The maximum thermal degradation was found in the untreated case compared to alkali and silane-treated CG fibers. The maximum thermal resistance was found in the case of silane-treated fibers which is 50% higher at the elevated temperature. Summarizing this investigation, CGF can be used as a potential reinforcement in various biopolymeric composites that can be used in the aviation, aerospace, sports, and automotive industries.

Acknowledgments

Data availability statement

The data that support the findings of this study are available upon request from the authors.

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