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Effect of Alkali Treatment under Ambient and Heated Conditions on the Physicochemical, Structural, Morphological, and Thermal Properties of *Calamus tenuis* Cane Fibers

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Abstract: This study explores the effect of alkali treatment at ambient (25 °C) and elevated temperatures (100 °C) on the physicochemical, structural, morphological, and thermal properties of *Calamus tenuis* cane fibers (CTCFs) for the first time. Our purpose is to investigate their potential use as reinforcement in polymer composites, since cane fibers are generally known for their accurate and consistent geometrical orientation. Treatment with 8% (*w*/*v*) sodium hydroxide (NaOH) is carried out at ambient temperature and at 100 °C for 4 h. Chemical analysis and Fourier transform IR spectroscopy (FTIR) indicate some removal of non-cellulosic elements from CTCFs during alkali treatment, resulting in increased surface roughness, as confirmed by using SEM micrographs. This removal of non-cellulosic elements leads to an enhancement in the density of the treated CTCFs. Untreated and treated fibers are analyzed for maximum degradation temperature, thermal stability, and kinetic activation energy (E_a) using thermogravimetric analysis (TGA). In particular, E_a was considerably diminished with treatment and temperature. X-ray diffraction (XRD) results show an improved crystallinity index (37.38% to 44.02%) and crystallite size (2.73 nm to 2.98 nm) for fibers treated with 8% NaOH at ambient temperature. In conclusion, a general benefit was achieved by treating CTCFs, though the influence of increasing temperature treatment appears controversial.

Keywords: *Calamus tenuis* cane fibers; chemical analysis; alkali treatment; thermal analysis; morphological properties

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1. Introduction

Plant fibers are increasingly used in composites due to their availability, low cost, renewable (if not waste) origin, light weight, and specific mechanical strength, so have been proposed for use in industries ranging from automotive parts, sport equipment, furniture making, and even marine applications [1–4]. In particular, numerous ligno-cellulosic fibers, not included amongst the most diffused ones, have also been proposed for characterization in recent years, and this trend does not appear to lessen over time. Examples of these include Saharan aloe vera cactus leaves [5], areca palm leaf stalk [6], *Furcraea foetida* [7], *Ficus religiosa* tree root [8], ichu [9], cabuya [9], sisal [10], pineapple leaves [10], betel nut [10], okra [10], agave [11], and *Pongamia pinnata* bark [12], etc., which have been extensively studied and characterized by researchers. In many cases, these fibers have also been proposed as reinforcement materials in the fabrication of polymer composites. This trend

is even further fostered by the fact that increasing amounts of secondary raw materials originating from agricultural and food waste are available [13,14]. These are gradually and purposely redirected from a destiny of bare biomass combustion for energy recovery to comply with the objective proposed by the 2008/98 European Commission directive. The design of new composite materials is a possible strategy for this aim [15].

Plant fibers primarily consist of cellulose, hemicellulose, and lignin, with most of them being hydrophilic in nature [1,9,16]. Cellulose is a vital component of natural fibers, imparting excellent mechanical strength, structural stability, and toughness [1,9,12]. Additionally, the presence of lignin in these fibers offers protection against microbial attack [6,12]. However, a high concentration of hemicellulose and wax content negatively affects natural fibers, causing fiber disintegration and resulting in poor mechanical strength [1,9,12]. Furthermore, the hydrophilic nature of these fibers presents a challenge during composite fabrication, as they exhibit weak adhesion to hydrophobic polymer matrices. This lack of adhesion can lead to poor mechanical properties in natural fiber-reinforced polymer composites [12]. To overcome this issue, chemical modifications are often employed to enhance the adhesion between the fibers and the polymer matrix. These modifications create a rough surface on the fibers, facilitating a strong interlocking mechanism with the matrix. Several chemical treatments have been explored for this purpose, including alkylation, acetylation, silane treatment, potassium permanganate ($KMnO_4$) treatment, seawater treatment, and more [4,9,12,17,18]. Among the various chemical modifications, the alkali treatment process has emerged as the most effective and cost-efficient method for treating natural fibers [4,9,12,19]. Consequently, it is a somewhat standard procedure to start from alkali treatment using sodium hydroxide solutions on poorly investigated and/or newly applied lignocellulosic fibers, such as is the case of *Calamus tenuis* cane fibers, before expanding and diversifying the investigation to other treatments. When the fibers undergo alkali treatment with different concentrations under ambient and heated conditions, it removes lignin, pectin, waxy substances, and hemicellulose from the fiber surface. This treatment also leads to an improvement in the crystallinity index, thermal stability, tensile strength, flexural strength, and impact toughness of the fibers and their resulting composites [4,14]. It is noteworthy that these parameters are not only influenced by the NaOH concentration but also by the treatment time of the natural fibers. In practice, as known from early studies on plant fibers, together with plant fiber treatment, fiber length and the precision of their orientation also have a significant influence on the prospected final composites' performance [20].

In a prior study, Umashankaran and Gopalakrishnan reached the conclusion that treating *Pongamia pinnata* bark fiber with 5% (*w*/*v*) NaOH for 60 min led to significant improvements in its structural, morphological, thermal, and tensile properties [12]. This treatment increased the cellulose content from 62.34% to 71.32% and resulted in the highest values of the crystallinity index (42.43%), tensile strength (343.6 $\pm$ 13.04 MPa), and tensile modulus (12.71 $\pm$ 0.132 GPa) for the *Pongamia pinnata* bark fiber [12]. Similarly, Baskaran and colleagues discovered that treating *Dichrostachys Cinerea* bark fiber with 5% (*w*/*v*) NaOH for 90 min improved the structural and thermal properties of the fibers [14]. The alkali treatment increased the crystallinity index by 7.81% and raised the maximum degradation temperature from 359.3 °C (for raw fiber) to 370.3 °C (for optimally treated fiber) [14]. Additionally, Vijay and his team investigated the extraction and characterization of untreated and treated *Pennisetum orientale* grass fibers for potential use as reinforcement in polymer composites [21]. They found that treating the fibers with 5% NaOH resulted in the highest cellulose content (66.7%), crystallinity index (38.38%), and crystallite size (42.92 nm). Furthermore, the fibers treated with 5% NaOH exhibited greater thermal stability compared to both untreated and HCl-treated *Pennisetum orientale* grass fibers [21].

