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Effect of Alkali and Permanganate Treatments on *Terminalia catappa* Leaf Fibers: Efficient Reinforcements to Be Used in Eco-Friendly Composites

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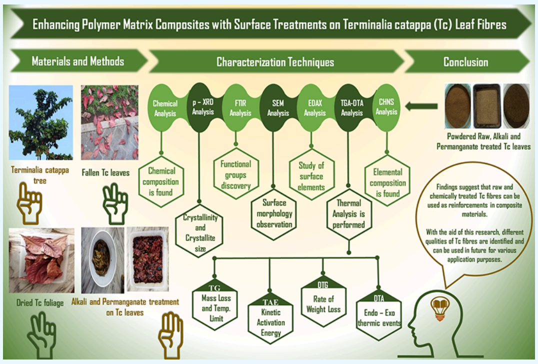

ABSTRACT: Natural fiber-reinforced composites could be obtained by utilizing agricultural wastes, fallen leaves, or abandoned materials as reinforcements after their usage as a way to reduce environmental impacts such as to stop deforestation, i.e., cutting down of plants and trees for their fibers, and in waste management, which includes recyling of natural wastes and minimizing the use of nonbiodegradable synthetic composites by replacing it with their natural fiber counterparts. As an outcome, leaves from a *Terminalia catappa* (Tc) tree grown in the Kanyakumari district that falls off in large quantities throughout the winter are gathered and examined. The leaves are treated with NaOH and $KMnO_4$. In this investigation, all three powdered samples (raw, alkali treated, and permanganate treated Tc leaf fibers) are sent to chemical analysis, powder X-ray diffraction (p-XRD), Fourier transform infrared (FTIR) analysis, scanning electron microscopy (SEM), energy dispersive spectroscopy (EDAX), thermal analysis (TGA-DTA), and carbon hydrogen nitrogen sulfur (CHNS) analysis. The results of the research showed that the powdered Tc leaf samples had high levels of cellulose (≈ 50%), crystallinity index (≈ 50−65%), and crystallite size (≈ 3 nm). There are various vibrational bands associated with them. The rough surface of the raw and treated Tc powder samples and their particle size (≈ 50−100 μm), as revealed by the SEM images, might aid in good adherence with the matrix. According to EDAX and CHNS analyses, there is a high carbon content in all of the samples. The fibers have an activation energy (≈ 55−60 kJ/mol) and maximum temperature limit (≈ 500 °C) that are comparable to many other natural fibers. The density of Tc leaf fibers (≈ 0.2 g/cm^3) is very low and so could be used in lightweight composite applications. The chemical treatments enhanced the qualities of Tc fibers to a certain extent. In various polymers, rubber, or concrete matrices where the inclusion of natural fiber reinforcement is desired, all three samples could function as potential reinforcements.

1. INTRODUCTION

More efficient use of the resources at hand is essential for the social and economic growth of humans. Composites are designed from two or more constituent materials that have very diverse physical or chemical characteristics. They remain macroscopically distinct from one another within a completed structure. The earliest example of the use of composites is found in clay bricks reinforced with straw. When combined, the two components, clay and straw, served the purpose that each could have performed on its own. Glass composites, sometimes known as fiberglass, are used to construct boats and aeroplanes. These composites are made of glass fibers reinforced in resin.[1] The composite material's synthetic fibers harm the environment because they are not compostable. The use of natural fiber as a substitute for glass has become more common in the past 10 years because of potential advantages such as weight reduction, low-cost raw materials, thermal recycling, and the environmental advantages of using renewable resources.[2,3] Yet global efforts to produce natural fiber composites have been sparked by growing environmental consciousness, particularly about recycling conventional materials and the extraordinary depletion of forest resources brought on by global warming. The development of high-performing, completely biodegradable "green" composites, which will be a crucial component in the development of materials to address the present ecological and environmental issues, depends heavily on natural fibers. The natural fiber-reinforced composite is an economical, lightweight, and pleasing use of renewable resources. Both the nation's economy and the

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cultivation of fiber plants will benefit from it. These days, leaf fiber substitutes are discovered to be economically viable and are becoming more and more important as substitute materials in the composites industry.[4]

Natural cellulosic fibers have attracted the attention of researchers due to the current need for materials that are better for the environment. These fibers have successfully replaced synthetic fibers as reinforcements in various applications.[5] Natural fibers are typically extracted from plants. Primary and secondary plants are separated based on how they are used. The primary plants are those that are grown for their fiber. Primary plants include kenaf, hemp, sisal, and jute.[6] Fibers are produced by secondary plants as a byproduct. Examples of secondary plants are coir, oil palm, and pineapple. There is a great diversity of plants and animals in nature.[7] Fiber-producing plants are ranked second among plant species in terms of economic significance after food plants. Thus, it is necessary to identify and assess the characteristics of new and potential fiber plants. The strength, availability, fineness, length, weight, durability, density, texture, chemical composition, wetness, part in which the fibers are present, cost, elastic modulus, and simplicity of fiber extraction are the elements to be taken into account while choosing plant fibers.[8]

The primary constituents that dictate the physical characteristics of fiber are lignin, pectin, hemicellulose, and cellulose. Greater cellulose concentrations are correlated to toughness and strength. Hemicelluloses, lignin, and pectin are responsible for the absorption of moisture, thermal breakdown, and biodegradation of fiber. Depending on the plant, stem section, age, climatic conditions, extraction method, moisture level, etc., specific fiber characteristics and the structure of the fiber might differ significantly. Cellulose makes up the majority of plant fibers.[9] Multiple cell walls make up the fiber. These cell walls consist of semicrystalline cellulose microfibrils that are oriented and reinforce one another, embedded in a matrix of varied compositions made up of hemicellulose and lignin. The fiber acquires mechanical strength from it. The natural fibers must be chemically treated to gain hydrophobicity with appropriate coupling agents or coated with suitable resins to produce composites with improved mechanical and environmental performance. In addition to lowering moisture absorption, this type of fiber surface modification also improves the interfacial bond strength and wettability of the fibers with resin, two crucial elements for enhancing a composite's mechanical qualities. It is possible to copolymerize natural cellulosic fibers by grafts, cover them with polymeric solutions, and apply chemical enhancements.

A high interfacial adhesion is necessary to achieve increased mechanical characteristics. The degree of interfacial attachment is determined by the way matrix molecules cling to the fiber surface through adsorption or chemical reactions. For load transfer, the area where the fiber reinforcement and bulk polymer separate is essential.[10] To comprehend the nature of the interfacial bonding and its characterization, a great deal of research has been done. Enhancing the matrix and fiber's adherence efficiency can be done in a number of ways. Various physical techniques, including stretching, calendering, thermotreating, and hybrid yarn manufacturing, can be employed to enhance the interactions between fibers and matrices.[11] Additionally, chemical treatments on the fiber surface are employed to fortify the contact. Natural fiber reinforced composites can be utilized as environmentally beneficial alternatives to traditional composite fibers in sectors like packaging, automotive, and construction. Plastination is a technique that can be used to increase the environmental durability of natural fibers. This approach uses curable polymers like silicone and epoxy to replace the water and lipids in natural fibers.[12]

Government rules, the increasing awareness of the harm inorganic materials used in many industries due to the environment, and the misappropriation of nonbiodegradable and nonrenewable resources have all influenced the creation of new sustainable materials and products.[13] Numerous nations have made significant investments in research and development and production techniques aimed at reducing vehicle weight to attain fuel economy through automobile manufacturing. To obtain lightweight vehicles, they favor materials based on natural fiber composites over standard materials like steel for certain purposes. Over the projection period, the market is anticipated to grow as a result of people's shifting focus toward biocomposites due to rising environmental awareness and fluctuating fuel prices.[14]

