



Research article

Effect of chemical treatment on physio-mechanical properties of lignocellulose natural fiber extracted from the bark of careya arborea tree

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ABSTRACT

For the first time, the current work has carried out a chemical treatment of a novel ligno-cellulose fiber that is extracted from the bark of an unexplored plant of *Careya arborea*. *Careya arborea* (CA), a flowering tree known for its green berries, thrives in the Indian subcontinent and Afghanistan. This research was focused on extracting fibers from the bark of the Cary tree for the first time to corroborate the influence of chemical treatment on its different characteristics. These CA fibers have a high proportion of cellulose, consisting of 71.17 wt percent, together with 27.86 wt percent of hemicellulose, and a reduced density of 1140 kg/m³, making them a suitable candidate for creating lightweight applications in a variety of industries. Chemical treatment has done on the cay fiber with the concentrations of NaOH 5 (wt%), 10 (wt%), and 15 (wt%) solution mixture to improve their characteristics. Estimated the difference between Chemically processed and non-processed Cary fibers and corroborated in results. We performed a number of experiments, including FTIR, XRD, SEM, EDAX, AFM, and TGA, to fully comprehend the changing properties. Chemical testing showed that cellulose changed from its non-crystalline state to cellulose, proving that the treatment was successful in changing the fibre structure. Additionally, the thermo-gravimetric examination showed higher thermal stability 248 °C–325 °C and a rise in the crystallinity index, indicating the treated fibers' improved potential for high-temperature applications. The treated Cary fibers exhibited excellent surface properties, promising improved adhesion, mechanical performance, offering lightweight and sustainable solutions for diverse applications.

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1. Introduction

Cellulose, a naturally abundant and renewable biopolymer, is a key component of various plant structures, including the bark of trees. Few lignocellulose extracted materials viz are jute [1], hemp [2], sisal ([3,4]), and banana [5], have been potentially studied in the scientific literature and, therefore, there is a growing purpose to identify new plant fibers such as *Carica papaya* L (PBF) fibers ([6; 7]), *Corypha taliera* fruit (CFT) fibers [8], *Cyperus platystylis* (CPS) fibers [9], *Furcraea foetida* (FF) fibers [10], and others, to learn about their properties and limitations. Raw cellulose fibers exhibit certain limitations in terms of strength, flexibility, and water resistance, which can restrict their widespread use in practical applications [11]. To overcome these limitations and enhance the performance of cellulose fibers, chemical treatments have emerged as a promising approach [12]. Chemical treatment methods offer an effective approach to modify the cellulose natural fiber surfaces through various chemical agents, such as alkalis [13], acids [14], and coupling agents [15], to alter their molecular structure and physical characteristics. These treatments to induce structural changes in reinforcement surface to improve their interfacial relation with continuous phase polymer materials, and to modify surface properties and enhance the physio-mechanical and enhanced resistance to environmental factors attributes of these fibers, making the fibers more suitable for use in diverse industrial sectors [16]. The sustainable nature of cellulose fibers complements the eco-friendly aspect of these composites, reducing the overall carbon footprint and making them a more responsible choice for various industries. *Careya arborea*, is a deciduous tree found in tropical and subtropical regions. The bark of this tree has noticed as an alternative, ecofriendly source of cellulose natural fibers, which holds great promise in numerous versatile industrial applications, since it's biodegradable, and low cost. Some novel plant fibers that have been identified to date, *Careya Arborea* (CA) referred to as Cary, which is a tree native to India and its subcontinent has never been used in composites for industrial use. *Careya Arborea* has not yet been recognized and used in any applications so far, our objective is to verify if they are amenable to applications belonging first to industries such as aerospace, naval, packaging, automotive, energy storage, textile, biomedical, and, secondly, it can replacement of domestic products, toys industry petrochemicals. In this research it was extracted Cary fibers and investigated the impact of chemical treatment on the thermal, microstructural, physical, and mechanical properties of cellulose natural fibers from *Careya arborea* lingo bark, to evaluate suitability for their applications. This article contributes valuable insights to identify the versatile applications of these fibers as a green and renewable resource for a wide array of lightweight composites, sustainable products. *Careya arborea* lingo



Fig. 1. Extraction of *Careya arborea* fibers by water retting method.

bark represents a significant stride towards the development of advanced lightweight composites, sustainable materials, and renewable products. By harnessing the innate properties of cellulose fibers through chemical treatment, we can foster a more sustainable and environmentally conscious approach in modern industries, contributing to a greener and more sustainable future. Furthermore, exploring eco-friendly and sustainable alternatives to synthetic fibers can contribute in terms of carbon foot print and promote the development of greener sustainable materials.

2. Materials and methods

2.1. Isolation of the CA fiber

Careya arborea (Cary) bast source selected for this current study, it belonging to Lecythidaceae family from Indian subcontinent and Indochina rejoin along with Afghanistan, and it grows from 15 to 20 m' height with the changing the colour of leaves in different seasons. it flowers with the white and yellow colour flower that can be yielded as large green berries. Fig. 1. Indicating the steps of extraction, Careya arborea fiber extracted from the Banjeeru-Jalakalingapuram village, Meliaputti mandala, Srikakulam district in Andhrapradesh, India. The bunch of collected bast strips are washed properly with clean water to remove the dust particles attached on the surface, then those cary strips are immersed in tub of clean water for 18 days for micro bacterial reaction for easy isolation of the fiber. Cary fibers are isolated with help of human intervention (hand rupture techniques) along with the plastic combing and brushing. After collection of the fibers cleanly washed with fresh cold water and repeated it for 4 to 5 cycles to remove the additional debris from the fiber. followed by these cary fibers are placed in daylight 30⁰c to 39⁰c for 76 h.

The process involves pulling from bast, retting, washing, combing and drying. Now the dried fibers are subjected to characterizations such as chemical, physical, mechanical and thermal properties. I have observed an interesting aspect from the investigations that, these fibers have highest degree of polymerization and make fiber more stable, this is ascribed the witness of the β -crystalline-cellulose. The weight percentage of this α -cellulose differentiate to various extensively used ligno-cellulose fibers is much more indicating the suitability for biodegradation. Resulting in less toxicity and it can be used in biomedical applications.

2.2. NaOH surface treatment on CA fiber

The surface treatment followed for the raw CA fiber with the 5% (wt/v), 10% (wt/v) and 15% (wt/v) of the NaOH concentrated mixture prepared for 90 min at room temperature as exhibited in Fig. 2. NaOH treatment was done for the CA fiber to improve the surface roughness for improve the adhesive bonding and to remove the additional content of nanocrystalline elements as hemicellulose, aliphatic and aromatic lignin, crystal wax, and pectin and it will be helpful to convert the nanocrystalline form of the cellulose to convert as the cellulose.

2.3. Chemical and physical observations

To measure the minimum and maximum changes in main constituents, elemental change, morphological change of raw cary fiber and NaOH treated cary fiber has followed standard techniques as Fibre Chemical Composition Testing (G2100052-1) according to SITRA Coimbatore and UTM machine for single fiber pullouts.



