EFFECT OF CHEMICAL TREATMENT ON THE PROPERTIES OF *CYNODON DACTYLON* ROOT FIBER

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Natural fiber composites have gained much interest for application in industries, such as shipbuilding (for small boats), automotive manufacturing, and aerospace. Researchers have been investigating the use of natural fibers, such as alfa, jute, and sisal, as reinforcement in composite materials, due to their desirable physicochemical and mechanical properties. However, the hydrophilic nature of vegetable fibers presents a challenge, because it results in poor adhesion between the fibers and the polymer matrix in composites. This study focuses on the effects of alkaline treatment on the morphological structure of *Cynodon dactylon* plant root fiber. The treatment lasted 1 hour and used sodium hydroxide solutions at concentrations of 2%, 4%, 6%, and 8%. The fibers were characterized by XRD, FTIR, TGA, EDX and SEM analyses.

Keywords: composite, alkali treatment, *Cynodon dactylon*, adhesion

INTRODUCTION

Natural fiber-reinforced composites have recently gained popularity due to their low cost, low density, biodegradability, availability, ease of processing, and high specific modulus.[1-7] These benefits are particularly appealing for a wide range of applications in various fields, including everyday items (furniture, flooring, utensils) and the automotive industry, which require lightweight, high-performance materials with low environmental impact and material costs. However, some limitations prevent the use of natural fibers in the production of polymer matrix composites.

Natural fibers are hydrophilic because they are made up of lignocellulose, which contains hydroxyl groups.[8,9] As a result, they are incompatible with hydrophobic thermosetting materials like unsaturated polyester and have low moisture resistance. These two characteristics are significant drawbacks to using plant fibers as polymer reinforcements. As a result, these properties must be improved to avoid having a negative impact on the composite's mechanical performance.

Several studies have looked into changing the surface properties of cellulose fibers to improve adhesion to a polymer matrix and reduce moisture absorption. It has been demonstrated that proper fiber treatment can improve their compatibility with the polymer matrix, resulting in better adhesion and, as a result, higher-quality composites. For example, Benyahia *et al*.[10] used chemical treatments, such as mercerization, permanganate (0.02%), acetylation, and bichromate (0.02%), to modify Alfa fibers, with a view to enhancing the mechanical properties of composites reinforced with them. FTIR analysis of the fibers revealed a decrease in the number of hydroxyl groups (-OH), also, a 20.53% increase in the crystallinity index (CrI) was obtained after alkaline treatment. SEM observations of the fractured surfaces of the prepared composites revealed that the various chemical treatments of the fibers resulted in improved matrix-fiber adhesion.

In another study,[11] Alfa fiber was treated with 5% (w/v) sodium hydroxide for various durations (1, 3, 5, and 24 hours). The FTIR results revealed that treating Alfa fibers with a 5% alkaline solution resulted in the partial removal of hemicelluloses and lignin. Thus, following the treatment, the fibers' CrI values increased, the highest CrI – of 41.3% – being recorded for Alfa fibers treated with the 5% alkali solution for 5 hours, which represented a 33% increase over untreated fibers.

Mouissa *et al*.[12] investigated the effects of chemical treatments of wood waste on the mechanical and thermal properties of a clay-based composite. The wood waste was treated with solutions of NaOH (5 wt%) and $KMnO_4$ (0.01 wt%). As the alkaline treatment caused the removal of hemicelluloses, the CrI of sawdust treated with NaOH and $KMnO_4$ increased to values of 45.96% and 39.19%, respectively, compared to that of the untreated sawdust, which was 17.89%. Barrera-Fajardo *et al*.[13] investigated the matrix-fiber adhesion in chemically treated Colombian banana and coconut fibers embedded in polylactic acid (PLA) and unsaturated polyester resin (UPR) matrices. Both types of fibers were treated with a 5% NaOH solution for one hour. FTIR spectra showed a decrease in hemicellulose and lignin contents, as well as the removal of waxy substances from the surface of the treated banana and coconut fibers.