There has been growing interest in recent years to replace traditional synthetic fibers with natural leaf fibers to reinforce polymer resins, as it eliminates the environmental issues and fossil fuel depletion. The leaf fibers such as alfa, cantala, fique, henequen, PALF, sisal, *Sansevieria cylindrica*, *Sansevieria ehrenbergii*, *Sansevieria roxburghiana*, and *Sansevieria trifasciata* reinforced polymer composites are some very popular leaf fiber biocomposites.[15] Another such novel leaf fiber to be used as reinforcements in composite materials, examined in this work, is obtained from *Terminalia catappa* tree, also known as the Java Almond, Tropical Almond Tree, or Indian Almond. It may reach a height of 40 m and is mostly found in Asia, Africa, and Australia. The tree resembles a pagoda, because of the clear levels in which the branches are arranged. The crown of the tree widens and flattens at the top as it ages, creating the appearance of a wide-spreading vase. Large, glossy, leathery, 15-in.-long by 6-in.-wide leaves turn gorgeous hues of red, yellow and purple before falling in the winter.[16] Some people might perceive these enormous old leaves as an annoyance, because of their size. The tree is barren for a little while because new growth replaces the leaves very quickly. The edible fruits follow the small, greenish-white, springtime blooms that grow in terminal clusters that are 6-in.-long. The thin green flesh within is surrounded by a corky fiber husk. The dried fibers of this husk are treated with alkali and studied.[17] Also, these fibers reinforced in a PVAc matrix are also analyzed.[18] The edible kernel, which resembles an almond, is inside. Because the fruit contains a lot of tannic acid, it may discolor sidewalks, pavement, and automobiles.

The local area has an abundance of tropical almond trees. Papermaking might benefit from the use of *Terminalia catappa* as a raw material. Consumption of *Terminalia catappa* leaves and seeds is appropriate due to their high nutritional and mineral salt content. The development of food products may result from the good physicochemical features of *Terminalia catappa* kernel oil, which includes low levels of peroxide and acidity and a high quantity of unsaturated fatty acids. The wood is susceptible to termite attacks while being rather sturdy, strong, moderately heavy, and reasonably durable. Therefore, it can be utilized for floors, paneling, crates and boxes, boats, bridges, and carpentry work.

Twice a year, leaves fall; these can be gathered and put to a variety of uses. The contribution of a plant that serves as a reinforcement for composite materials along with medicinal applications is very rare. One such plant is *Terminalia catappa*.[19]

Figure 1. *Terminalia catappa* leaf fibers: (a) Tc tree, (b) Tc leaves, (c) dried Tc leaf, (d) dried Tc foliage, (e) NaOH pellets, (f) NaOH solution, (g) alkali treatment on Tc leaves, (h) dried NaOH treated Tc leaves, (i) potassium permanganate salt, (j) permanganate solution, (k) permanganate treatment on Tc leaves, (l) dried $KMnO_4$ treated Tc leaves.

Steam exploded Tc fibers are used as reinforcements in polypropylene composites.[20] *T. catappa* is an herb that is commonly used in Ayurveda. Fresh leaf juice is taken internally for headaches and stomach aches, and it is also used to manufacture scabies and leprosy medicinal ointment. Tc leaves can be used to dye cotton in various colors very efficiently. They have been used to create shades on silk garments with good to great rapidity qualities. The residual material can be used as fertilizer after the dye is extracted, and all of the liquid material left over can be used to breed fish. So dyeing is an entirely ecologically conscious technique.[21,22] Abundant *Terminalia catappa* trees are available in the Kanyakumari district, and so these leaves are selected as sample material in this work.

2. METHODOLOGY

2.1. Collection and Chemical Processing of Tc Leaf Fibers. The fallen leaves of the tropical almond tree are big and easy to gather. The Holy Cross College campus in Nagercoil, Kanyakumari district, Tamil Nadu, India is where those leaves are gathered. Pinkish-reddish leaves that fall on a daily basis during winter season are collected and washed. They are then allowed to dry at room temperature. These leaves are ground and sieved finally. The resulting raw Tc leaf powder sample is subjected to a thorough evaluation before considering it as reinforcement in composite structures. Figure 1 depicts the process of gathering and chemically treating Tc leaf fibers.[21,23] The chemicals employed in this study are purchased from Spectrum Reagents and Chemicals Pvt. Ltd., Cochin.

2.1.1. Alkali Treatment of Tc Leaf Fibers. NaOH treatment for natural fibers is the most popular method. This covers four different approaches, including (a) maintaining a constant NaOH concentration for a fixed amount of time, (b) applying varying NaOH concentrations for a fixed duration, (c) using the same NaOH concentration over various time intervals, and (d) utilizing various NaOH concentrations throughout various time intervals. The most popular approaches to ascertain the ideal circumstances for natural fiber alteration are the second and third practice procedures.[24] Natural fiber's thermal stability and thermal resistance are enhanced by alkaline treatment.[25] By giving the fiber surface the right amount of roughness, it raises the tensile strength of composite materials.[26−28] This treatment can potentially provide improved mechanical bonding. For 20 min, the dried *Terminalia catappa* leaves are immersed in a 0.1 M alkali solution. After that, the leaves are allowed to dry at ambient temperature to remove any remaining moisture. Applying large quantities of chemicals and letting them sit for a long time causes the fiber particles to degrade significantly, and therefore 20 min of immersion in alkali solution is performed in this work. To obtain the powdered sample, the leaves are meticulously ground in a mixer once they have dried fully. After that, they undergo an intense sieving process to remove large particles. Following alkali treatment, these Tc fiber samples are sent for a variety of characterization analyses.[23,29]

2.1.2. Permanganate Treatment of Tc Leaf Fibers. Treatments with permanganate are less common but are still effective. Most permanganate treatments involve the use of potassium permanganate ($KMnO_4$). The permanganate treatment, which alters the macromolecular and crystallographic structure of the fibers and improves their tensile properties, superior flexural strength, increased stiffness, and other properties, is one of the most effective methods for improving the binding at the fiber−polymer interface.[30−33]

The following is the procedure for surface modification using permanganate. The dried leaves of Tc have been collected. A sodium hydroxide pretreatment is applied to the leaves. An alkali solution of 0.1 M is made. For around 20 min, the leaves are immersed in the alkaline solution. After that, the leaves are dried for a week at room temperature. The alkali pretreated leaves are immersed in a 0.1 M potassium permanganate in acetone solution for 20 min after they have been completely dried.[24] After that, they are dried for a week at room temperature. The fibers from the dried Tc leaves treated with permanganate are crushed and sieved. The material in powder form is sent out for several characterization inquiries. The three Tc leaf powder samples are displayed in Figure 2.

3. CHARACTERIZATION TECHNIQUES

Figure 3 sketches the characterization techniques used in the study of Tc leaf fibers.

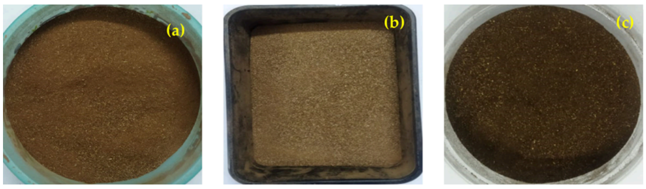

Figure 2. (a) Raw Tc leaf powder. (b) Alkali treated Tc leaf powder. (c) Permanganate treated Tc leaf powder.

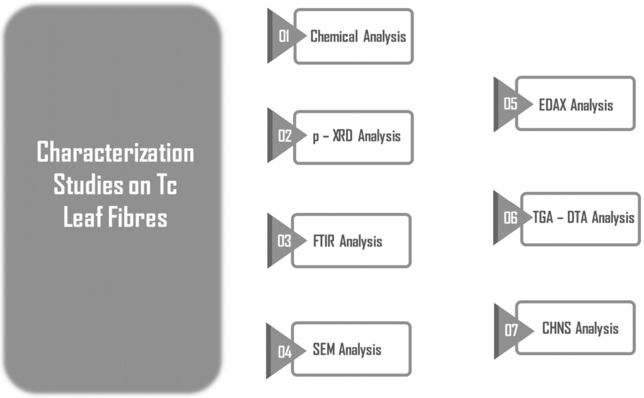

Figure 3. Characterization studies for the analysis of Tc leaf fibers.