Fig. 2. Chemical treatments of cary fibers with 5%, 10%, and 15% of NaOH.

2.4. Thermogravimetry test (TGA)

To measure the thermal characteristics of NaOH treated cary fiber was used the sample size of 10 mg loaded in aluminum container with the nitrogen-controlled environment in furnace with the flow rate 20 ml/min, and the temperature raised from 30 °C to 800 °C in the chamber with step of 10 °C/Min. The analysis carried on the SII 6300 EXSTAR equipment model.

2.5. Morphological analysis (SEM)

Scanning electron images were helpful to inculcate the NaOH chemical treated fiber surface roughness, porosity and surface modifications, these scanning electron images were captured from Oxford Instruments ZEISS EVO 18 special addition instrument.

2.6. Fourier transform infrared spectroscopy (FTIR)

NaOH treated CA fiber samples FTIR analysis reveals the insights of various bonding presented, different stretching with functional groups available in the fibers samples, the NaOH solution processed CA fiber blended with combination of potassium bromide and conducted the spectroscopy analysis. Bruker alpha Eco-ATRIR with ZnSe crystal instrument were used for these spectra recording.

2.7. X-ray diffraction (XRD)

Crystallographic analysis on NaOH treated CA fibers with help of Rigaku, Smart Lab X-Ray produced with 9 kW for Photon Max rotating anode with detector HyPix-3000 Maximum energy intensity with 2D HPAD detector with minimum angular increment size of 0.0001°. The graphs were generated using Origin Pro software, which also facilitated the identification of crystallographic planes within CA fibers. This enabled the determination of the crystalline and amorphous, and subsequently, the identification of the crystalline property i. e index (C₁ %) of CA fibers using Eq. (1):

$$\%C_1 = (I_{002} - I_{am} / I_{002}) \times 100 \tag{1}$$

where I₀₀₂ represent the peak intensity of the (002) lattice diffraction at a 2θ angle ranging from 22° to 23°, while I_{am} denotes the lowest intensity observed in an amorphous region at a 2θ angle between 18° and 19° [17,18].

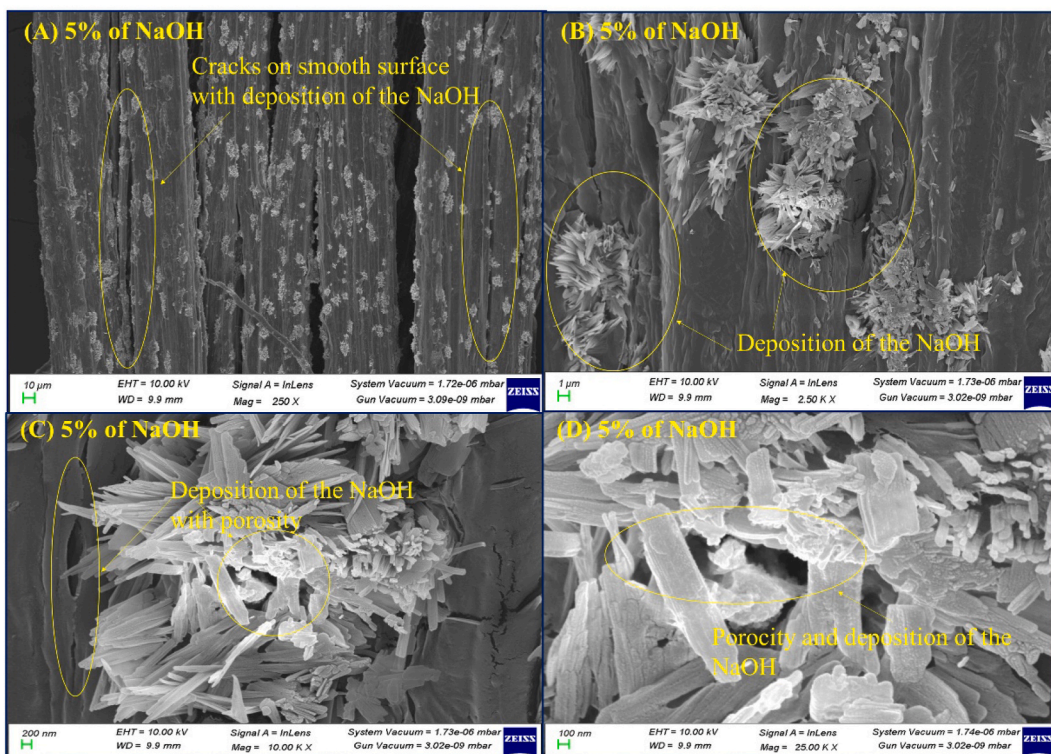


Fig. 3. SEM images of 5% NaOH-treated CA fibers.

3. Results and discussion

3.1. Mechanical observation

The unique versatility of this fiber lies in its remarkable hemicellulose content, surpassing that of other extensively utilized fibers within the global community. This hemicellulose-rich composition imparts exceptional strength and toughness to the fiber, rendering it an ideal candidate for applications demanding elevated levels of mechanical resilience. This advantageous property stems from the fiber's robust amorphous nature, which establishes strong bonds with both lignin and cellulose components. Particularly noteworthy is the remarkable lignin content present in Cary fibers, surpassing the levels observed in other known fibers as documented in scientific literature. This revelation underscores the fiber's inherent rigidity and heightened resistance to decay. Significantly, a higher lignin content aligns with enhanced suitability for biomedical applications, attributed to its diminished toxicity profile. Within this intricate interplay, the pivotal roles of lignin and hemicellulose quantities become evident. The amorphous hemicellulose intricately interacts with the rigid lignin structure, thereby synergistically amplifying the overall strength of the fiber. This symbiotic relationship is further substantiated by mechanical characterizations, including tensile strength assessments, revealing that the Cary fiber outperforms a diverse array of available counterparts. The accompanying figure provides a microscale visualization of the Cary fibers, offering a tangible representation of the structural intricacies that contribute to its exceptional mechanical attributes.