This study explores a new type of fiber derived from the roots of *Cynodon dactylon* plant, an invasive species abundantly found worldwide. Varying concentrations of NaOH (2%, 4%, 6%, and 8% w/v) were tested to extract the fibers from the raw lignocellulosic roots. The obtained fibers were characterized morphologically and structurally, as well as in terms of their crystallinity and thermal behavior, to investigate their potential use as reinforcement in thermosetting polymer composites.

EXPERIMENTAL

Raw material

Roots of *Cynodon dactylon* were collected in the M'sila region (Algeria) in May 2024. The roots were washed with tap water to eliminate dust and contaminants, and then air-dried for three days at ambient temperature.

NaOH treatment of *Cynodon dactylon* roots

The roots of the *Cynodon dactylon* plant underwent several processing steps. First, they were thoroughly washed with distilled water to remove any impurities and surface contaminants. The cleaned roots were then air-dried at room temperature for 48 hours before being placed in an oven at 80 °C for 24 hours to eliminate residual moisture. After drying, the roots were treated with 2%, 4%, 6%, and 8% (wt) NaOH solutions for 1 hour at room temperature, maintaining a solid-to-liquid ratio of 1:15 (w/v). The residual NaOH was neutralized using a 2% sulfuric acid solution in distilled water for 10 minutes. The samples were then repeatedly rinsed with distilled water until a neutral pH was achieved. Finally, the treated material was dried in an air oven at 105 °C for 5 hours. Table 1 presents the codes assigned to the studied samples.

Table 1
Coding of different samples prepared

Code	Sample
UCD	Untreated *Cynodon dactylon* roots
ATCD21	Alkaline treated *Cynodon dactylon* with 2% NaOH for 1 h
ATCD41	Alkaline treated *Cynodon dactylon* with 4% NaOH for 1 h
ATCD61	Alkaline treated *Cynodon dactylon* with 6% NaOH for 1 h
ATCD81	Alkaline treated *Cynodon dactylon* with 8% NaOH for 1 h

Characterization of root-derived fibers

X-ray diffraction analysis (XRD)

XRD analysis was performed using an X'Pert High Score PW3209 diffractometer to assess the effects of the treatments on fiber crystallinity. The diffraction patterns (2θ) were recorded from 10 to 40° using Cu-Kα radiation at 40 KV and 20 mA. The crystallinity index (CrI) was calculated using Segal's empirical method:[14]

$$CrI\ (\%) = 100X\ \frac{I_{002} - I_{am}}{I_{002}} \quad (1)$$

where *CrI* is the crystallinity index, *I(002)* is the intensity of the (002) plane crystal phase at 2θ = 22°, and I_{am} is the intensity of the amorphous phase at 2Θ=17°.

Fourier transform infrared spectroscopy (FTIR)

In this study, the infrared spectra of the plant materials before and after the alkali treatment were recorded using an FTIR (Shimadzu, Series 8300). The spectra were recorded using pellets of powdered plant material in potassium bromide. Transmittance was measured in the range of 4000 to 400 cm^{-1}.

Thermogravimetric analysis

Thermogravimetric analysis (TGA) was used to monitor the thermal stability of the fibers over time. The measurements were performed in a nitrogen atmosphere with thermal analysis equipment (SDT Q600 TA, USA), under heating from 20 °C to 700 °C, at a heating rate of 10 °C per minute.

Scanning electron microscopy (SEM)

Cynodon dactylon samples were analyzed with a JOEL JSM 7001F SEM (Japan). The SEM images were obtained using the secondary electron imaging method, at a beam acceleration voltage of 15 kV.