3.1. Chemical Analysis. Cellulose, hemicellulose, lignin, pectin, and wax are the principal components of natural fibers. Cellulose gives adequate strength and stiffness to natural fibers. Hemicellulose is responsible for the fiber's ability to absorb moisture. The structural behavior, morphological traits, and fire-resistant behavior of fibers are all influenced by their lignin content. The binding of cellulose fibers to other fiber components is caused by pectin. The amount of ash present in the fibers of composite materials does not affect their mechanical properties. Wax and moisture content in fibers are considered to be impurities. The peculiarity lies in the fact that all fibers have the same elements but differ in composition, leading to diverse behaviors from one fiber to the other. Additionally, the density, ash content, and moisture content are also measured while the samples are being submitted to SITRA (South India Textile Research Association) in Coimbatore, Tamil Nadu, India, for chemical analysis. They perform chemical evaluations on raw and chemically treated *Terminalia catappa* leaf fibers using specific procedures. A natural fiber's constituent parts can be distinguished by their respective weight percentages.[3,34,35]

3.2. p-XRD Analysis. Using X-ray radiation on crystalline organic and inorganic materials is known as the powder X-ray diffraction (XRD) technique. The way that light is diffracted depends on the dimensions, position, and arrangement of the crystal's constituent elements. When photons are dispersed and then potentially interact with each other, they create a unique diffraction pattern exclusive to the crystalline powder. It is possible to distinguish between the crystalline and amorphous phases of a material by creating patterns of X-ray diffraction from a sample.[36−38]

The Tc fibers are cut into thin, powder-like strips, and the resulting powdered leaf fiber samples are studied using a D8 Advance A25 model X-ray diffractometer (made by Bruker, Billerica, MA, USA). Within an operational temperature range of 3 to 70 °C, two scans per minute are conducted as part of the X-ray investigation. Natural fiber crystallographic information is assessed by XRD examination using the crystallite size (CS), crystallinity index (CI), and percentage crystallinity (%Cr) equations.

$$\text{Crystallinity percentage} = \frac{I_{200}}{I_{200+I_{\mathrm{am}}}} \times 100 \tag{1}$$

$$\text{Crystallinity Index} = \frac{I_{200-I_{\mathrm{am}}}}{I_{200}} \tag{2}$$

where I_{200} and I_{am} represent the crystalline and amorphous intensities on the 2θ scale.[39]

$$\text{Crystallite size} = \frac{K\lambda}{\beta \cos \theta} \tag{3}$$

where θ = bragg's angle, $\beta = pi/180 \times \mathrm{fwhm}$, $K = 0.9$ and $\lambda = 1.54060 \times 10^{-10}$ m.[40]

3.3. FTIR Analysis. The ability to analyze any material in any state is one of the main benefits of infrared spectroscopy. It is possible to study, for instance, films, liquids, pastes, gases, powders, solutions, fibers, and surfaces. By using FTIR, which provides an amazing array of tools regarding the characteristics of natural fiber-related composites, scientists can gain a better understanding of natural fibers and the technologies associated with their modification.[41−43]

Based on the vibrational spectra of materials, Fourier transform infrared (FTIR) spectroscopy is a commonly used analytical technique for sample identification and quantification. A Fourier transform infrared (FTIR) spectrometer (manufactured by PerkinElmer, an American Global Corporation) is utilized, and a mortar and pestle is used to grind the fiber sample in order to combine it with KBr. After that, they are pressure-pelletized in a controlled environment. The Tc fiber's FTIR spectra are produced at a resolution of 2 cm^{-1}, a scan rate of 32 scans per minute, and a wavenumber range of 400−4000 cm^{-1}.[33,44]

3.4. Scanning Electron Microscopy (SEM) Analysis. SEM can measure hundreds of fibers automatically in a matter of minutes and produce high-resolution images. According to studies, hemicellulose, lignin, and wax/impurity manifest as minute discontinuous sections, a nail-like morphology, and white layers, respectively. A fiber's surface roughness or smoothness can be determined by analyzing its outward appearance using SEM, which will provide insight into the fiber's bonding properties.[45,46]

The material surfaces are analyzed using an electron microscope (SEM) fitted with a differential scanning system (EDS) to study its topography and morphology. To accomplish elemental detection, an EDS is used. The one used for the sample analysis is the JEOL 6390LV (made in Japan) kind. Its range is 0.5−30 kV, its magnification is 300 000×, and its resolution is 4 nm. All three sample surfaces are captured as images at various magnifications.[47,48]

3.5. EDAX Analysis. The basis for EDS measurement was previously electron beam microanalysis. To create a specimen map, energy-dispersive X-ray spectroscopy (EDX) and SEM (scanning electron microscopy) are combined to assess the kinds and amounts of elements on the surface. For elemental detection, this method is employed. The sample analysis is conducted using the JEOL 6390LV model. Every sample undergoes three analyses, with the average serving as the outcome.[49]

3.6. TGA-DTA Analysis. Thermal analysis is the study of the structural, chemical, and physical changes that a change in temperature makes to a material. Thermogravimetry (TG) and its derivative (DTG) to calculate weight loss and its rate and differential scanning calorimetry (DSC) to calculate heat flow are now considered the fundamental techniques used in thermal investigations. The PerkinElmer Diamond TG/DTA thermal analyzer manufactured in the United States is used for the analysis.[33,46,50]

3.6.1. TG Analysis. TG is an analytical technique that measures a specimen's weight change while heating to ascertain its thermal stability and volatile component proportion. On the other hand, sample weight is measured experimentally at either time or temperature using thermogravimetric analysis (TG). Typically, the sample is heated steadily.[48,51,52]

3.6.2. Thermal Activation Energy Analysis. Activation energy is the minimum amount required to initiate a process such as a chemical reaction. It may be described as the energy required to start the deterioration process in natural fibers. Higher activation energy results in increased fiber thermal stability.[53,54] With the use of the Coats-Redfern approximation, an activation energy is obtained. As a result, the first-order reaction's mathematical expression can be given as follows:

$$\log\left[\frac{-\log(1-\alpha)}{T^2}\right] = \log\frac{AR}{\beta E_a}\left[1-\frac{2RT}{E_a}\right] - \frac{E_a}{2.303RT} \tag{4}$$

where β = linear heating rate, T = absolute temperature, E_a = activation energy, R = gas constant, A = frequency factor, and α = percentage of the sample that has broken down at time t.

$$\alpha = \frac{W_0 - W_t}{W_o - W_f}$$

where W_t is the sample's weight at any given temperature, W_o is the sample's weight at the start of the decomposition process, and W_f is the sample's weight after the reaction. Using a linear fitting procedure done to the plot of

$$\log\left[\frac{-\log(1-\alpha)}{T^2}\right]$$

versus $1000/T$, the activation energy is determined.[39]

3.6.3. DTG Analysis. DTG is the derivative of the TG curve and automatically displayed along with the TG. The rate of change of mass concerning time or temperature is plotted against time or temperature in the data presented as a derivative thermogravimetric (DTG) curve. The rate of mass loss is given by the DTG peak height at any temperature.[55]

3.6.4. DTA Analysis. DTA (differential thermal analysis) is used to estimate endoexothermic event temperatures and to depict phase transitions. With the use of a Mettler Toledo DSC analyzer (model DSC 822e, Ohio, US), the thermal transitions of raw and processed Tc leaf fibers are investigated. An aluminum pan is filled with 3 g of fiber samples for each condition, and it is heated to 750 °C. A steady heating rate of 10 °C/min is maintained throughout the analysis, with a precision temperature of ±0.2 °C.[56]

3.7. CHNS Analysis.

"Flash combustion," a quick and complete oxidation of the sample that establishes the proportions of hydrogen, nitrogen, carbon, and sulfur in the molecules of organic matter, is the basis of an automated Elementar Vario EL III model CHNS/O Elemental Analyzer (made in Germany). Calculating the amounts of C, H, N, and S in natural sources which typically burn at 1800 °C is an efficient use of this technique.[57]

4. RESULTS AND DISCUSSIONS

4.1. Chemical Analysis.

The chemical makeup of fibers influences their physical characteristics. Moreover, the chemical makeup of lignocellulosic fiber is influenced by the kind of plant, its age, the climatic and geographic conditions in which it grows, and the method of harvesting the fiber. The mechanical characteristics of Tc leaf fiber composites will be influenced by the chemical composition, cellulose content, and fiber cell wall structure of Tc leaf fibers. The quantity of Tc leaf fiber's chemical makeup is shown in Table 1, and the comparison charts for the three samples are given in Figures 4, 5, and 6.[1]