The authors found and reported that raw *Calamus tenuis* cane fibers (CTCFs) could be considered a promising candidate for reinforcement in polymer composites [22]. These fibers are obtained from a climbing palm belonging to the Arecaceae family and are commonly known as Assam cane in the market. This is also suggested because it is a plant that has further possible applications as a provider of cytotoxic and antioxidant action in phytotherapy, uses that offer large amounts of biomass waste that can be exploited in other production systems [23].

However, the uses are more widespread, and this suggests verifying the possible use of *Calamus tenuis* excess production and/or waste as the filler for polymer composites, in the form of short fibers. In particular, *Calamus tenuis* canes are extensively used in Northeast India for creating lightweight furniture, decorative items, and other products. Although significant progress has been made in various areas, there has been a lack of investigation into the alkali treatment of *Calamus tenuis* cane fibers (CTCFs). Thus, the primary purpose of this study is to examine the impact of alkali treatment on *Calamus tenuis* cane fibers (CTCFs) for the first time. This study involved a treatment using 8% (*w*/*v*) NaOH performed under two different conditions, at ambient temperature and at 100 °C, in both cases for a duration of 4 h. The untreated and treated samples were subjected to various analyses, including chemical analysis, Fourier transform infrared spectroscopy (FTIR) analysis, X-ray diffraction (XRD) analysis, thermogravimetric analysis (TGA), and scanning electron microscope (SEM) analysis. The significance of the treatment was proven by the fact that the fiber morphology was changed, enhancing their roughness and therefore possibly providing a stronger grip for a polymer matrix.

2. Materials and Methods

2.1. Materials

Calamus tenuis cane fibers (CTCFs) were sourced from the Naharkatia region (27.2870° N, 95.2476° E) in the district of Dibrugarh, Assam, India. Following extraction, the canes underwent a 14-day retting process aimed at promoting microbial degradation, as from [24]. Afterward, the canes were washed in tap water to remove impurities from the surface. The canes were then sun-dried for 8 days. For surface treatments, 98% pure sodium hydroxide (NaOH) pellets from Nice Chemicals (P) Ltd. in Kerala, India, were used.

2.2. Alkali Treatment of CTCFs

Calamus tenuis cane fibers (CTCFs) were subjected to two different treatments: one with 8% (*w*/*v*) NaOH at room temperature (32 °C) and another using the same tenor of alkali, but at 100 °C, both for a duration of 4 h. The selection of the 8% (*w*/*v*) NaOH concentration was based on the literature suggesting that it yields the most favorable improvements in fiber properties [25,26]. Following the treatment process, the canes were thoroughly rinsed with distilled water to remove any residual NaOH and subsequently sun-dried for a period of 8 days. Sample preparation for different analyses is displayed in Figure 1.

2.3. Characterization of Untreated and Treated CTCFs

2.3.1. Measurement of Bulk Density and Diameter of *Calamus tenuis* Canes

The bulk density (ρ_b) of untreated and treated *Calamus tenuis* canes was determined following the guidelines outlined in KSF 2198 [27]:

$$\rho_b = \frac{W}{V}. \quad (1)$$

In Equation (1), w represents the weight, and v denotes the volume of the sample.

The microscopy was used to measure the diameter of 20 samples of *Calamus tenuis* canes at three different locations, and the resulting measurements were averaged to obtain the final value.

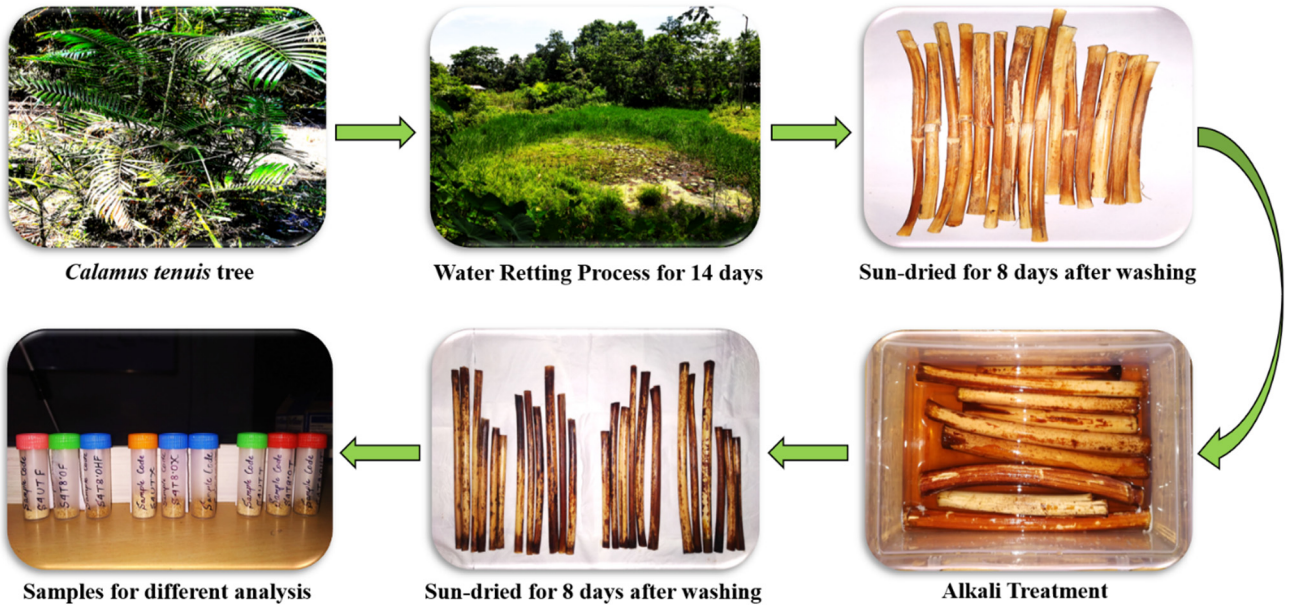

Figure 1. Sample preparation from the extraction of the canes from the *Calamus tenuis* tree, their retting, leading to the separation of the looser materials (leaves), then cutting, treating in water, sun-drying, and removal of the different samples for the various analyses for this study.