3.2. Morphological observation (SEM)

It is an apt procedure to identify and understand the fiber surface, and morphology of the NaOH-treated CA fibers. Fig. 3, Fig. 4, and Fig. 5 indicated the observations of SEM images of 5%, 10%, and 15% NaOH-treated CA fibers. From the micrographs, it was identified that after chemical modifications, remarkable enhancement in the CA fibers morphologies. The extracted fibers displayed in Fig. 3 is implicative to the surface with a slit at equidistance and forms a box like structure Fig. 3A and 3B. This is an important characteristic realizable that the applications of these fibers in low weight and high strength applications is substantiated. At the meantime, the fiber thickness is uniform and indicating the excellent stress distribution characteristics. The CA fibers revealed that they applicable due to low density with respect to recent studied fibers in the literature, based on this realized that will be help full to develop lightweight materials. The surface roughness in the 5% NaOH-treated CA fibers surface was enhanced after elimination of nanocrystalline elements

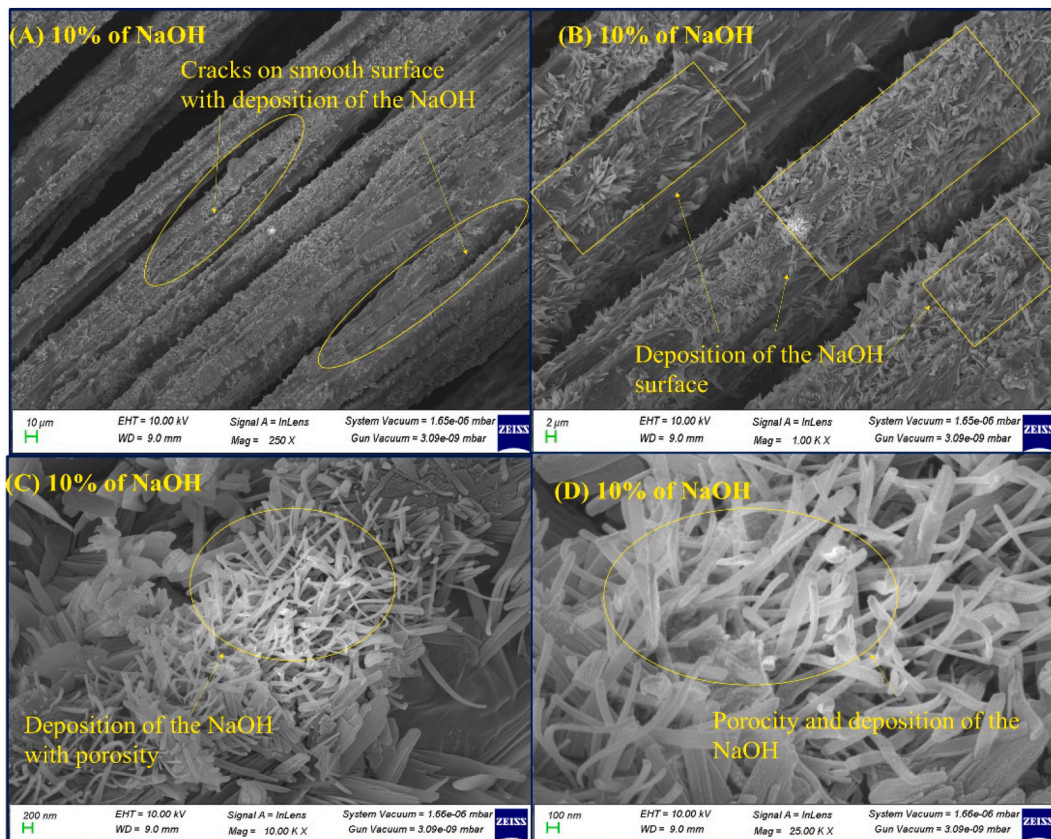


Fig. 4. SEM images of 10% NaOH-treated CA fibers.

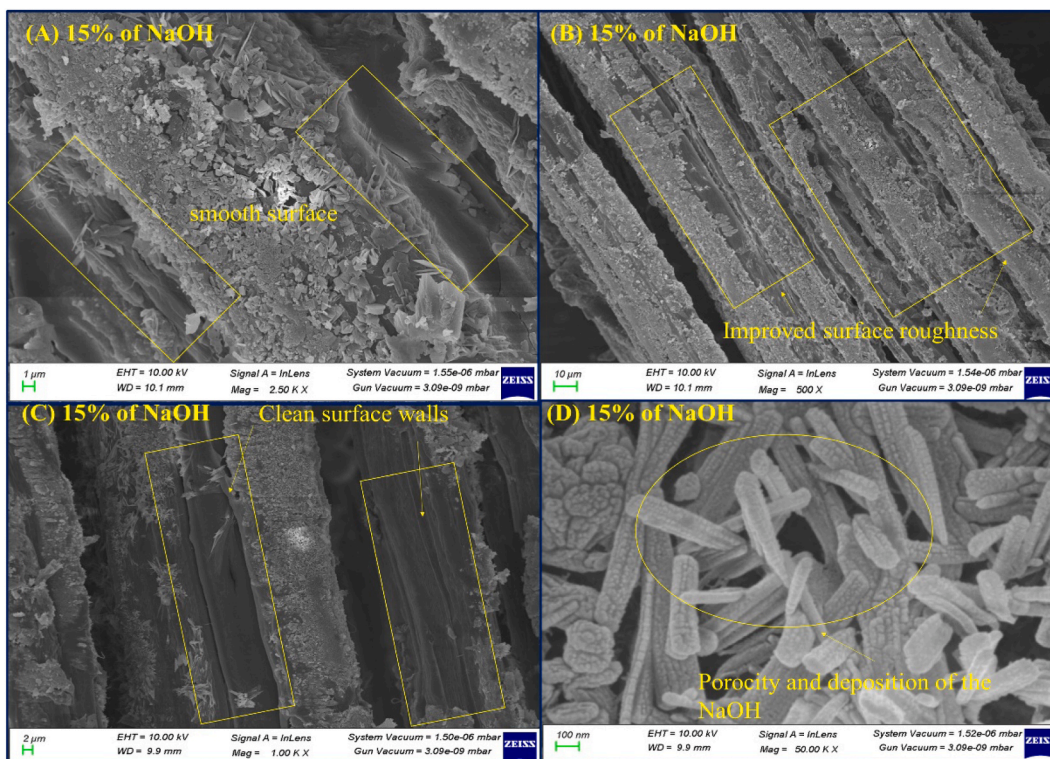


Fig. 5. SEM images of 15% NaOH-treated CA fibers.

as hemicellulose, aliphatic and aromatic lignin, crystal wax, and pectin, CA fibers surface exhibited in Fig. 3C [19]. As per the studies, fiber irregular surface patterns are different for processed fibers due to elimination hemicellulose along with impurities [20]. Thus, Alkaline treatment shows the impact with considerable variations to the surface textures of CA fibers, which can improve the fiber's wettability. Additionally, removing surface impurities from fibers improved fiber interlocking with matrix and the coupling response, which is amplifying for fiber-matrix bonding relation as shown in Fig. 3D [21]. During the biological studies to see the amenability of the fiber for biomedical applications, we have identified that these materials can be biodegradable too and found to be amenable for bone tissue engineering applications. The alterations in surface characteristics observed in CA fibers treated with 5% NaOH offer valuable insights into the intricate interplay between chemical treatment and bio-composite behavior. The changes in porosity, deposition phenomena, and the emergence of cracks on smooth surfaces underscore the multifaceted nature of these modifications. While these alterations may present challenges, they also offer avenues for tailoring bio-composite properties for specific applications. A comprehensive understanding of these surface modifications is pivotal for advancing the field of bio-composites and unlocking their full potential in various industries.