RESULTS AND DISCUSSION

XRD analysis

Figure 1 presents the X-ray diffraction patterns of both untreated and alkali-treated fiber samples. The CrI calculations provided in Table 2 revealed a significant increase in the crystallinity of alkali-treated *Cynodon dactylon* fibers, compared to the initial material. Specifically, the crystallinity increased significantly, reaching a maximum of 70.8% for sample ATCD81, followed by samples ATCD61, ATCD41, and ATCD21. This increase is attributed to the removal of amorphous constituents, such as lignin, pectins and hemicelluloses, resulting in better compaction of cellulose microfibrils and relaxation of internal stresses.[15-19] The removal of these amorphous components also exposes the internal fibrillar structure and increases the roughness of the fiber surface.[20-22] In a potential use of the fibers as composite reinforcement, this modification promotes the mechanical anchoring of the thermoset matrix, thus strengthening the cohesion at the fiber-matrix interface.

The CrI values obtained for alkali-treated *Cynodon dactylon* fiber samples ATCD61 and ATCD81 are comparable to those reported in the literature for other natural fibers, including elephant grass (62.4%), flax (70%), sisal (75%), coconut (68%), *Acacia planifrons* (65.38%), *Sansevieria cylindrica* (60%), *Althaea officinalis* (68%), and luffa sponge (59.9%) fibers.[23-25] This similarity allows the hypothesis that *Cynodon dactylon* root-derived fibers could be an effective reinforcement for thermoset matrix composites, providing improved mechanical performance through better fiber-matrix adhesion.[26]

Table 2
Crystallinity index (CrI) of untreated and alkali-treated *Cynodon dactylon* root-derived fibers

Sample	I 002	I am	Cr I (%)
UCD	440.23	326.56	25.82
ATCD21	1765.32	1079.15	38.87
ATCD41	580.94	309.82	46.67
ATCD61	859.5	268.72	68.73
ATCD81	901.98	196.54	78.21

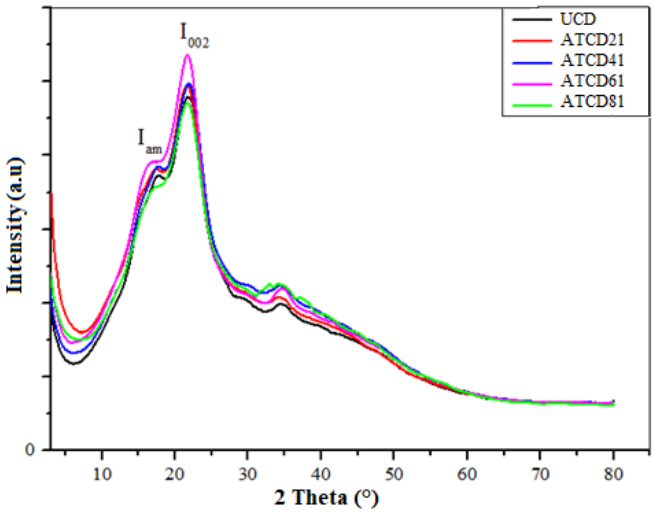

Figure 1: X-ray diffraction patterns of untreated and alkali-treated *Cynodon dactylon* root-derived fibers

FTIR spectroscopy

FTIR spectroscopy can be used to identify the composition of the fibers (lignin, cellulose, and hemicelluloses), as well as the functional groups (ketone, alcohol, and ester).[27-29] Figure 2 shows the FTIR spectra of the raw material and alkali-treated *Cynodon dactylon* fibers. The broad absorption band at 3390 cm^{-1} is attributed to the hydroxyl group (OH) bound to cellulose and hemicelluloses.[30] In all the spectra, a prominent absorption band was observed between 2900 and 2850 cm^{-1}, which is associated with the symmetrical and asymmetrical C-H stretching vibrations of alkyl groups present in the cellulose and hemicellulose components of natural fibers.[31-33]

The absorption bands at 1630 cm^{-1} and 1370 cm^{-1} indicate asymmetric stretching of lignin components' acetyl groups (C=C and C-O, respectively).[34] The intensity of these peaks (1370 cm^{-1} and 1630 cm^{-1}) decreased in the spectra of the treated fibers of *Cynodon dactylon* (ATCD21, ATCD41, ATCD61, ATCD81), compared to that of the initial material, indicating some lignin and hemicellulose removal. The peak at 1050 cm^{-1} is due to hemicelluloses C-O bond vibration.[35] The reduction in the peak at 1050 cm^{-1} indicates that the alkaline treatment removed the hemicellulose components from the samples.