2.3.2. Chemical Analysis

Untreated and treated CTCFs were chemically analyzed using standard methods to determine the cellulose, hemicellulose, lignin, and ash content. The cellulose content in untreated and treated CTCFs was assessed using the Updegraff method [28]. The hemicellulose content was estimated through the natural detergent fiber (NDF) method [29]. Lignin contents were determined using the TAPPI T 222 om-02 method, while the ash contents were estimated using TAPPI T 211 om-02. All chemical analyses were performed in triplicate, and the average results are presented.

2.3.3. Fourier Transformed Infrared (FTIR) Analysis

The untreated and treated *Calamus tenuis* cane fibers (CTCFs) were subjected to Fourier transformed infrared (FTIR) spectroscopy to investigate the functional groups present in the samples. Test samples were prepared by mixing 1 mg of powdered *Calamus tenuis* cane fibers with potassium bromide (KBr), forming pellets. Subsequently, the analysis of these pellets was performed using a Nicolet Impact 410 FTIR Spectrometer (USA) with a 1 cm^{-1} resolution, covering the wavenumber range from 400 cm^{-1} to 4000 cm^{-1}.

2.3.4. X-ray Diffraction (XRD) Analysis

The crystalline properties of untreated and treated CTCFs were examined using a PANalytical Empyrean Powder XRD, equipped with a Cu low-frequency filter (LFF) X-ray lamp with a power of 600 W, with an angle range of 5° to 80° (2θ angle) and a step size of 0.026°. The generator was set at 45 kV, 40 mA, and operated at 25 °C. The crystallinity index (CI) of untreated and treated CTCFs was estimated using Segal's equation [30]:

$$CI = \left(1 - \frac{I_{am}}{I_{002}}\right) \times 100\%. \quad (2)$$

In Equation (2), I_{002} signifies the intensity of the peak observed at 22°, which corresponds to the (002) plane in the crystalline region. On the other hand, I_{am} denotes the intensity valley between the two peaks at approximately 18°, hence positioned between the planes (101) and (002) in the amorphous region.

Scherrer's equation was utilized to estimate the crystallite size (CS) of untreated and treated CTCFs [12].

$$\mathrm{CS} = \frac{K\lambda}{\beta COS\theta}. \quad (3)$$

In Equation (3), the symbol λ stands for the wavelength of CuKα radiation. Scherrer's constant is denoted by K, where K = 0.89. The symbol β is used to represent the full width at half maximum (FWHM) of the peak, and θ denotes Bragg's angle.

2.3.5. Thermogravimetric Analysis (TGA)

The degradation characterization of the samples was studied using a METTLER TOLEDO (Columbus, OH, USA) Thermogravimetric Analyzer. The analysis involved heating around 5 mg of powdered untreated and treated *Calamus tenuis* cane fibers (CTCFs) in an alumina crucible at a rate of 10 °C per minute. The temperature range for the analysis spanned from 25 to 700 °C, and a nitrogen atmosphere with a gas flow rate of 20 mL per minute was maintained throughout the process. The estimation of kinetic activation energy (E_a) involved graphing $\ln[-\ln(1-x)]$ against 1000/T using the Coats and Redfern equation [31].

$$\ln[-\ln(1-x)] = \ln\frac{ART^2}{\beta E_a} - \frac{E_a}{RT}. \quad (4)$$

In Equation (4), A represents the pre-exponential factor, β is the heating rate (10 °C per minute), R denotes the universal gas constant (8.32 kJ mol^{-1} K^{-1}), T is the absolute temperature in Kelvin (K), and $x = \frac{w_0 - w_t}{w_0 - w_f}$, where w_0 is the initial weight, and w_t represents the weight of the sample at a particular time. w_f denotes the final weight of the sample.

2.3.6. Scanning Electron Microscope (SEM) Analysis

The surface morphology of untreated and treated CTCFs was analyzed using a scanning electron microscope (SEM) (Model: JSM 6390LV by JEOL, Akishima, Tokyo, Japan). Prior to analysis, the samples were coated with gold.

3. Results

3.1. Density and Diameter of Untreated and Treated Calamus tenuis Canes

The bulk density of *Calamus tenuis* canes is determined using Equation (1). Untreated canes have a density of 526 ± 16 kg/m^3. Alkali treatment increases the density to 549 ± 12 kg/m^3 at ambient temperature and to 557 ± 18 kg/m^3 at 100 °C. The rise in density is a result of selectively removing amorphous components like hemicellulose, lignin, and pectin, thereby leading to an increase in cellulose content within the composition [32]. The diameter of the untreated and treated whole-cane CTCFs are also measured, and it is found that the diameter of the fiber decreases from 8.31 ± 0.48 mm to 7.56 ± 0.32 mm after alkali treatment. This suggests the removal of non-cellulosic elements such as lignin, wax, hemicellulose content, etc., from the surface of the fiber.

3.2. Chemical Analysis

To investigate the impact of alkali treatment under ambient and heated conditions on the fibers, we have evaluated the primary chemical constituents of both untreated and treated CTCFs. From Table 1, it can be clearly observed that the untreated CTCFs contained 31.06 ± 1.03% hemicellulose, 37.43 ± 1.40% cellulose, 28.42 ± 0.81% lignin and 4.11 ± 0.62% ash. This appears to be on the lower side of lignocellulosic fibers, compared to hard fibers such as coir, where the amount of cellulose in untreated fibers is between 33 and 42% [33]. In a previous study, other cane-based fibers, such as sugar cane bagasse, starting from 38% cellulose, were able to increase this content up to 61% via an enzymatic treatment [34]. In bamboo, the cellulose content is clearly higher, reaching more than 50%, which in itself does not suggest the application of a treatment is necessary, such as in other cane plants [35].