In similar context, were identified five more such fibers and investigated their mechanical, structural and thermal characteristics and found intriguing nature which have versatile potential in various industries and provision to tailor the properties at different scales. However, after the CA fibers being treated with 10% NaOH, a plane surface pattern was identified and gaps on the CA fiber outer layer were recognized in shown Fig. 4A and 4B. This was associated with the removal of fatty components from the fiber surface [16]. In case of higher porosity level in the fiber may leads towards compromising the mechanical characteristics it replicated in Fig. 4D [22]. CA fibers treated with 10% NaOH displayed potentially increased surface roughness compared to fibers treated with 5% NaOH. The higher concentration of NaOH promotes a more vigorous removal of surface materials, resulting in a rougher texture. The chemical treatment with 10% NaOH increased surface porosity compared to the 5% NaOH treatment. The higher NaOH concentration caused more significant swelling and partial dissolution of surface components, leading to the formation of gaps and voids. The characteristics of 10% NaOH-treated CA fibers compared to those treated with 5% NaOH exhibit several alterations in terms of porosity, deposition, and cracks on smoothness because of NaOH solution effect showed in Fig. 4C. The CA fibers treated with 10% NaOH might display increased porosity compared to those 5% NaOH-treated CA fibers. The 10% of NaOH solution treatment extends towards more extensive decay of amorphous regions within the fibers, potentially resulting in the creation of additional pores and voids on the surface. This increased porosity can influence the fiber's overall surface area and may impact its interaction with other materials or substances. With 10% NaOH treatment, promoted a greater likelihood of material deposition or accumulation on the fiber surfaces showed in Fig. 4C. The 10% of NaOH in chemical treatment increased the reduction of nanocrystalline contents (like hemicelluloses or lignin) from the CA fibers, leaving behind a relatively cleaner surface. This cleaner surface could facilitate the bonding with other materials [23]. The CA fibers treated with 10% NaOH exhibited more noticeable cracks or fractures on their

smooth surfaces compared to those 5% NaOH-treated CA fibers. The stronger alkaline conditions of the 10% NaOH treatment caused more distinct modifications in the fiber structure, potentially leading to the formation of cracks as a result of structural rearrangements or stress within the CA fibers. These cracks could be visible on the smooth surfaces of the CA fibers and affected their mechanical properties and surface integrity.

The treatment of CA fibers with 15% of sodium hydroxide (NaOH), has led to notable alterations in their surface characteristics, particularly in terms of porosity, deposition, and the occurrence of cracks on smooth surfaces showed in Fig. 5. These changes can be attributed to the chemical interactions and reactions induced by chemical treatment. 15% NaOH-treated CA fibers causes more intense breakdown of non-cellulosic materials, these components re-deposited on the CA fiber surface as the NaOH solution is neutralized showed in Fig. 5A and 5B. This deposition could result in visible changes to the fiber's surface texture, potentially affecting its overall appearance and properties. The increase in 15% NaOH-treated CA fibers was revealed the significant enhancement in the porosity of CA fibers' surfaces showed in Fig. 5D. The higher alkaline concentration is likely causing a more vigorous reaction with the cellulose matrix. This reaction leads to the disruption of hydrogen bonds and other intermolecular forces within the CA fibers, thereby loosening the fiber structure and exposing internal pores as shown Fig. 5C. Distinct differences in deposition patterns have been observed between CA fibers treated with 15% NaOH and those treated with 5%, and 10% NaOH. The 15% NaOH-treated CA fibers appears to promote more substantial and uniform deposition of materials onto the fiber surface. This was attributed to the increased availability of reactive functional groups and enhanced surface reactivity resulting from the more aggressive chemical treatment. Therefore, the uniform deposition observed on the 15% NaOH-treated CA fibers suggests a more homogenous surface, but 5% NaOH-treated CA fibers have more implications positives for applications such as composite materials. One intriguing observation is the cracks appearance on the surfaces of 15% NaOH-treated CA fibers. These cracks indicate a potential structural modification caused by the higher alkaline concentration. It is plausible that the aggressive nature of the 15% NaOH treatment could induce internal stress within the CA fibers, leading to microcracking on the smooth surface. This effect resulted from both the disruption of intermolecular forces and the removal of certain components, causing a weakening of the fiber's structural integrity.

3.3. FTIR analysis

It identify the functional group in CA fibers. Fig. 6 illustrate the FTIR spectra of 5% (curve in pink), 10% (curve in red), and 15% (curve in blue) NaOH-treated CA fibers. The chemical groups available in the Carry fibers was studied with FTIR spectrum in Fig. 6 revealing the wavenumber of FTIR spectra ($4000\text{--}500\text{ cm}^{-1}$) of NaOH-treated CA fibers. The spectra in Fig. 6 corroborate for 5%, 10%, and 15% NaOH-treated CA fibers. Identified peaks 3323 cm^{-1} , 2919 cm^{-1} , 1646 cm^{-1} , 1419 cm^{-1} , 1020 cm^{-1} , and 551 cm^{-1} attribute the (O–H), (C–H), (C=O), (CH_2), (C–OH), and (C–OH) bonds, respectively. Higher intensity band was point out at 3323 cm^{-1} which attributes the (OH) group present in the β -cellulose [24]. Medium peak noticed at 2919 cm^{-1} & 2840 cm^{-1} proved the existence of C-H structural presence with cellulose. The peak position at 1646 cm^{-1} showcases the hemicellulose with C = O group, [25]. The peaks at 1419 cm^{-1} are correspond with CH_2 identical bending in main constituent of fiber [26]. The corresponding peaks at 1020, (C–OH) and (C–O–C) band of polysaccharides (C. M [27]). However, the 10%, and 15% NaOH-treated CA fibers had the increased peaks at 3323 , 2919 cm^{-1} , and 2840 cm^{-1} . The peak in 1646 cm^{-1} was modified and conforms the elimination of nanocrystalline hemicellulose. The peaks at 1419 cm^{-1} , and 1120 cm^{-1} are organized the changes perfectly. It is witnessed that the chemical treatment eliminates the all unnecessary reduces and debris from the CA fibers [28,29].

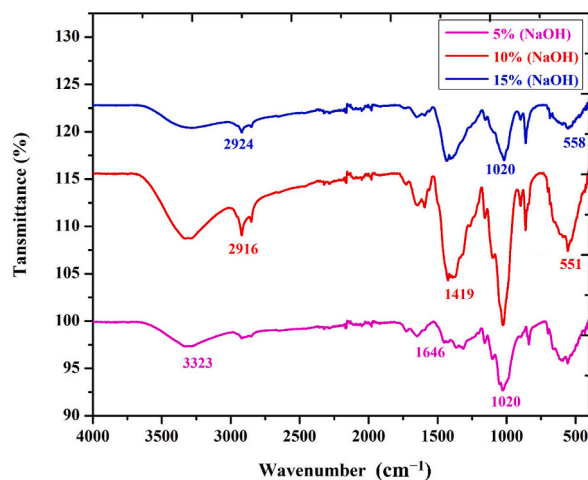


Fig. 6. FT-IR spectrum analysis of *Careya arborea* fibers.