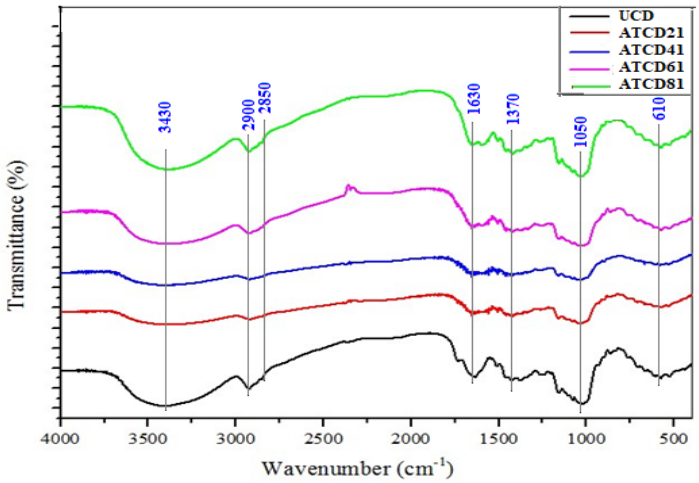

Figure 2: Infrared spectra of initial material and alkali-treated *Cynodon dactylon* fibers

SEM and EDX analysis of *Cynodon dactylon* fibers

Figure 3 presents the micrographs of the root-derived fibers treated with 2, 4, 6, and 8 wt% NaOH. It can be observed that different NaOH concentrations resulted in varying degrees of surface modification. Hemicelluloses, lignin, waxes, oils, and surface impurities were removed from the fiber surface. It has also been reported elsewhere[36-38] that the above-mentioned substances on the fiber surface are soluble in NaOH aqueous solution. According to Alawar *et al*.,[39] this effect is attributed to the increasing severity of the reaction as the NaOH concentration rises. When the plant material was treated with 2 and 4 wt% NaOH, some impurities remained visible on the surface, as shown in Figure 3 (b and c). In contrast, the images in Figure 3 (d and e) depict a much cleaner surface, with a noticeable absence of impurities. In addition, individual fibrils are more visible. The alkali treatment is believed to have cleared the micro-pores in fibres and roughened the fibre surface.[40-42]

Figure 4 shows the EDX spectra of the raw material and the alkali-treated *Cynodon dactylon* fibers. Carbon (C) and oxygen (O) are found in higher concentrations in the samples than other elements, such as silicon and magnesium (Table 3). Trace levels of sodium were detected in the treated samples (ATCD21, ATCD41, ATCD61, and ATCD81) as a result of the treatment with the sodium hydroxide solution (Fig. 4 (b, c, d and e)). The presence of sodium is more significant with higher concentration alkali treatment of the fibers.[28,43,44]