Table 1. Physical and chemical properties of untreated and treated CTCFs.

CTCFs Samples	Physical Properties		Chemical Properties			
	Bulk Density (kg/m^3)	Diameter (mm)	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)
Untreated	526 ± 16	8.31 ± 0.48	37.43 ± 1.40	31.06 ± 1.03	28.42 ± 0.81	4.11 ± 0.62
Treated at 25 °C	549 ± 12	8.06 ± 0.32	51.11 ± 1.62	21.57 ± 1.41	22.32 ± 1.01	4.79 ± 0.32
Treated at 100 °C	557 ± 18	7.56 ± 0.27	53.33 ± 0.78	19.31 ± 1.12	20.81 ± 0.58	5.87 ± 0.58

In our case, after treatment at ambient temperature and at 100 °C, the hemicellulose content decreased to 21.57% ± 1.41% and 19.31 ± 1.12%, respectively. Alkali treatment also reduced the lignin content to 22.32 ± 1.01% for the CTCFs treated at ambient temperature and 20.81 ± 0.58% for the CTCFs treated at 100 °C. This demonstrates the effectiveness of alkali treatment in removing non-cellulosic constituents from CTCFs. In contrast, the cellulose content significantly increased to 51.11 ± 1.62% after treatment at ambient temperature and 54.33 ± 0.78% for CTCFs treated at 100 °C, which may be due to the transformation of a little amount of hemicellulose to native cellulose, as is recognized, e.g., in [36]. The increased cellulose content may significantly enhance the tensile strength, crystallinity index, and thermal stability of the natural fiber [6,8]. The increased ash content from 4.11 ± 0.62% to 4.79 ± 0.32% for CTCFs treated at ambient temperature and to 5.87 ± 0.58% for CTCFs treated at 100 °C may be a consequence of the improvement in the cellulose content in the alkali-treated CTCFs [37,38].

3.3. Fourier Transform Infrared (FTIR) Analysis

Figure 2 displays the FTIR spectra of untreated CTCFs and treated CTCFs observed under ambient and heated conditions. Several distinct peaks are observed at specific wavenumbers. For example, the peak at 3419.69 cm^{-1}, corresponding to the stretching vibration of hydroxyl groups [39–42], decreases in intensity and shifts to a higher wavenumber after treatment with NaOH at ambient temperature and at 100 °C. This indicates a reduction in the presence of hydrogen bonds, leading to a decrease in the hydrophilic nature of the CTCFs [43]. Two moderate peaks at around 2928 and 2848 cm^{-1} represent the C-H stretching vibrations of hemicellulose and cellulose, respectively [44,45]. The untreated fibers exhibit a peak at 1739 cm^{-1} attributed to the stretching vibration of the C=O bond in hemicellulose [46], a typical occurrence in more cellulose-based fibers as well, or ester linkages of carboxylic groups in lignin [47], which is absent in the alkali-treated fibers. This confirms the partial removal of hemicellulose and lignin [48,49]. Peaks at 1634 and 1507 cm^{-1} correspond to the C=O linkage of hemicellulose and lignin, and the C=C stretching of the phenyl propane group in aromatic lignin, respectively [50,51]. The detection of peaks at 1375 and 1247 cm^{-1} indicates the presence of C-H groups of lignin and C-H bending of hemicellulose, respectively [52]. The intensity of these two peaks decreases and disappears after NaOH treatment, suggesting the effective removal of hemicellulose and lignin [6,37]. The wavenumber at 1072 cm^{-1} indicates the existence of C-O groups of cellulose [43]. The peaks detected at 1462 and 851 cm^{-1} are attributed to the CH_2 scissoring motion and C-O-C stretching at the β-glycosidic linkage of cellulose, respectively [40,41]. These peaks offer information about the crystalline and amorphous properties of cellulose, and are specifically pronounced in the CTCFs treated at 100 °C, indicating a disordered structure of cellulose [40]. At this stage, crystals are dissolved, hence cellulose I begins to lose its crystalline nature and transforms into cellulose II, which is amorphous, leading to the lower crystallinity index and reduced thermal stability of CTCFs treated at 100 °C.

3.4. X-ray Diffraction (XRD) Analysis

Figure 3 illustrates the XRD diffractograms of both untreated CTCFs and treated CTCFs, observed under ambient and heated conditions. The diffractograms of untreated and treated CTCFs show three prominent diffraction peaks. The most distinct peak at approximately 2θ = 22° corresponds to the (200) plane, indicating the presence of cellulose I [24,46–48]. Two other less intense peaks are observed at 2θ = 16° and 34.5°, belonging to

the (101) and (004) planes, respectively, as extensively reported in the literature on the XRD characterization of cellulose I-based fibers [49–51]. The crystallinity index of the fibers is evaluated using Equation (2). Initially, the untreated fibers have a crystallinity index of 37.38%. Treatment with 8% NaOH at ambient temperature (25 °C) increases the crystallinity index to 44.02%, while treatment with 8% NaOH at 100 °C decreases it to 41.43%, which is clearly seen in Table 2. The improvement in the crystallinity index is attributed to the removal of amorphous components, like hemicellulose, lignin, wax, etc., thereby increasing the presence of crystalline cellulose and reorganization of crystalline regions within the fibers. Conversely, as reported above, the reduction in the crystallinity index occurs because an 8% concentration of NaOH at 100 °C may dissolve the more external crystals [53]. The treated fibers also exhibit a slightly increased crystallite size, from 2.73 nm to 2.98 nm, resulting in reduced chemical reactivity and moisture absorption capacity. This suggests that despite the fairly high level of treatment applied, alkali was not able to penetrate the crystalline structure, which is likely to be attributed to the very dense packing. In other cases, e.g., with *Fimbristylis globulosa* straw, where the crystallite size is in the order of 10 nm, the penetration of the chemical into the structure has been demonstrated to be possible and harmful for the structural characteristics of the fiber [54].