3.4. EDAX and chemical observation

Fig. 7 illustrates the EDAX analysis of treated CA fibers, revealing the presence of various chemical elements in its composition, including O, Na, C, Al, Si, P, Cl, K, Ca, and S. The carbon weight percentage of the untreated Cary fibers is 15.66%, which is lower than the corresponding percentages in the compared all treated CA fibers (5%, 10%, and 15% NaOH) as exhibited in Fig. 7. Similarly, the identified oxygen percentage in the untreated CA fibers is 26.37%, also lower compared to all treated CA fibers (5%, 10%, and 15% NaOH). The chemical treatment has a higher impact in calcium content have strong reason for the elongation of the natural fibers [30].

Table 1 presents summarized information about elemental analysis of treated CA fibers in the form of weight and atomic percentage. The EDX analysis employed in this study is a highly popular technique for conducting both quantitative and qualitative elemental analysis. The composition of CA fibers was found, primarily it consisted with abundant cellulose, minor lignin, moderate hemicelluloses, and small percentage waxes. The detailed chemical composition of the CA fibers has been previously reported by Rao, H. Jeevan et al. [31,]. The primary chemical elements in the Cary were C and O. Other elements, such as Na, Al, S and Ca, can also be presented on the fiber surface, but in trace amounts as shown Table 1. Table 1 indicates that a moderate percentage of Na was existed in the exterior portion of the CA fibers after chemical treatment with NaOH as showed in Fig. 7. Which impact the interlocking and bonding formation on the surface of the CA fibers while the NaOH treatment as exhibited in Eq. (2).

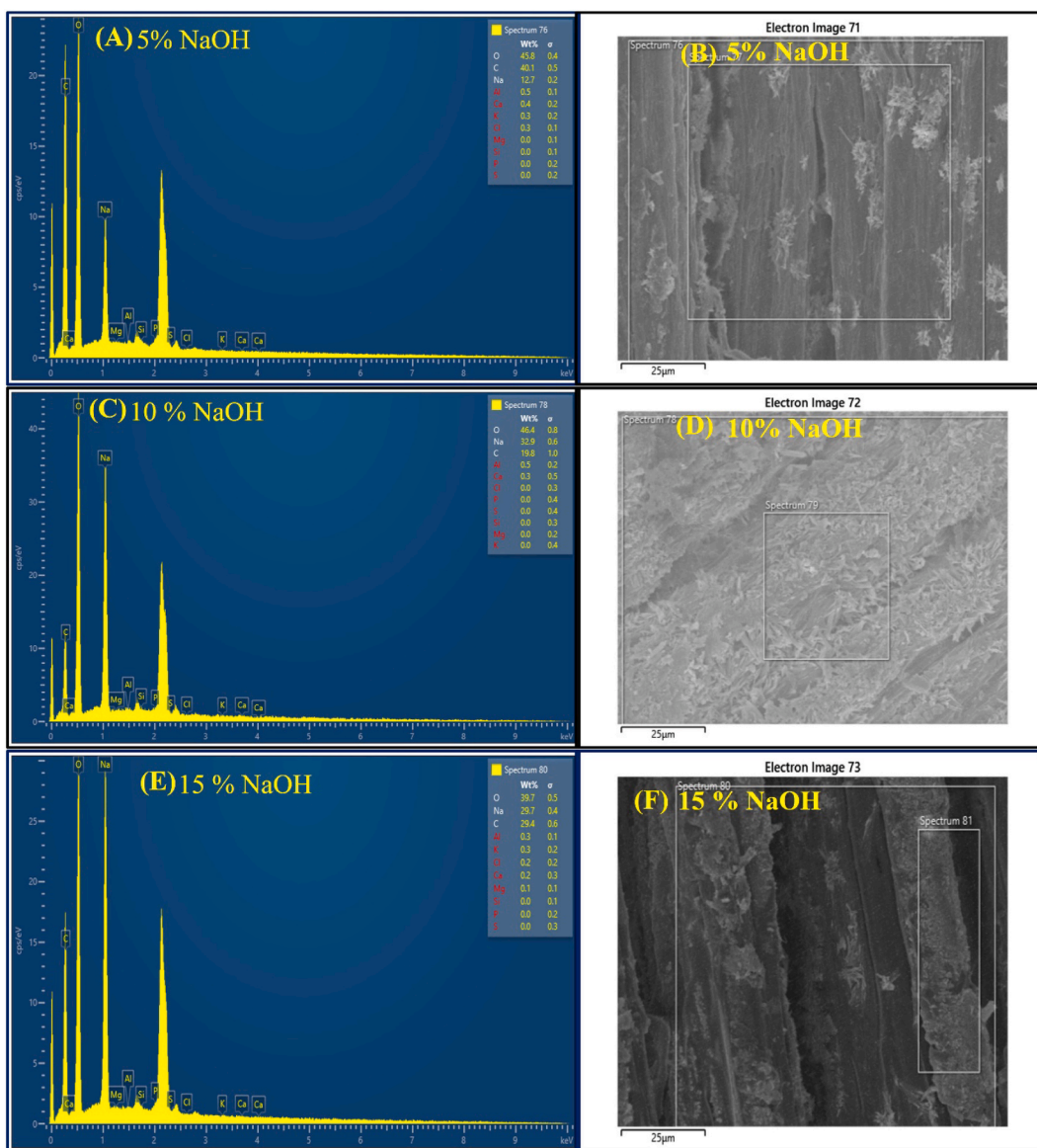


Fig. 7. Weight and atomic percentage of treated CA fibers using EDX.

Table 1
Quantitative elemental of treated CA fibers.

Elements	Treated CA fibers to 5% NaOH		Treated CA fibers to 10% NaOH		Treated CA fibers to 15% NaOH	
	%Weight	%Atomic	%Weight	%Atomic	%Weight	%Atomic
C	40.06	49.10	19.85	27.49	29.45	39.15
O	45.75	42.11	46.39	48.24	39.71	39.64
Na	12.74	8.16	32.94	23.84	29.74	20.66
Mg	0.02	0.01	0.00	0.00	0.13	0.09
Al	0.45	0.25	0.49	0.31	0.33	0.19
Si	0.00	0.00	0.00	0.00	0.00	0.00
P	0.00	0.00	0.00	0.00	0.00	0.00
S	0.00	0.00	0.00	0.00	0.00	0.00
Cl	0.28	0.12	0.01	0.00	0.24	0.11
K	0.33	0.12	0.00	0.00	0.25	0.10
Ca	0.37	0.13	0.32	0.13	0.16	0.06



Na was incorporated into the CA fibers after they were treated with a NaOH solution. The findings show a clear presence of salt in the NaOH-CA fiber structure, which may be explained by the CA fibers' cellulose contents becoming alkaline. Table 1 further indicates a decrement in the carbon content percentage in CA fibers treated 10% NaOH, and 15% NaOH compared to 5% NaOH (see Fig. 7A and 7B), as well as no significant change in oxygen percentage with chemical modifications on CA fibers treated 5% NaOH, and 10% NaOH (see Fig. 7C and 7D) compared to 15% NaOH, due to the possibility that some of the pectin and hemicelluloses on the surface of the treated CA fibers were eliminated by the surface treatments with 15% NaOH (see Fig. 7E and 7F) [32]. This EDAX analysis and elemental composition of the treated CA fibers provide valuable insights into its chemical composition to understand the variations, and potential implications for its properties and applications. Table 2 indicates the data from the quantitative chemical composition analysis of treated CA fibers after chemical modification, in terms of cellulose, lignin, hemicellulose and wax and their weight percentage.