Table 1. Chemical Constituents of Tc Fibers

Chemical components	Raw Tc leaf fibres (%)	Alkali treated Tc leaf fibres (%)	Permanganate treated Tc leaf fibres (%)
Cellulose	50.77	52.22	48.72
Hemicellulose	27.45	30.86	30.83
Lignin	18.08	21.59	17.56
Pectin	8.37	12.91	18.55
Wax	3.73	3.42	3.75
Ash content (on dry basis)	8.01	14.08	14.93
Moisture content	10.52	10.84	8.57
Density (g/cm^3)	0.23	0.24	0.26

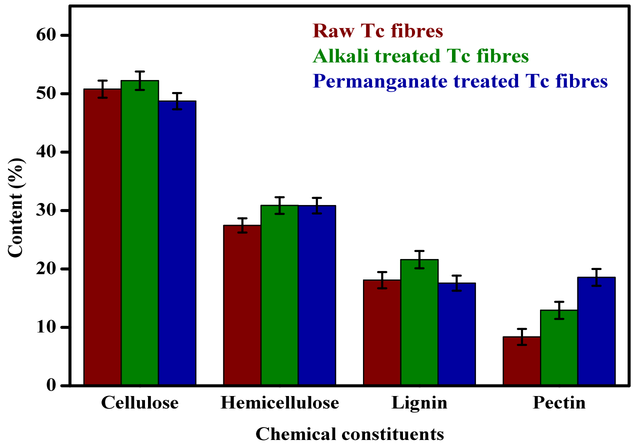

Figure 4. Chemical constituents of Tc fibers.

The compounds in cellulose have a significant impact on the mechanical, fire resistance, biodegradability, and resilience of fibers. Numerous uses exist for cellulose, a biopolymer that is plentiful.[34] The cellulose content of plant fibers is the primary factor determining their stability. The superior mechanical properties of the composite materials containing Tc leaf fibers may be attributed to the high cellulose content, which is around 50.77% for raw Tc, 52.22% for Tc treated with alkali, and 48.72% for Tc treated with permanganate. The cellulose chain's hydrogen bonding can increase crystallinity, which will enhance the mechanical qualities of composites. The majority of lignocellulosic agricultural byproducts have 40−45% cellulose.[58] Compared to bamboo (26−43%), kudzu (33%), pandan duri (43.21%), piassava (28.6%), sugar palm fibers (43.88%), and coconut tree leaf sheath (27%), Tc leaf fibers have a higher cellulose concentration. Tc fibers do, however, have less

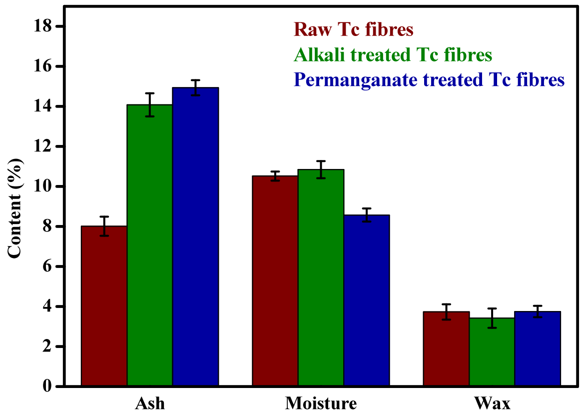

Figure 5. Residual constiuents of Tc fibers.

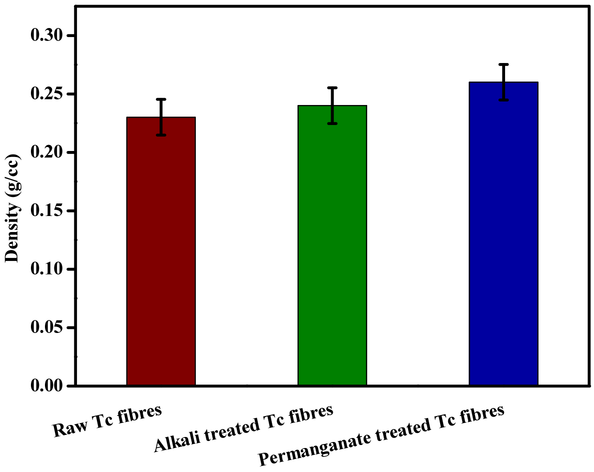

Figure 6. Density chart of Tc fibers.

cellulose than many other well-known natural fibers including sisal (67–78%), *Coccinia indica* (64.56%), abaca (56–63%), sea grass (57%), flax (64–71%), hemp (70.2%), banyan tree roots (67.32%), jute (61%), and hemp (70.2%). It is similar to eucalyptus (50.30%), banana bracts (56.48%), *Acacia concinna* (59.43%), wheat straw (51%), kenaf (53.14%), and *Catharanthus roseus* (47.3%).[59,60]

The presence of hemicellulose causes the microfibrils to degrade and also reduces the fiber's strength. The fiber's high hemicellulose concentration improves its thermal properties, moisture absorption, and biodegradability. Tc leaf fibers have a hemicellulose content of 27.35% in raw, 30.86% in alkali-treated, and 30.83% in permanganate-treated samples. Table 1 indicates that Tc fiber has a medium amount of hemicellulose concentration. Between cellulose and lignin, hemicellulose functions as a catalyst. Tc fibers have a hemicellulose concentration that is similar to that of several other natural fibers, such as bamboo (30%), piassava (25.8%), napier grass strands (31.27%), and *Cyperus dicrostachus* (22.72 ± 9.61%). In comparison to Tc leaf fibers, the following have less hemicellulose content: hemp (15–22.4), *Cryptostegia grandiflora* (17.20%), sisal (12%), flax (18.6–20.6%), coconut tree leaf sheath (14%), wheat straw (15–31%), *Prosopis juliflora* (16.14%), and ramie (13–16%).[10,11]

The presence of lignin affects the shape, characteristics, and structure of fibers. The plant gets its rigidity from lignin. Compared to other lignocellulosic fibers, the lignin level of *Terminalia catappa* leaf fibers that are raw (18.08%), alkali-treated (21.59%), and permanganate-treated (17.56%) is comparatively high. While lignin's low proportion contributes to the fiber's structural qualities, its higher content protects the fiber from biological attacks. The lignin contents of the root fibers of *Coccinia indica* (12.55%), *Zea mays* (19.04%), *Acacia concinna* (14.64%), and *Tithonia diversifolia* (15.06%) are comparable to that of Tc fibers.[6,61]

The presence of a minimal amount of wax is essential for the better interfacial bonding between the polymer and natural fiber. The low wax percentages of 3.73% for raw Tc, 3.42% for Tc treated with alkali, and 3.75% for Tc treated with permanganate help to promote efficient matrix adherence when developing composite materials. Improved adhesion between the fiber and resin will be facilitated by the rough surface of the fiber that results from the removal of wax. The wax concentrations of some additional natural fibers are as follows: *Cyperus dichrostachus* (1.55%), flax (1.5–1.7%), sisal (2%), abaca fibers (3%), nettle (3.1–4.2%), banana inflorescence bracts (1.05%), *Catharanthus roseus* (2.3%), okra (3.9%), *Albizia saman* (1.5%), and *Eleusine indica* grass (2.9%).[62]

The fiber's resistance to fire is enhanced by its low ash content. The high ash concentration of Tc fibers, whether they are raw (8.01%), alkali-treated (14.08%), or permanganate-treated (14.93%), reduces the material's fire resistance and leads amorphous components to be lost.[63,64] The ash concentration of Tc leaf fibers is higher than that of *Cyperus dichrostachus* (1.3%), *Prosopis juliflora* (5.2%), *Tithonia diversifolia* (6.78%), and *Cyperus pangorei* (3.56%).[13,65]

The Tc fibers have a moisture content of 10.52% when they are raw, 10.84% when they are treated with alkali, and 8.57% when they are treated with permanganate. It is on par with other natural cellulosic fibers and similar to *Bauhinia vahlii* (10.5%) fibers. Tc fibers are desirable for creating lightweight materials due to their low density. In comparison to many other natural fibers, it is extremely low.[9,66]

Plant fibers that are chemically processed usually have different chemical compositions. The cellulose content of Tc fibers grew positively upon alkali treatment. Simultaneously, alkalized Tc fiber's hemicellulose increased. Another beneficial change is the decrease in wax in the Tc fiber that has been alkali-treated. Increased ash concentration demonstrated how the cellulose fraction of Tc fibers treated with alkali had improved. Tc fiber density increased somewhat following alkali treatment, as predicted. The cellulose microfibrils are not impacted by the alkali treatment. In composite applications, the elimination of contaminants may result in improved mechanical properties, fiber-wetting characteristics, and fiber-matrix adhesiveness. The application of permanganate led to a reduction in the cellulose and lignin contents and a rise in the hemicellulose and pectin content. As a result of individual fibers becoming detached during treatment, the reduction in the lignin proportion has a positive impact. In the same manner, the moisture content dropped while the wax, ash, and density increased.[2,8,23]

4.2. p-XRD Analysis.