3.5. Thermogravimetric Analysis (TGA)

The TGA and DTG spectra of both untreated CTCFs and treated CTCFs observed under ambient and heated conditions are depicted in the accompanying Figure 4a,b. The spectra reveal a distinct three-stage degradation pattern for both fiber types. In the initial stage (25 °C to 133 °C), weight losses of 11.25%, 16.92%, and 25.85% are observed for untreated fibers and fibers treated with 8% NaOH at ambient temperature and at 100 °C, respectively. This indicates the evaporation of moisture and volatile substances and highlights the enhanced hydrophobic nature of the treated fibers. The second stage (175 to 370 °C) results in weight losses ranging from 12% to 78% for untreated fibers, 19% to 85% for fibers treated at ambient temperature, and 28% to 85% for fibers treated at 100 °C. Within this range, two peaks are observed in the untreated fibers, occurring around 285.24°C (weight loss of 36.17%) and 317.09 °C (weight loss of 58.35%). The first peak represents the degradation of hemicellulose, cellulose glycosidic links, and loosely linked materials, such as pectin, and the second peak arises due to the decomposition of crystalline cellulose [40]. Interestingly, the treated CTCFs show only one peak between 260 °C and 320 °C, mostly indicating the removal of hemicellulose during alkali treatment [55]. This finding aligns with the FTIR spectra and SEM micrographs. The maximum degradation temperature of CTCFs observed in the main peak of alkali-treated fibers at ambient temperature and at 100 °C is decreased by 30.01 °C and 52.41 °C, respectively, while no early secondary peak, pertaining to hemicellulose, is observed. This shift indicates a decrease in thermal stability following the treatment, not due, as shown before, to alkali penetration into the crystal, but possibly to accelerated degradation due to the more uniform characteristics of the fibers. Also, according to Chen and his colleagues, the changes in crystallinity and the cellulose chain transformation may play a significant role in reducing thermal stability [56], while, as suggested by Raia et al. [57] and Ramasamy et al. [58], the reduction in the thermal stability is caused by the not completely selective removal of amorphous regions of the fibers such as lignin and hemicelluloses, which might also affect specific cellulose regions. A similar result was also reported for alkali-treated Juncus effusus fiber [4] and alkali-treated Raffia textilis fiber [59]. In the third stage (370 °C to 700 °C), weight loss is attributed to lignin degradation. At 700 °C, residual weights measure 18.20%, 26.90%, and 33.74% for untreated and treated fibers under ambient and heated conditions, respectively. By examining the slope of the graph in Figure 4c, the kinetic activation energy of the untreated and treated samples was calculated. The analysis reveals decreased kinetic activation energy from 57.74 kJ/mol to 54.03 kJ/mol when the fibers were treated with 8% NaOH at room temperature. Additionally, the value dropped further to 45.26 kJ/mol when the fibers were treated with 8% NaOH at a temperature of 100 °C. Overall, it can be suggested

that alkali treatment does slightly affect the stability of the fibers, despite the measured increase in crystallinity and the removal of non-structural material. This is also indicated by the limited variation, within experimental accuracy, of the slope in Figure 4c, measured between 264.68 and 287.08 °C, as from the peak degradation temperatures of the three samples. However, in view of a smoother yet still effective treatment, the possibility of applying alternative chemicals or else reducing the concentration of the alkali solution might be explored.

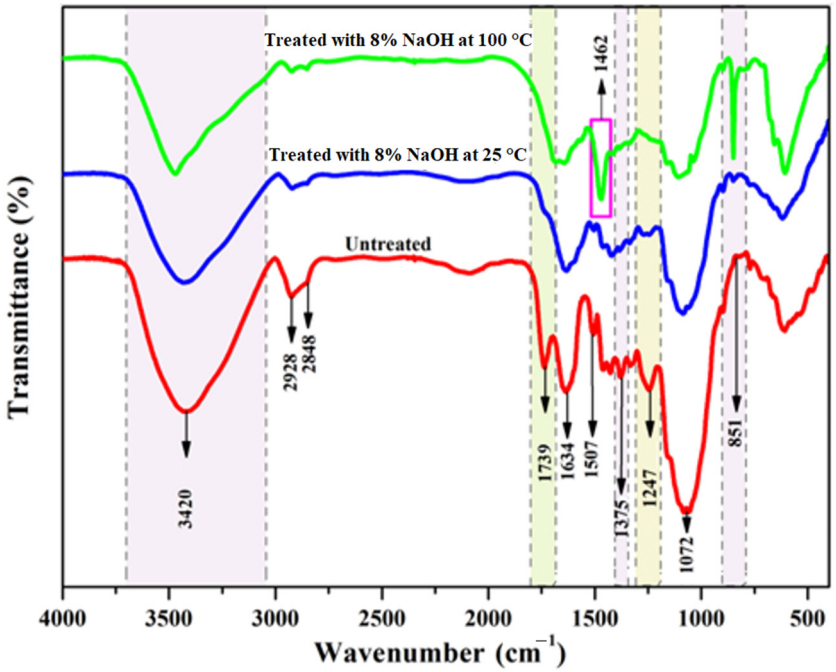

Figure 2. FTIR spectra of untreated and treated CTCFs at ambient temperature (25 °C) and at 100 °C.