3.5. XRD

There are two forms of cellulose that are found in nature, i.e. cellulose I α and I β . The I β crystalline plane, which is primarily present in higher plants extracts, it was the exclusive focus of this study on CA fibers; I α is rare and generally displayed in bacterial and algal celluloses [33,34]. Hydrogen-bonded sheets are formed by parallel chains that make up cellulose I β . Because of van der Waals interactions, these sheets stack with an alternating shear parallel to the chain's axis [35]. XRD spectra of raw fiber and treated CA fibers are displayed in Fig. 8. As shown in Fig. 8, the Major peaks were seen in the X-ray diffraction pattern of the CA fibers at angles of roughly $14.54^\circ \geq 2\theta \leq 17.32^\circ$, $16.12^\circ \geq 2\theta \leq 18.2^\circ$, $22.2^\circ \geq 2\theta \leq 23.2^\circ$ and $33^\circ \geq 2\theta \leq 35.02^\circ$, which corresponded to the lattice planes of peak 1 (1–10), peak 2 (110), peak 3 (200), and peak 4 (004) for cellulose I β crystalline ([36,37]; C. M [27]). The peak 1, peak 2 and, peak 3 are the characteristic identification of cellulose I β [38,39] Due to their wide full width at half-maximum (FWHM), peaks 1 and 2 seem as a single broad peak and are most likely overlapping [40,41]. It was evident from Fig. 8 that the peaks in the regions $2\theta = 14.54^\circ$ - 17.32° (1–10) 16.12° - 18.2° (110), 22.2° - 23.2° (200), and 33° - 35.02° (004) were similar. The structural transition of crystalline cellulose to amorphous cellulose was also demonstrated by XRD data following chemical treatment with 10% and 15% NaOH, respectively, as opposed to 5% NaOH.

The cellulose C₁ calculated from the XRD patterns of untreated and treated CA fibers (5%, 10%, and 15% NaOH) is shown in Table 3. The percentages of C₁ in the CA fibers that were left untreated and those that were treated with NaOH were roughly 67.96%, 83.3%, 44.87%, and 27.78%, in that order. Fascinatingly, following the chemical treatments with 5% NaOH, the cellulose crystallinity

Table 2
Comparative study of chemical composition of treated CA fibers.

Fibre Name	Cellulose [% wt]	Hemicellulose [% wt]	Lignin [% wt]	Ash [% wt]
Careya arborea (Treated fiber)	79.3	18.7	10.3	0.34
Careya arborea (Raw fiber)	71.17	27.86	14.95	0.78
Okra	60–70	15–20	5–10	–
Prosopis juliflora	61.65	16.14	17.11	5.2
Bagasse (sugarcane)	32–44	27–32	25.3–24.3	–
Sisal	78	10	8	–
Flax	81–85	16.7–20.6	3	–
Furcraea foetida	68.35	11.46	12.32	6.53
Coir	41–45	0.3	45	–
Ramie	73–77	15	1	–
Jute	72	13	13	–
Grewiatilifolia	62.8	21.2	14.9	–

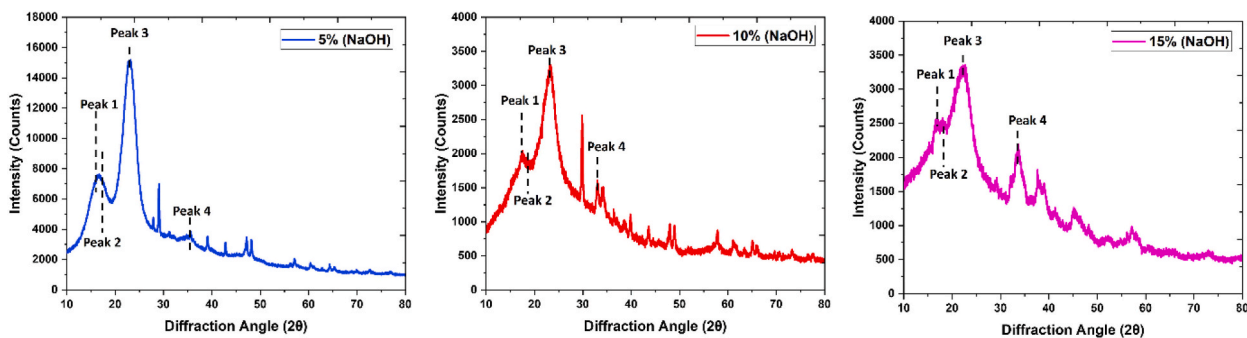


Fig. 8. X-ray diffraction analysis of NaOH-treated CA fibers.

Table 3
Comparative of Crystallinity index, peak intensity, and 2θ values in CA fibers.

CA Fiber	C _i (%)	I ₁₋₁₀		I ₁₁₀		I ₀₀₂		I ₀₀₄	
		Peak	2θ	Peak	2θ	Peak	2θ	Peak	2θ
5% NaOH	83.3	7390	16.16°	7335	17.02°	15,008	22.96°	3532	35.02°
10% NaOH	44.87	1916	17.32°	1852	18.12°	3205	23.2°	1554	33°
15% NaOH	27.78	2420	16.92°	2475	18.2°	3246	22.46°	1981	33.68°

index rose. The CA fibers hemicelluloses and lignin were partially eliminated by the chemical treatments with 5% NaOH, which may have enabled the fibers to take on a more crystalline structure [42]. It was determined that the destruction of amorphous components in CA fibers and the rearranging of cellulose molecules could lead to an increase in cellulose crystallinity following surface treatment with 5% NaOH [43]. However, treated CA fibers with 10%, and 15% of NaOH exhibited decrease in crystallinity, and the main reduction of the crystallinity peak (002), due to the amorphization of the CA fibers as well as, increase of amorphous cellulose upon chemical treatment with 10% and, 15% NaOH [44]. The enhanced characteristics of the CA fibers treated with 5% NaOH may be attributed to the increased CI in them, which made them perfect for developing hybrid and biocomposite applications.