The X-ray diffraction patterns of untreated and chemically treated Tc leaf fibers are displayed in Figure 7. All patterns have two prominent peaks. The crystalline peak, a strong, highly intense peak in all three diffraction patterns, amply demonstrates the cellulose content. It conveys the Tc fibers' crystalline character. Typically, noncellulose materials are indicated by an amorphous peak that appears before a crystalline peak with a lower intensity. The cellulose crystallinity is quantified using the crystallinity index (CrI).

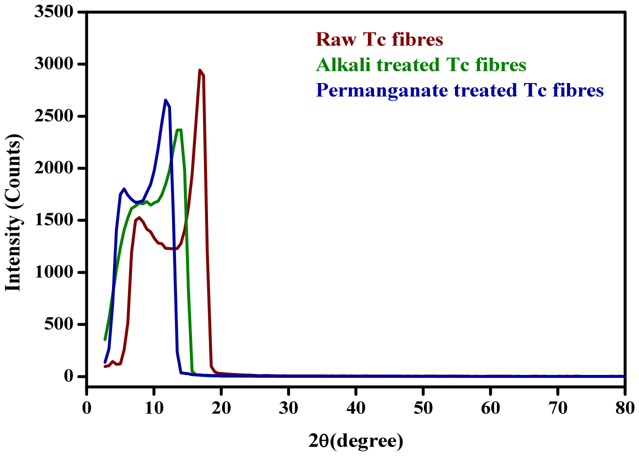

Figure 7. p-XRD pattern of Tc fibers.

Table 2. Crystallographic Data of Tc Fibers

Sample	% Crystallinity	Crystallinity index	Crystallite size (nm)
Raw Tc leaf fibres	65.70	0.47	3.62
Alkali treated Tc leaf fibres	58.54	0.29	3.10
Permanganate treated Tc leaf fibres	59.74	0.32	3.85

While increased CrI tends to make fibers more brittle, it also makes them stronger. The crystalline percentage decreased after surface treatments because the crystalline peak's intensity decreased. As a result, the treated Tc fibers' crystallinity index (CI) also decreased. Details gained from each Tc sample's diffraction pattern are displayed in Table 2. Tc fibers have crystallinity percentages that are similar to those of *Acacia planifrons* (65.38%), agave fibers (64.4%), *Sansevieria cylindrica* (60%), napier grass strand (62.4%), and *Bauhinia vahlii* (56%). While Mengkuang leaves (69.5%), *Careya arborea* (85.5%), soy hulls (69.6%), banyan fiber (72.47%), and *Cannabis sativa* (88%) have higher crystallinity than Tc fibers, sugar cane bagasse (50%), *T. Populnea* (48.17%), *Sansevieria ehrenbergii* (52.27%) and *Furcraea foetida* (52.6%) have slightly lower percentages of crystallinity than Tc fibers.[1,2,7]

Less surface area due to a greater crystallite size means less water absorption and chemical reactivity in the fibers. Raw Tc fiber crystallite sizes improved from 3.62 to 3.85 nm after permanganate treatment, which is equivalent to reported cellulose crystallite sizes (4 to 7 nm usually). Tc fiber's crystallite sizes are similar to those of other naturally occurring fibers, such as *Pithecellobium dulce* (3.46 nm), *Retama raetam* (3.62 nm), jute (3.58 nm), *Bauhinia vahlii* (3.85 nm), and cornstalk (3.8 nm). *Careya arborea* (7.40 nm), *Ficus religiosa* root (5.18 nm), *Albizia julibrissin* (6.75 nm), *Piliostigma racemose* (5.25 nm), aerial roots of banyan fiber (28.36 nm), and ramie (16 nm) are among the fibers that exhibit larger crystallite sizes.[6,61,66,67]

4.3. FTIR Analysis.

Figure 8 displays the FTIR spectra of Tc fibers that have been chemically treated and those that are raw. The spectra of Tc fibers show the distinctive peaks of cellulose, hemicellulose, pectin, lignin, and other components. Table 3 includes a list of the significant wave numbers, corresponding chemical components, and stretching.

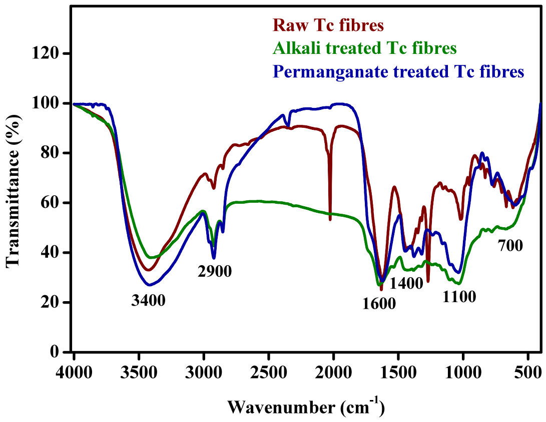

Figure 8. FTIR spectra of Tc fibers.

Table 3. Vibrational Band Assignments of Tc Fibers

Wavenumber (cm^{-1})				
Raw Tc leaf fibres	Alkali treated Tc leaf fibres	Permanganate treated Tc leaf fibres	Vibrational band assignments	References
3425.35	3409.94	3417.09	O–H stretching of cellulose bonded to hydrogen	6, 69
2922.34	2922.26	2922.46	C–H stretching of cellulose	70
2852.34	2853.18	2853.46	stretching vibration of hemicelluloses with C–H symmetry	71
2327.78		2348.69	wax or wax likes substance	72
1631.54	1649.19	1621.81	carboxyl stretch of C–O, signifying hemicellulose contains an acetyl group	1, 73
	1535.70		C=C group of lignin	74
1436.41	1430.70	1447.49	$C-H_2$ wagging	75
	1383.63	1380.24	asymmetric COC stretching of lignin	1, 71
1350.40	1323.41		C–O groups in polysaccharide aromatic ring	2
	1238.10	1236.02	acetyl group's C–O stretching vibration in lignin	6, 7
1156.85		1159.26	C–O–C group of cellulose and hemicellulose	6
1111.14	1103.88	1099.59	C–O stretching	66
1020.53	1033.98	1036.46	CO group of cellulose	61
831.05	826.05	831.84	lignin components	7, 74
762.04	780.66	778.16	β-glycosidic bond connecting the monosaccharide ring	69
668.01	673.22		vibration of out-of-plane bending involving rings	71
617.12		600.37	C–X stretching of organic halogen compounds	72
574.21			OH bending out of plane	5, 73

In both the raw and processed spectra, nine common peaks—the ideal peaks for plant fibers—can be seen at various wavelength ranges. The hydrogen-bonded OH groups in the cellulose are responsible for all three spectra's broad peaks at roughly 3400 cm^{-1}. The apparent peaks at 2900 cm^{-1} are related to the C–H stretching of cellulose. The peaks at 2800 cm^{-1} demonstrate the C–H symmetric stretching of hemicellulose. Wax has been eliminated from alkali-treated Tc fibers, as evidenced by the removal of stretching at 2300 cm^{-1} from the spectrum of alkali-treated Tc fibers. The presence of an acetyl group is indicated by the peaks at 1600 cm^{-1}. The wiggling of $C-H_2$ is connected to the peaks at about 1400 cm^{-1}. The