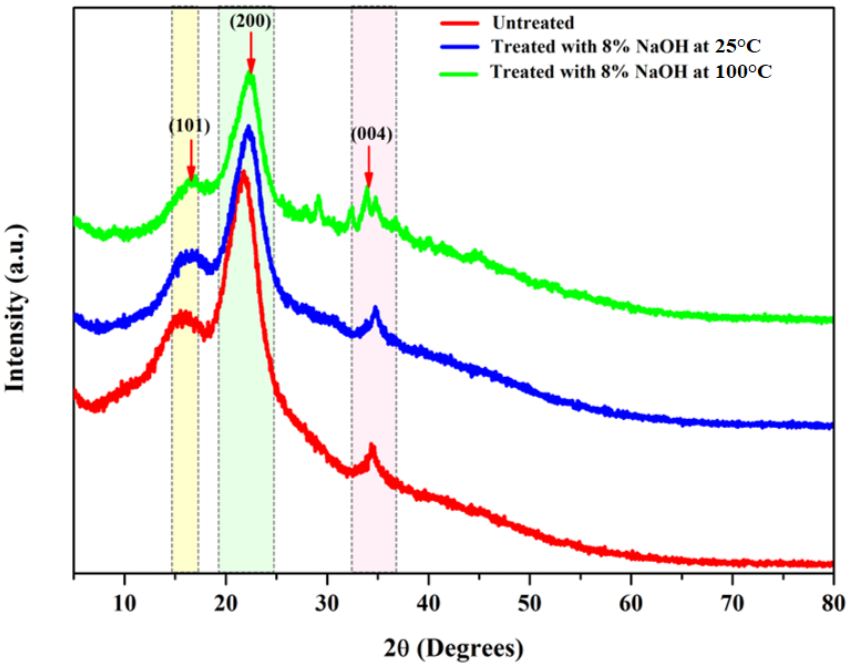

Figure 3. XRD diffractogram of untreated and treated CTCFs at 25 °C and at 100 °C.

Table 2. Crystalline properties of untreated and treated CTCFs.

CTCFs Samples	Crystallinity Index (%)			Crystallite Size (nm)		
	I_{002} (a.u.)	I_{am} (a.u.)	CI (%)	2θ (Degrees)	FWHM (Radians)	CS (nm)
Untreated CTCFs	5559	3481	37.38	21.67	0.05109	2.73
Treated at 25° C	4373	2448	44.02	22.08	0.04672	2.98
Treated at 100° C	3664	2146	41.43	22.18	0.05024	2.78

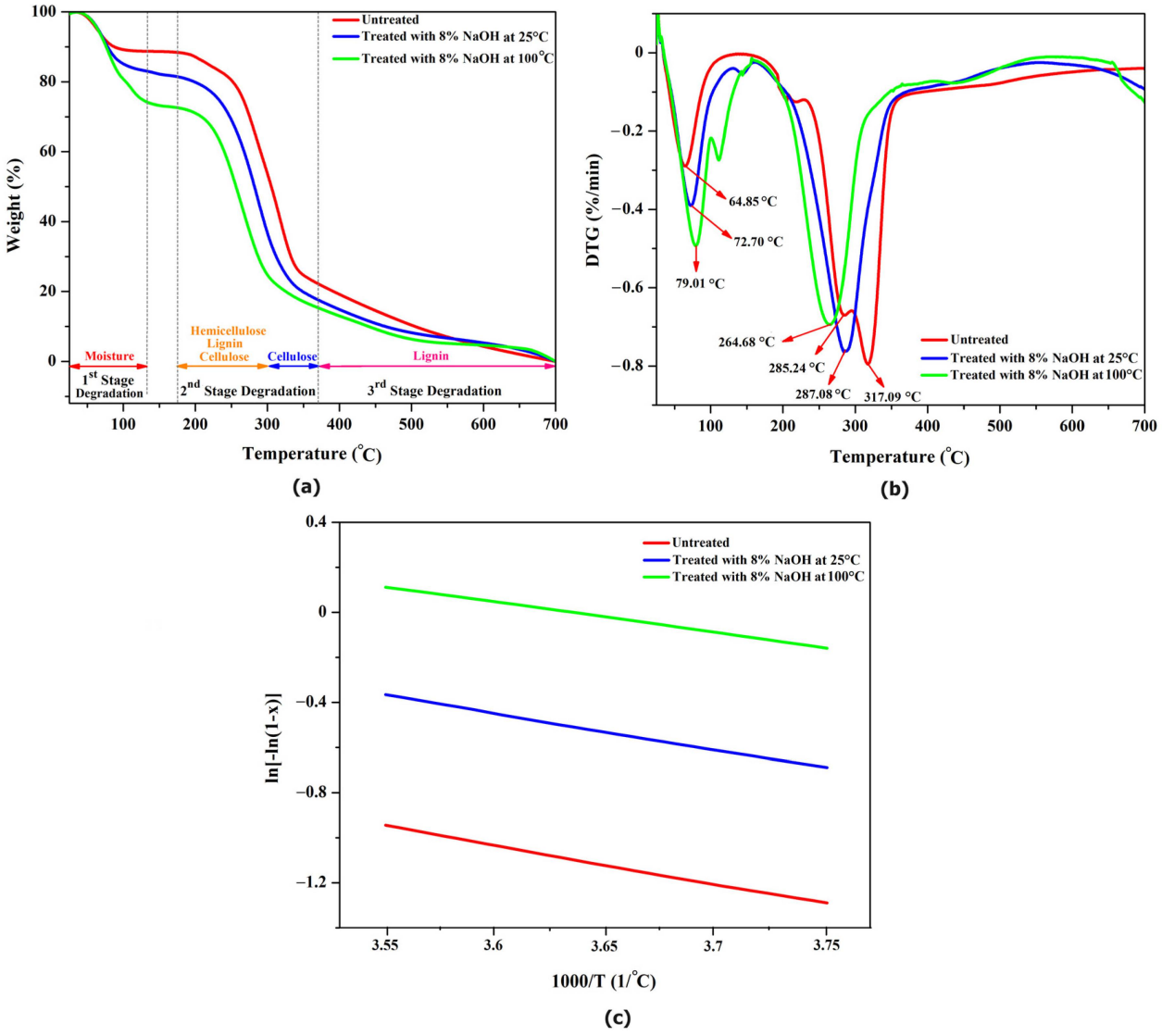

Figure 4. (**a**) TGA graph of untreated and treated CTCFs, (**b**) DTG graph of untreated and treated CTCFs, (**c**) plot for the determination of activation energy of untreated and treated CTCFs.