3.6. Thermogravimetric analysis

The TGA and DTG curves of CA fibers treated with 5%, 10%, and 15% NaOH are displayed in Fig. 9, and Table 3 displays the thermal degradation range with weight loss percentage. Three stages of mass degradation for CA fibers treated with NaOH are shown in Fig. 9A. It was noted that the early, sluggish mass deterioration happened in $35 \geq T_{Initial} \leq 213 \text{ }^\circ\text{C}$, $35 \geq T_{Initial} \leq 214 \text{ }^\circ\text{C}$, and $35 \geq T_{Initial} \leq 210 \text{ }^\circ\text{C}$, for treated CA fibers with 5%, 10%, and 15% NaOH respectively. This happened because of the CA fiber’s structurally integrated water molecules breaking down, which let the moisture that had been held to escape [45,46]. The first breakdown of water molecules in treated CA fibers with 5%, 10%, and 15% NaOH happened at temperatures of $35 \geq T_{Initial} \leq 213 \text{ }^\circ\text{C}$ with 1.68% weight loss, $35 \geq T_{Initial} \leq 214 \text{ }^\circ\text{C}$ with 1.3% weight loss, and $35 \geq T_{Initial} \leq 210 \text{ }^\circ\text{C}$ with 1.27% weight loss, respectively as exhibited in Table 4.

After the first phase of the TGA, zero weight loss was seen until 200.4 °C for CA fibers treated with 5% NaOH, 204 °C for CA fibers

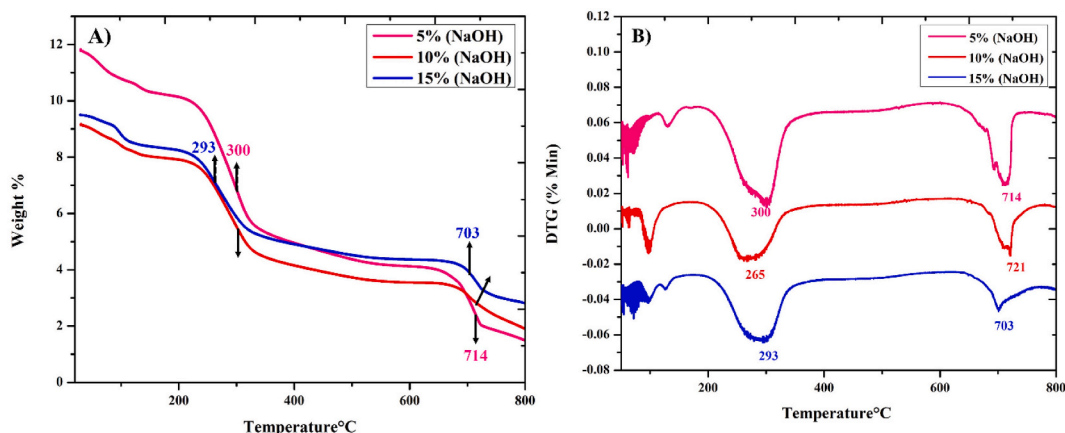


Fig. 9. A) Thermogravimetric curve, B) differential thermogravimetric curves for NaOH-treated CA fibers.

Table 4
Thermal properties of treated CA fibers.

CA Fiber	1 st Phase of degradation		2 nd Phase of degradation		3 rd Phase of degradation	
	Temperature range	Weight loss	Temperature range	Weight loss	Temperature range	Weight loss
5% NaOH	35–213 °C	1.68%	251–325 °C	4.00%	685–721 °C	1.44%
10% NaOH	35–214 °C	1.3%	241–325 °C	2.79%	688–725 °C	0.60%
15% NaOH	35–210 °C	1.27%	237–307 °C	1.16%	699–724 °C	0.69%

treated with 10% NaOH, and 195.6 °C for CA fibers treated with 15% NaOH. This temperature range may be interpreted as a measure of the fibers' thermal stability [47,48]. It can be because the CA fibers' amount of amorphous hemicellulose has decreased as a result of chemical treatment. Tonset degradation, the second stage of degradation, is distinguished by a sharp drop in mass between 248 °C and 325 °C, 241 °C–325 °C, 237 °C–307 °C, for treated CA fibers with 5%, 10%, and 15% NaOH respectively. It is linked to the disintegration of extremely stable cellulose and thermally unstable hemicellulose. A prominent peak was observed at 300 °C, 265 °C, and 293 °C, respectively, in the DTG curve (Fig. 9B) for treated CA fibers with 5%, 10%, and 15% NaOH. This peak indicated the polymerization of cellulose (partially degrading cellulose I β and completely disintegrating cellulose) and hemicellulose [49,50]. In the second stage (T_{onset}) stage, the treated CA fibers with 5% NaOH showed a maximum weight loss of 4.00% compared to treated CA fibers with 10%, and 15% NaOH, which was 3.5 times, and 1.4 times higher than that treated CA fibers with 15% (1.16%), and 10% NaOH (2.79%) respectively, owing to the fact that CA fibers treated with 5% NaOH had more amorphous hemicellulose, which was less thermally stable cellulose than CA fibers chemically treated with 10% and 15% NaOH. pronounced peak at 300 °C, 265, and 293 °C respectively. The temperature at which the CA fibers begin to degrade is indicated by the greatest peak on the DTG curve. The DTG curve revealed that the 10% and 15% NaOH-treated CA fibers had thermal degradations of 265 °C and 293 °C, respectively, and that the 5% NaOH-treated CA fiber had thermal degradations of 300 °C, or 11.7% and 2.3% higher, respectively. The breakdown of lignin, wax, and other components of treated CA fibers resulted in the final stage of degradation (T_{final}), which happened in the temperature range of 685 °C > T_{final} < 725 °C. Due to its complex structure, lignin breaks down slowly and takes a long time to break down [51,52].