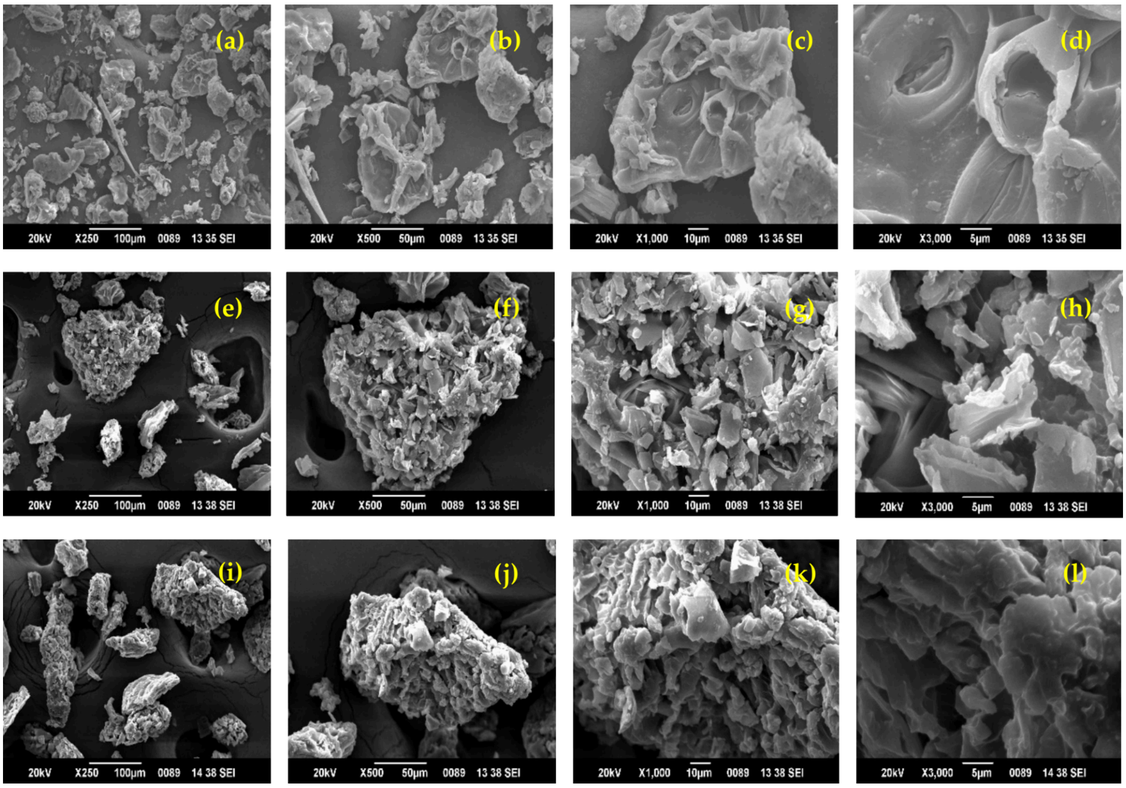

Figure 9. SEM images of powdered Tc fibers. (a−d) Raw Tc leaf fibers; (e−h) Alkali treated Tc leaf fibers; (i−l) Permanganate treated Tc leaf fibers.

presence of the CO groups is shown by the peaks at 1100 and 1000 cm^{-1}. The peaks at 800 cm^{-1} show the lignin components of the Tc fibers. The connection between monosaccharide rings via β-glycosidic bonding is indicated by the peak at 700 cm^{-1}.[1,2,6,7,61,66]

The peak at 1535 cm^{-1} indicates the presence of the C=C group of lignin. This peak is absent in raw and permanganate treated fibers. The inference made from the presence of this peak is that there is a high quantity of lignin present in alkali treated Tc leaf fibers than raw and permanganate treated samples. This view is supported by chemical analysis results, too (Table 1 and Figure 4). The three peaks approximately at 600 and 500 cm^{-1} are present in the IR spectra of raw Tc samples. Due to permangante treatment, the peak due to the vibration of out-of-plane bending disappeared. Because of alkali treatment, the peak due to stretching of halogen compounds got eliminated. The peak which accounts for OH bending, which is out of plane, vanished during both alkali and permanganate treatment of Tc fibers.[68]

4.4. SEM Analysis. At varying magnifications, the surface characteristics of both raw and surface-modified fibers can be observed with scanning electron microscopy. The ability of a fiber to provide a reinforcing effect for composites based on polymers is greatly influenced by its surface morphology. High-resolution SEM images are displayed in Figure 9 and are utilized to identify the microcracks and micro holes on the powdered Tc fiber surface, respectively. Also, the particle size can be identified from SEM images of the powdered samples. The three samples of Tc leaf fiber powder have particles with sizes approximately in the range of 50−100 μm in diameter. The variation in the size of the particles in the same sample can be attributed to the nonhomogeneous grinding of the leaf samples. The geometries of the shape of particles in all three samples are irregular.[76] The fiber surface's pores and cracks may provide a large contact area with the polymer matrix. The irregular geometry and surface roughness will aid in improved interfacial adhesion between the fiber and matrix. A white layer on the raw fiber's surface indicates the presence of contaminants. To remove contaminants and improve the bonding of the interfacial surfaces, these samples underwent chemical treatment. The surface of chemically treated Tc fibers is rougher than that of the raw fibers, as clearly seen from Figure 9.[1,2,6,61,67,77]

4.5. EDAX Analysis. On the outer layers of both raw and treated Tc fibers, the distribution of different components is illustrated in Figure 10.

Table 4 provides a summary of the atomic and weight percentages of the elements on the fiber surface. Carbon (C) and oxygen (O) contents in treated and untreated cellulosic fibers are often higher than those of other elements. The elimination of lignin, wax and other contaminants from the exterior surface of the treated Tc fibers is demonstrated by the decreased concentrations of magnesium (Mg) and chlorine (Cl) in those fibers. Only the surface of Tc fibers treated with alkali has a tiny quantity of sodium (Na); this may be the result of sodium being deposited on the outer layer of the fiber during the alkalization treatment.[2,61]

The oxygen-to-carbon ratio in natural fibers is to be measured using EDAX analysis. A fiber that has more carbon also contains more lignin. Tc leaf fiber's carbon content improved while their oxygen content declined following an alkali treatment. In the same way, the carbon concentration decreased, and the oxygen content climbed following permanganate treatment. The higher O/C ratio suggested a lower level of lignin. Fibers with a lower O/C ratio are thought to be more hydrophobic.[78−80] The O/C ratio for Tc fibers is provided in Table 5.