Table 3 offers indications on the physical and chemical properties of a number of fibers, which might in some sense be comparable with *Calamus tenuis* cane fibers, as they are not among the fibers most commonly used as materials. The table illustrates that both untreated and treated CTCFs exhibit significantly lower density in comparison to other natural fibers reported. This is likely to be attributed to the fact that the fiber is taken at a higher hierarchical level, therefore not sorting out the minimal dimension technical fiber but rather the higher-level structure (cane fiber), which includes a significant number of internal voids, as exploited in the production of cement composites. In this way, CTCFs can be utilized as reinforcement in a polymer matrix to create lightweight composite materials, though in view of the high thickness of the fibers, the possible applications are likely limited only to the construction of fibrous panels for the construction industry. All the studies

reported here indicate that alkali treatment leads to an increase in the density of the fibers, which is due to the reduction in porosity and to the enhanced amount of cellulose present in the treated fibers.

Table 3. Comparison of physical and chemical properties of untreated and treated CTCFs with other untreated and treated plant fibers.

Fibers	Alkali Treatment	Physical Properties		Chemical Properties				Ref.
		Density (kg/m^3)	Diameter (mm)	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)	
Calamus tenuis	Untreated	526 ± 16	8.31 ± 0.48	37.4 ± 1.4	31.1 ± 1	28.4 ± 0.8	4.1 ± 0.6	Here
	8% (*w*/*v*) (25 °C)	549 ± 12	8.06 ± 0.32	51.1 ± 1.6	21.6 ± 1.4	22.3 ± 1	4.8 ± 0.3	
	8% (*w*/*v*) (100 °C)	557 ± 18	7.56 ± 0.27	53.3 ± 0.8	19.3 ± 1.1	20.8 ± 0.6	5.9 ± 0.6	
Ficus religiosa	Untreated	1246	0.0256	55.6	13.9	10.1	4.9	[8]
	5% (*w*/*v*)	1272	0.0225	64.4	8.9	7.6	3.6	
Pongamia pinnata	Untreated	1345	-	62.3	14.6	12.5	5.5	[12]
	5% (*w*/*v*)	1362	-	68.4	6.3	5.1	8.3	
Phaseolus vulgaris	Untreated	934	0.352	62.2	7	9.1	-	[37]
	5% (*w*/*v*)	963	0.345	69.5	4.3	7	-	
Himalayacalamus falconeri	Untreated	1300	0.104	72.5	12.7	7.8	-	[38]
	5% (*w*/*v*)	1355	0.095	76.8	10.8	7.2	-	
Mucuna atropurpurea	Untreated	1082 ± 29	0.29 ± 0.02	58.7 ± 5.7	16.3 ± 3.2	14.2 ± 3.4	8 ± 2.5	[39]
	5% (*w*/*v*)	1136 ± 20	0.224 ± 0.014	75.2 ± 5.3	8 ± 3.1	6.7 ± 2.9	9.9 ± 2	
Thespesia populnea	Untreated	1412	0.161 ± 0.039	70.3	12.6	16.3	1.8	[41]
	5% (*w*/*v*)	1559	0.146 ± 0.090	76.4	9.6	12.8	2	
Perotis indica	Untreated	-	-	68.4	15.7	8.4	4.3	[60]
	5% (*w*/*v*)	-	-	72.4	11.3	6.6	7.6	

In Table 4, structural and thermal properties of CTCFs are compared with other untreated and alkali-treated plant fibers. It is noteworthy that the interval for thermal stability and especially the degradation process of CTCFs occurs at lower temperatures with respect to most of the other fibers considered. A possible interpretation of this trend is the lower crystallinity index of CTCFs, even after treatment, and possibly the considerable presence of coating-like and loose substances over the fibers that tend to degrade early.

Table 4. Structural and thermal properties of untreated and treated CTCFs with other untreated and treated plant fibers.

Fibers	Treatment	Structural Properties		Thermal Properties		Ref.
		CI (%)	CS (nm)	Thermal Stability (°C)	Max. Degradation Temperature (°C)	
Calamus tenuis cane	Untreated	37.48	2.73	217	317	Here
	8% (*w*/*v*) (25 °C)	44.02	2.98	211	287	
	8% (*w*/*v*) (100 °C)	41.43	2.78	204	265	
Saharan aloe vera cactus leaves	Untreated	52.6	5.6	225	350	[5]
	5% (*w*/*v*)	56.5	5.72	231	355	
Furcraea foetida	Untreated	62.05	2.44	204	357	[7]
	9% (*w*/*v*)	74.35	4.15	231	359	
Aerial roots of banyan tree	Untreated	72.47	6.28	230	358	[36]
	5% (*w*/*v*)	76.35	7.74	230	368	
Himalayacalamus falconeri culms	Untreated	58.92	3.39	250	356	[38]
	5% (*w*/*v*)	67.79	3.8	258	362	
Mucuna atropurpurea	Untreated	24.01	2.75	200	298	[39]
	5% (*w*/*v*)	49.89	1.6	200	320	
Perotis indica	Untreated	48.3	-	-	330	[60]
	5% (*w*/*v*)	55.43	-	-	349	

3.6. Scanning Electron Microscope (SEM) Analysis

The SEM micrographs displayed in Figure 5a–f illustrate the disparities between untreated CTCFs and the fibers subjected to treatment under ambient and heated conditions. All the figures indicate that the fibers extracted from *Calamus tenuis* cane are composed of a large number of aligned fibrils. However, in the treated fibers, both at ambient temperature (Figure 5c) and at 100 °C, all the fibrils are more clearly exposed, whilst in Figure 5a, the surface of most of them is concealed under non-homogeneous materials. In Figure 5a,b, the untreated fibers exhibit a smooth surface owing to the presence of various impurities such as wax, hemicellulose, lignin, and pectin. This appears typical of fibers obtained with not very selective retting methods from substantial cane-like stems [61,62]. However,

when treated with 8% NaOH at both ambient temperature and 100 °C, the fiber surface undergoes a significant change, as evidenced by the SEM micrographs shown in Figure 5c–f. This treatment effectively eliminates impurities, hemicellulose, and lignin, resulting in a roughened surface. The increased roughness of the treated CTCFs plays a crucial role in enhancing the adhesion between the fibers and the surrounding matrix [63–65]. More information would possibly be obtained through future energy dispersive spectroscopy (EDS) examinations. These considerations allow us to propose *Calamus tenuis*, after judicious alkali treatment, as a new fiber for application in thick polymer composites aimed for use in the construction sector. Other examples of the most recent works are given in [66–68]. Work with fibers up to a higher dimensional level does possibly guarantee easier retting and a less aggressive treatment, although both aspects would require a more thorough investigation in the future.