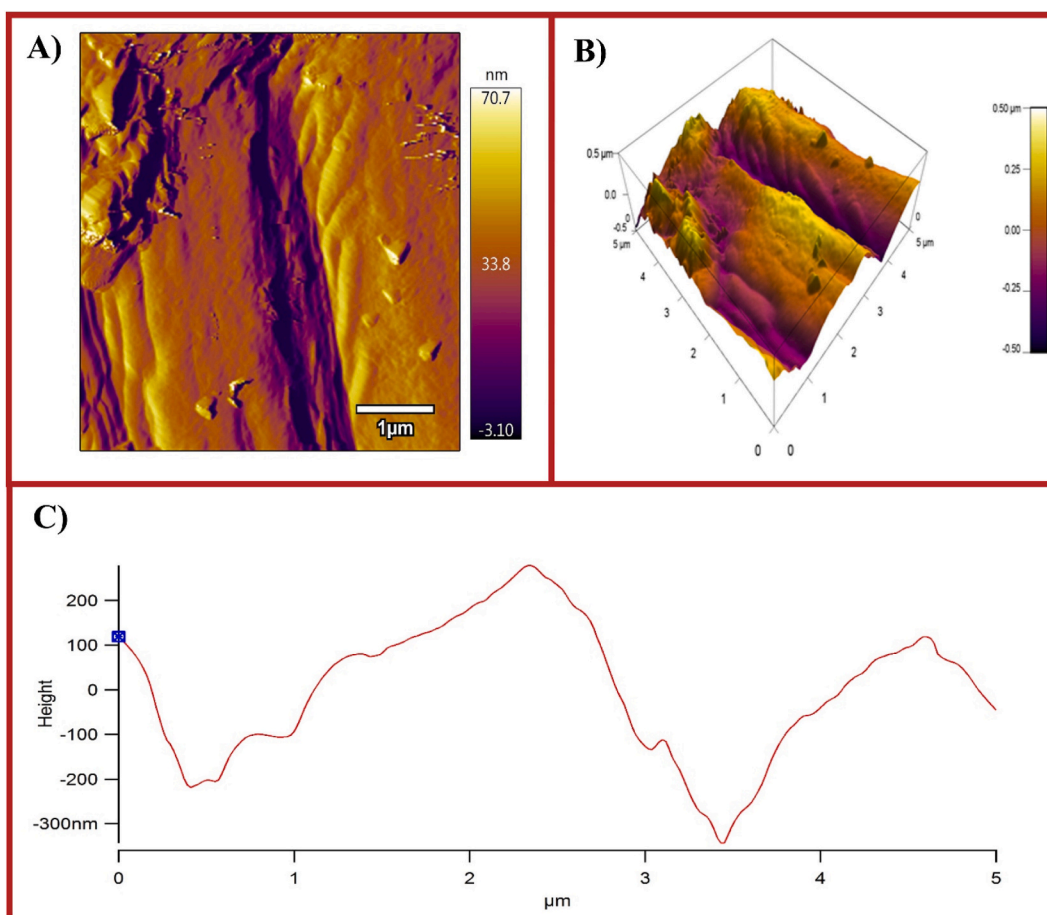


Fig. 10. Topography of 5% NaOH-treated CA fiber A) height profile, B) 3D image, and C) 2D image.

As of right now, the treated CA fibers' weight loss percentages with 5%, 10%, and 15% NaOH were 1.44%, 0.60%, and 0.69%, respectively. By dissolving impurities and breaking down hydroxyl groups, chemical treatment improved thermal stability and decreased the weight of hollow fibers [53,54]. Based on the investigation above, it was shown that CA fibers treated with 5% NaOH had superior thermal stability than CA fibers treated with 10% and 15% NaOH. These fibers may also be used in hybrid composites and as a bio-reinforcement in polymer matrix.

3.7. AFM analysis

Visual presentations of the 3D topographic pictures and surface roughness evaluations of CA fibers treated with 5% NaOH are made in Fig. 10. The 3D topographical views illustrated in Figs. 10A and 10B vividly showcase a significant enhancement in surface roughness following the application of 5% NaOH treatment. This chemical treatment effectively disintegrated surface contaminants and amorphous components inherent in CA fibers [10]. Notably, the treatment induced transformative alterations in the surface textures of the CA fibers treated with 5% NaOH, thereby potentially impacting their wettability and interfacial bonding characteristics with polymer matrices. Intriguingly, the distinctive peaks and valleys visible in Fig. 10C serve to accentuate the alterations in surface roughness, which could be advantageous for harnessing the potential of CA fibers in composite materials [55]. Furthermore, the chemical treatment exerted a positive influence on augmenting the surface roughness of the CA fibers. The changes in surface topography experienced by the 5% NaOH-treated CA fibers substantiate the notion that these treated fibers exhibit a favorable profile for utilization in the fabrication of lightweight, fiber-reinforced composites. In Table 5 the AFM Properties are summarized.

4. Conclusions

One of the notable advantages lies in the geographical abundance of these fibers within the Indian subcontinent, presenting a substantial potential for fostering economic growth and contributing to a sustainable global equilibrium. Therefore, the exploration of innovative fibers and the adept utilization of bio-waste offer a pathway to circumvent reliance on petrochemicals. Additionally, a primary objective is to create biodegradable materials that mitigate pollution and bolster environmentally friendly practices for a safer ecosystem. The morphological, chemical, crystallographic, and thermal characteristics of treated CA fibers were all examined in the experimental study. The chemical treatment (with NaOH) successfully removed the hydroxyl (OH) groups from the CA fibers' surface. This chemical process markedly eliminated non-cellulosic components within the fibers. Quantitative chemical analysis revealed a reduction in carbon content percentage in CA fibers treated with 10% NaOH and 15% NaOH, as compared to those treated with 5% NaOH. Notably, there was no substantial alteration in oxygen content following chemical treatments for CA fibers treated with 5% NaOH and 10% NaOH, in contrast to 15% NaOH. This difference may be due to the possible removal of hemicelluloses and pectin from the surface of CA fibers treated with 15% NaOH. This is an important factor that affects the fiber's crystalline index and thermo-mechanical properties. The thermal degradation temperature of CA fibers treated with 5% NaOH exhibited an elevated range, escalating from 248 °C to 325 °C, surpassing the values observed in fibers treated with 10% and 15% NaOH. This higher thermal degradation temperature is particularly suitable for processes involving polymerization. Additionally, the surface roughness parameter of fibers treated with 5% NaOH outperformed those treated with 10% and 15% NaOH, indicating superior surface characteristics. The findings from these experimental analyses underscored that the treatment with 5% NaOH spurred noteworthy structural and chemical transformations, thereby enhancing the overall characteristics of CA fibers. Analysis on the raw fiber and treated fibers reveals that the CA fiber suitable alternative natural fiber material for low impact polymer composite materials. These comprehensive characterizations serve to validate the viability of *Careya arborea* fibers as a promising bio-reinforcement option for composite materials.

Future scope

Researchers can explore the integration of hybrid and polymeric composite formulations with cellulose natural fiber extracted from *Careya arborea* linn. To attain improved mechanical and thermal properties, this entails experimenting with different combinations of polymer matrices and reinforcing fibers, such as glass, carbon, or aramid fibers. The objective would be to produce composite materials with the inherent biodegradability of cellulose fibers while exhibiting higher strength, stiffness, and endurance. Future studies can concentrate on creating hybrid and polymeric composites using bio-based polymers and environmentally acceptable additives as the need for environmentally friendly materials increases. This approach aligns with sustainable development goals and addresses environmental concerns associated with conventional composite materials.

Ethics approval and participation consent

Not relevant.

Permission to publish

Not relevant.

Table 5
AFM properties of treated CA fibers.

AFM Parameters	Values
Standard Deviation	31.742 nm nan m
RMS	31.741 nm nan m
Average Deviation	24.136 nm nan m
Skew	0.552 nan
Kurtosis	1.16 nan

Data and material availability

Not relevant.

Conflicting interests

None.

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Not relevant.

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Jeevan Rao H: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **S. Singh:** Writing – review & editing. **P. Janaki Ramulu:** Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Thiago F. Santos:** Writing – review & editing, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Caroliny M. Santos:** Writing – review & editing, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Sanjay M.R:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Indran Suyambulingam:** Writing – review & editing, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Suchart Siengchin:** Writing – review & editing, Visualization, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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