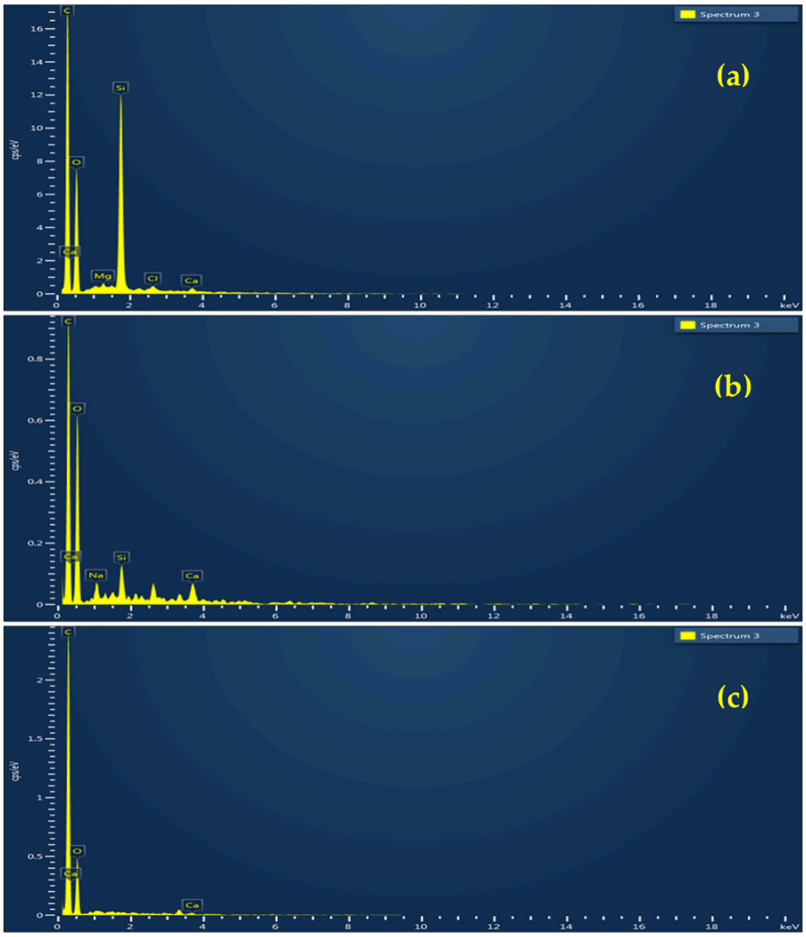

Figure 10. EDAX Spectra of Tc fibers. (a) Raw Tc leaf fibers; (b) Alkali treated Tc leaf fibers; (c) Permanganate treated Tc leaf fibers.

Table 4. Weight and Atomic % of Elements in Tc Fibers

	Raw Tc leaf fibers		Alkali treated Tc leaf fibers		Permanganate treated Tc leaf fibers	
Elements	wt %	atom %	wt %	atom %	wt %	atom %
C	62.27	70.84	71.52	74.83	72.68	66.66
O	29.49	25.19	26.3	24.04	27.27	33.32
Ca	0.27	0.09	0.69	0.26	0.05	0.02
Mg	0.21	0.12				
Si	7.6	3.7	0.83	0.44		
Cl	0.15	0.06				
Na			0.66	0.43		

Table 5. O/C Ratio of Tc Fibers

	Elements (atom %)			
Sample	O	C	O/C ratio	Lignin content (wt %)
Raw Tc leaf fibers	25.19	70.84	0.35	18.08
Alkali treated Tc leaf fibers	24.04	74.83	0.32	21.59
Permanganate treated Tc leaf fibers	33.32	66.66	0.49	17.56

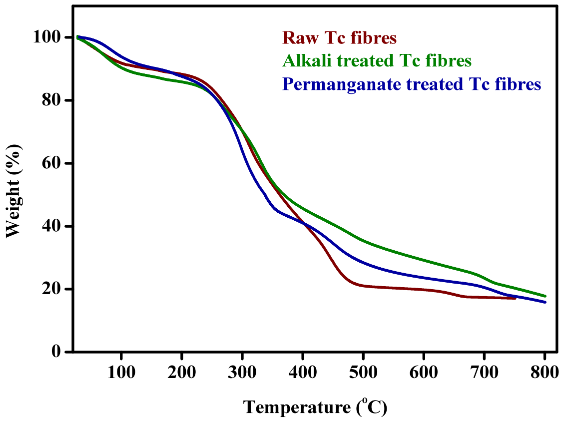

Figure 11. TG plot of Tc fibers.

4.6. TGA–DTA Analysis. *4.6.1. TG Analysis.* The TG curves (Figure 11) demonstrated the three stages of mass loss in both raw and chemically treated Tc fibers. Many branches make up the lignin structure's complicated aromatic ring component, which causes the breakdown to occur at a modest weight loss rate throughout the course of its whole temperature range, which is room temperature to 800 °C. Moisture evaporation is suggested as the first step of mass deterioration. The subsequent phase of mass loss pertains to the elimination of hemicelluloses, a small amount of lignin, and a significant amount of cellulose inside the fiber. The second stage, where deterioration flashed the exclusion of cellulosic components, is when there is a major mass loss. Wax and lignin are excluded during the third step of the degradation. Finally, the char residue marks the end of the breakdown. Because lignin contains many functional groups with varying thermal stabilities, it decomposes more slowly than cellulose and hemicellulose over a wide temperature range.

Tables 6 and 7 present the Tc fiber thermal analysis. Untreated Tc fibers have been found to have a maximum temperature limit of 487 °C; however, following treatments with alkali and permanganate, the temperature increased to 495 and 504 °C, respectively. Thus, it may be said that fibers treated with permanganate have the greatest resistance to heat deterioration and fibers treated with alkali come in next. Compared to other Tc fiber samples, the mass loss experienced by alkali-treated fibers is the least. For raw Tc fibers, the residual char is 17.57%. For Tc fibers treated with alkali, it is 23.54%; and for Tc fibers treated with permanganate, it is 20.95%. Figure 11 illustrates the thermal stability of Tc fiber samples, in both their raw and chemically modified states, up to 800 °C. This demonstrates that Tc fibers, with their relatively high starting temperature, are suitable for application as reinforcement in composites, particularly those based on polymers.[6,66,81]

Table 6. Thermal Analysis of Tc Fibers

Sample	Temperature during mass loss (°C)	Mass loss (%)	Residual char at 700 °C (%)	Thermal activation energy (kJ/mol)
Raw Tc leaf fibres	33−195	11.42	17.57	59.20
	195−350	34.82		
	350−487	32.53		
Alkali treated Tc leaf fibres	28−208	14.16	23.54	57.40
	208−356	32.53		
	356−495	17.40		
Permanganate treated Tc leaf fibres	41−208	13.04	20.95	56.73
	208−347	40.32		
	347−504	17.63		

Table 7. T_{max} Limit of Tc Fibers

Sample	Total mass lost (%)			Max. temperature limit (°C)	$T_{(50\%)}$ (°C)
	First stage	Second stage	Third stage		
Raw Tc leaf fibres	11.42	46.24	78.77	487	359
Alkali treated Tc leaf fibres	14.16	46.69	64.09	495	373
Permanganate treated Tc leaf fibres	13.04	53.36	70.99	504	337

4.6.2. Thermal Activation Energy Analysis. Figure 12 displays the Coats−Redfern plot of chemically treated and untreated Tc fibers. Kinetic activation energy (E_a) is a significant additional component that encourages the usage of reinforcing in high-temperature applications.[82] It can alternatively be seen as the minimum energy needed for a fiber sample to begin degradation. Chemical treatments caused the kinetic activation energy of Tc leaf fibers to drop.[2] The kinetic activation energies of raw and chemically treated Tc fibers are found and tabulated in Table 6.

The kinetic activation energies of some natural fibers which are found from literature are *Lygeum spartum* (68.77 kJ/mol), *Dracaena reflexa* (68.784 kJ/mol), *Furcraea fetida* (65.64 kJ/mol), *Careya arborea* (74.62 kJ/mol), *Cissus quadrangularis* root (74.18 kJ/mol), and *Prosopis juliflora* (76.72 kJ/mol).[1,7,61] These natural fibers have activation energy values higher than all the three samples of Tc leaf fibers.

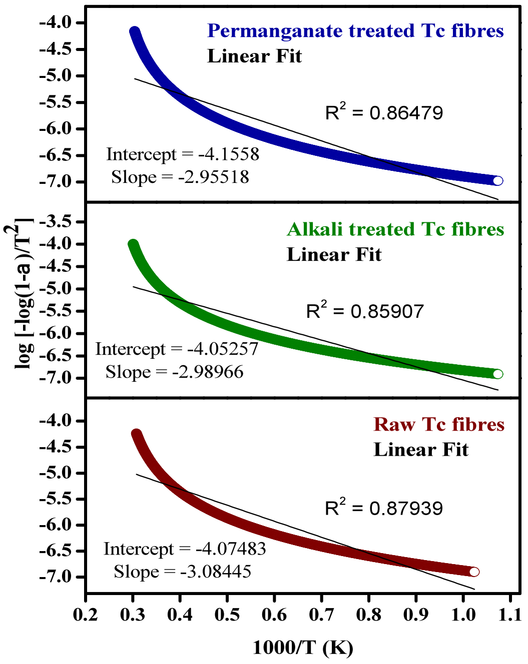

Figure 12. Coats−Redfern plot of Tc fibers.