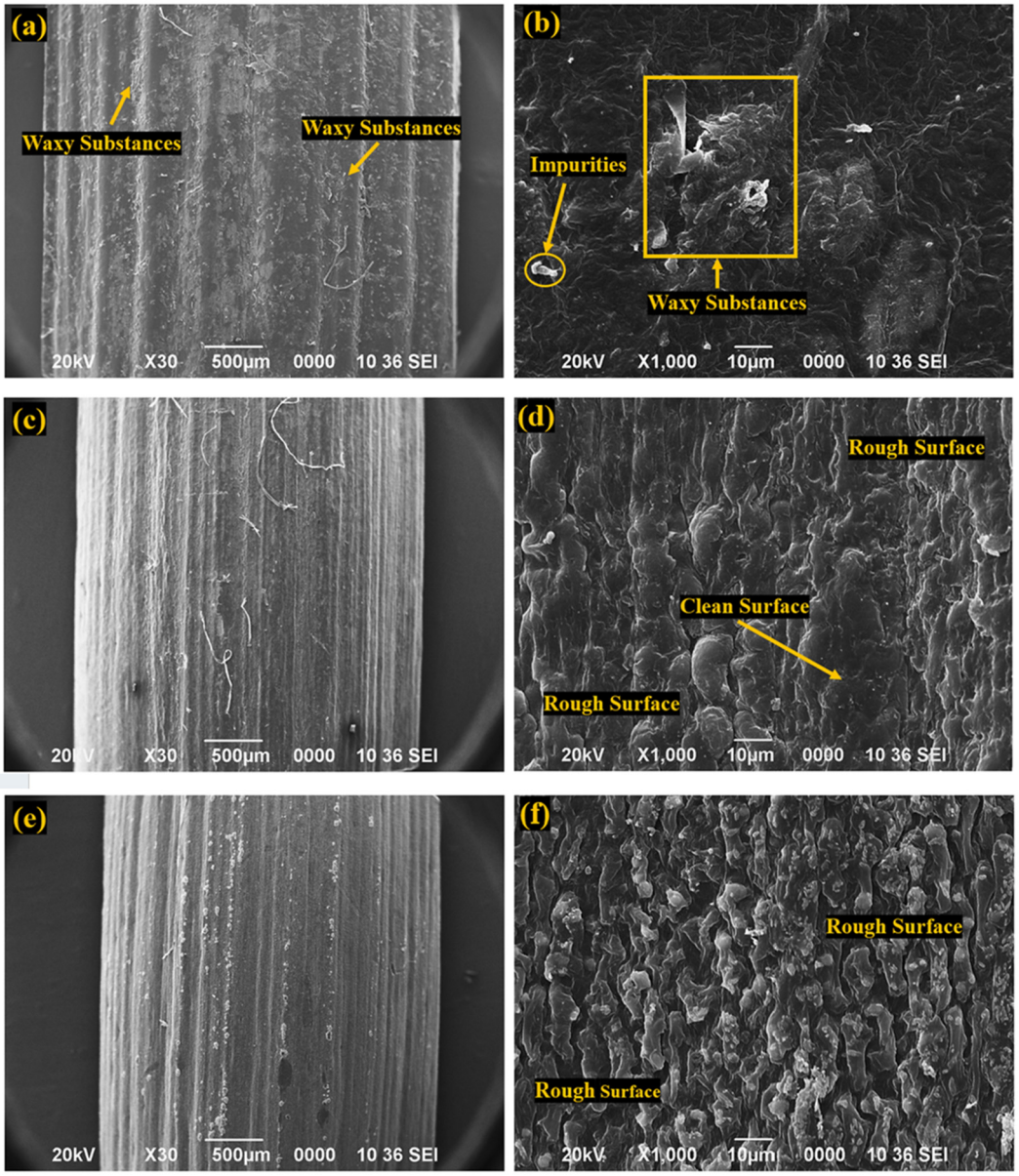

Figure 5. SEM micrographs of (**a**,**b**) untreated CTCFs, (**c**,**d**) 8% NaOH-treated CTCFs at ambient temperature, (**e**,**f**) 8% NaOH-treated CTCFs at 100 °C.

4. Conclusions

This study on the effect of alkali treatment on the physicochemical, structural, morphological, and thermal properties of *Calamus tenuis* cane fibers (CTCFs), kept within a fiber diameter of a few millimeters, therefore not proceeding to fibril separation, offered evidence for a number of conclusions. Specifically, treatment yielded a noticeable decrease in the content of hemicellulose and lignin, while the cellulose content showed an increase after the treatment, though the crystallinity and thermal stability were scarcely affected. Furthermore, the FTIR spectra of the treated CTCFs indicate that there was a partial removal of hemicellulose and lignin during the treatment process. This observation aligns with the results obtained from the chemical analysis. The XRD analysis suggests that the CTCFs treated with 8% NaOH at ambient temperature exhibited the highest values for crystallinity index and crystallite size. This implies that the treatment, particularly under these specific conditions, leads to a substantial increase in the crystallinity of the fibers, potentially enhancing their overall structural stability and strength. Moreover, the SEM micrographs depict a rough surface texture on the fibers, which can be attributed to the removal of lignin, pectin, and hemicellulose. This alteration in surface characteristics may positively impact the fibers' adhesion and compatibility in composite applications. In summary, the comprehensive findings of this study suggest that treating CTCFs with 8% NaOH at 100 °C for 4 h is not suitable for improving the properties of the fibers. However, CTCFs treated with 8% NaOH at 25 °C for 4 h had significantly improved physicochemical, structural, and surface properties. The reduction in hemicellulose and lignin content, along with the increased cellulose concentration, enhances the fibers' potential for use in composite materials, possibly in boards for construction, given their high thickness and uneven geometry.

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