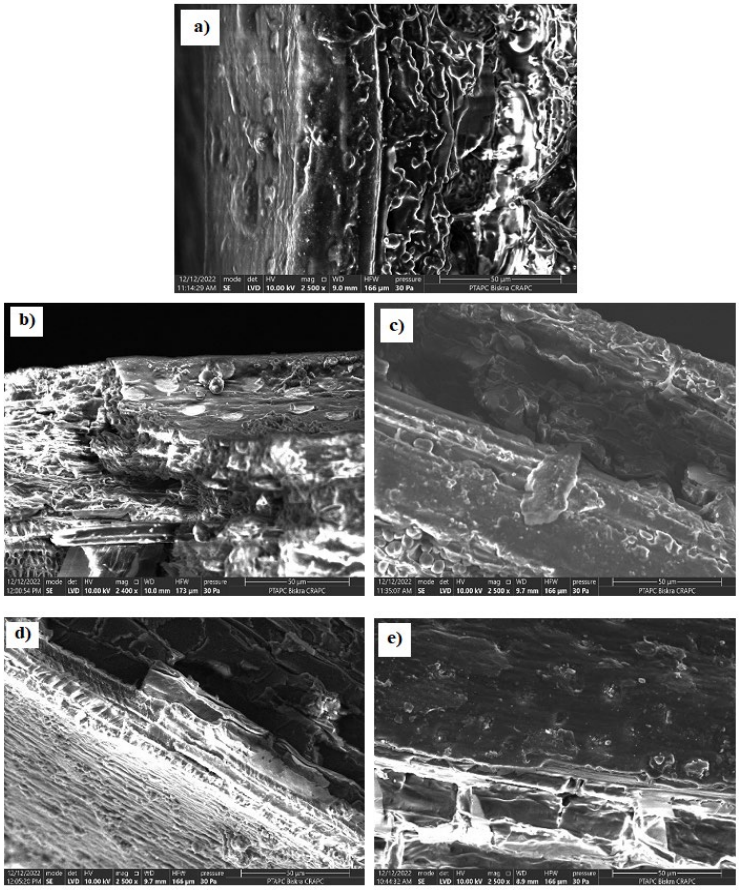

Figure 3: SEM images of (a) UCD; (b) ATCD21; (c) ATCD41; (d) ATCD61; (e) ATCD81

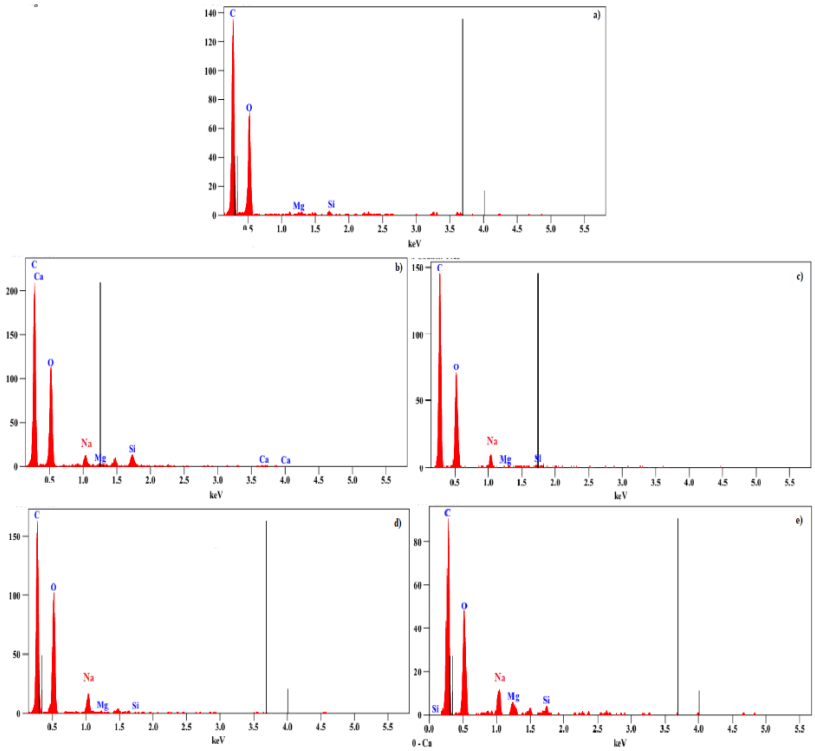

Figure 4: EDX spectra of (a) UCD; (b) ATCD21; (c) ATCD41; (d) ATCD61; (e) ATCD81

Table 3
Weight % and atomic % of various elements present in raw material and alkali-treated *Cynodon dactylon* fibers

Elements	UCD		ATC21		ATCD41		ATCD61		ATCD81	
	wt %	at %	wt %	at %	wt %	at %	wt %	at %	wt %	at %
C	40.46	47.86	39.60	47.09	38.07	46.70	37.34	45.87	35.68	42.80
O	57.45	51.02	57.71	51.52	52.70	48.55	52.23	48.17	62.33	56.13
Mg	0.71	0.41	0.26	0.15	1.01	0.61	0.30	0.51	3.62	5.96
Si	1.39	0.70	2.43	1.24	7.00	3.67	0.76	1.49	2.35	4.47
Ca	-	-	-	-	1.22	0.45	-			

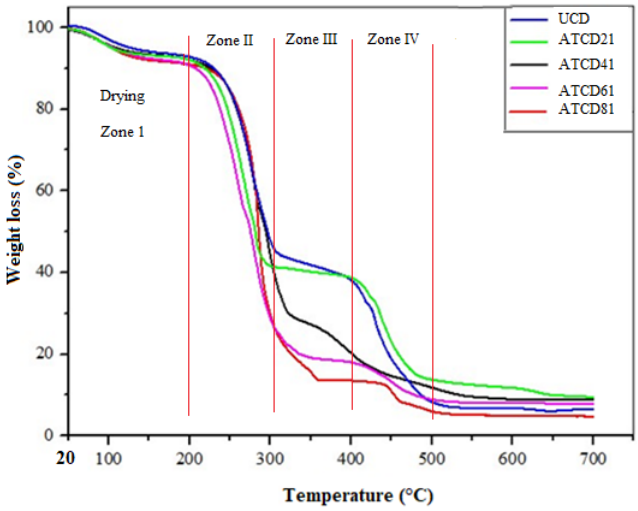

Figure 5: TGA curves of raw material and NaOH-treated *Cynodon dactylon* root-derived fibers at different concentrations (2, 4, 6 and 8%)

TGA analysis

The thermal decomposition behavior of *Cynodon dactylon* root-derived fibers treated with various sodium hydroxide concentrations, was analyzed using thermogravimetric analysis (TGA), compared to that of the initial plant material, at a heating rate of 10 °C/min in an inert atmosphere (Fig. 5). The TGA curves in Figure 5 highlight four degradation stages of the fibers. During the drying stage, from room temperature to 190 °C, the moisture and physically bound water in the fiber structure evaporated. At this stage, no significant weight loss was observed, with all samples exhibiting a similar weight reduction of approximately 9.03%. During the second degradation phase (zone II), between 200 at 300 °C, hemicelluloses and amorphous cellulose began decomposing. The third stage (zone III), between 300 at 400 °C, the degradation of crystalline cellulose is observed. In the last stage (zone IV), the breakdown of lignin occurs, it happens at high temperatures due to its complex structure. The untreated initial material, due to its higher lignin content, has greater thermal stability.[45] Increasing alkali concentration caused the removal of higher contents of lignin, thus leading to decreased thermal stability of the treated *Dactylon cynodon* root-derived fibers.

CONCLUSION

This study examined the chemical structure, morphology and thermal behavior of *Cynodon dactylon* root-derived fibers under the effect of alkali treatment. Experimental results led to the following conclusions. The treatment with sodium hydroxide (NaOH) caused the partial dissolution of lignin, pectins, hemicelluloses *etc*., as confirmed by infrared spectroscopy. The removal of amorphous components led to enhanced crystallinity index by approximately 67%, compared to the initial plant material. Scanning electron microscopy analysis further supported the removal of a significant amount of amorphous material, such as lignin, waxes, and contaminants, revealing a rougher fiber surface. These modifications are particularly beneficial for the application of fibers in composite reinforcement, a rougher fiber surface ensuring enhanced fiber-matrix adhesion, which is crucial for achiving good mechanical properties of polymer composites.

Thus, *Cynodon dactylon* root-derived fibers can serve as potential reinforcement for unsaturated polyester-based composites, which will be studied in further works, contributing to the development of bio-based materials.

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