4.6.3. DTG Analysis. As seen in Figure 13, for each of the three samples, degradation is represented by three peaks in the DTG curve. The evaporation of moisture is the cause of the initial peak. The depolymerization of both cellulosic and hemicellulosic components in the Tc leaf fibers is linked to the second peak. The third peak is the result of residues in the Tc fibers and the breakdown of the noncellulosic component, lignin.

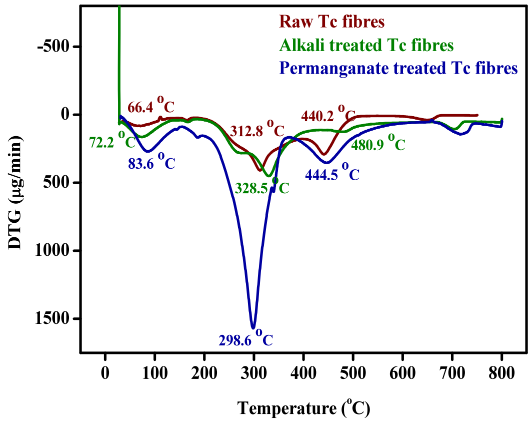

Figure 13. DTG curve of Tc fibers.

The maximum degradation rate of raw, alkali-treated, and permanganate-treated Tc fibers happens at 312.8 °C, 328.5 °C, and 298.6 °C according to the DTG data (Table 8). The DTG profiles (Figure 13) indicate that, for all three samples, the maximum temperature at which cellulose degrades is within the range of 250 to 350 °C.[83−85] The cellulose degradation has been connected to comparable peaks for various kinds of fibers, including *Lygeum spartum* (338.7 °C), *Thespesia populnea* (323.8 °C), *Chloris barbata* (324.6 °C), and the root of *Cissus quadrangularis* (328.9 °C).[8,86]

And at last, burned residue started to develop. Since noncellulosic components have the potential to promote higher

Table 8. DTG Data of Tc Fibers

Sample	Temperature (°C)	Rate of weight loss (μg/min)
Raw Tc leaf fibres	66.4	79.1
	312.8	406.6
	440.2	287.5
Alkali treated Tc leaf fibres	72.2	158.3
	328.5	447.6
	480.9	121.5
Permanganate treated Tc leaf fibres	83.6	268.7
	298.6	1555.4
	444.5	344.0

char formation, the samples had a higher charred residue. The samples' good heat stability is indicated by the substantial char production of Tc fibers.[67]

4.6.4. DSC Analysis. When a fiber material is heated, DSC analysis is used to calculate the amount of heat produced or absorbed during the chemical reactions between its components. At various temperatures during the breakdown process, a range of exothermic and endothermic events take place. The thermal phase transition of natural fibers is demonstrated by the endothermic and exothermic peak sizes and locations. Exothermic events cause the release of heat as opposed to endothermic reactions, which cause the sample to absorb heat. The thermal history of materials can be confirmed by measuring with a DSC.

Figure 14 shows the differential scanning calorimetric profiles of the chemically treated and raw Tc fibers. For raw fibers, there are two exothermic peaks seen. Fibers treated with alkali show two peaks: an exothermic peak and an endothermic peak. The fibers that have been treated with permanganate exhibit two exothermic and one endothermic peak. The endothermic peak observed within the temperature range of 38−153 °C corresponds to the vaporization heat of the water absorbed by the fibers and may be associated with loss of water molecules from the surface or the interstitial spaces within the fibers. The second exothermic peak indicates the decomposition of polysaccharides other than lignin such as α-cellulose. The third exothermic peak at around 400 °C may be attributed to the oxidative degradation of the charred residue.[87] Table 9 also provides the details regarding relevant energy consumption.[2,35]

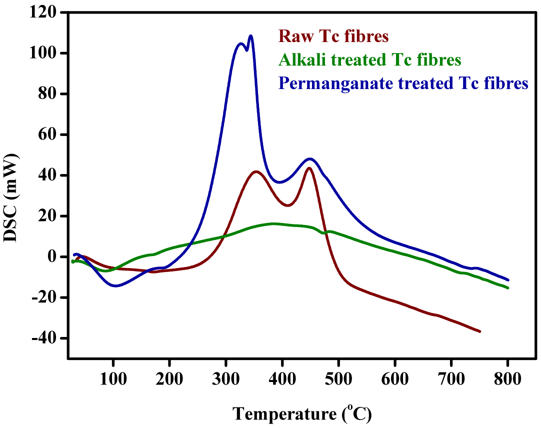

Figure 14. DSC curve of Tc fibers.

4.7. CHNS Analysis. Tc fibers have a carbon content of 37.97% when they are raw, but the percentage rises when they are treated with alkali and permanganate, according to a CHNS study (Figure 15). The high carbon content of natural fibers is one of the primary factors that could change the tribological and mechanical features of the finished product. All of the samples have the potential to be used as fillers that are conductive, in materials that exhibit dielectric loss.

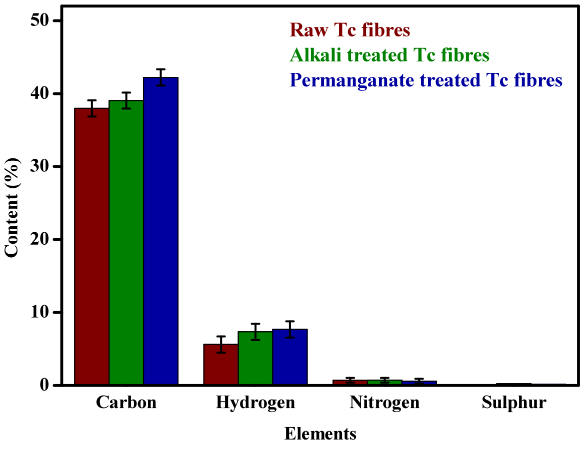

Figure 15. CHNS chart of Tc fibers.

Hydrogen is the second most abundant element in Tc leaf fibers, after carbon (Table 10). It is also discovered that chemical treatments cause it to rise as well. All of the samples include trace levels of nitrogen. Trace levels of sulfur have been found in processed samples but not in raw Tc fiber samples. Tc fibers have a carbon composition that is similar to many other natural fibers.[39,88,89]

Table 10. CHNS Table for Tc Fibers[a]

Sample	C %	H %	N %	S %	Sample weight (mg)
Raw Tc leaf fibres	37.97	5.62	0.70	ND	6.72
Alkali treated Tc leaf fibres	39.05	7.35	0.72	0.25	10.69
Permanganate treated Tc leaf fibres	42.22	7.68	0.59	0.15	10.61

[a] ND: not detected.

Table 9. DSC Data of Tc Fibers

Sample	Temperature at peaks (°C)			Energy consumption (mW)		
	Peak 1	Peak 2	Peak 3	Peak 1	Peak 2	Peak 3
Raw Tc leaf fibers		353.7 (exo)	448.3 (exo)		41.86	43.24
Alkali treated Tc leaf fibers	86.6 (endo)	389.1 (exo)		−6.70	16.19	
Permanganate treated Tc leaf fibers	99.5 (endo)	344.1 (exo)	450.1 (exo)	−13.80	107.09	48.02

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5. CONCLUSIONS

To check if they may be utilized to make composite materials, the powdered fibers from Tc leaves that fall off in the winter are gathered and analyzed. Fibers from fallen *Terminalia catappa* leaves underwent a variety of analyses, including chemical, structural, morphological, elemental, spectroscopic, and thermal evaluations. Deforestation may be decreased when these leftover leaves are utilized as reinforcements in composites reinforced with natural fibers. The fibers are chemically treated to improve their properties using sodium hydroxide and potassium permanganate solutions. To characterize the raw, alkali treated, and permanganate treated Tc fiber samples, chemical analysis, p-XRD, SEM-EDAX, FTIR, CHNS, and TG-DTA investigations have been carried out on them. The variations between the properties of the fiber before and after chemical treatments may be found as follows.

- Chemical analysis revealed that the quantities of hemicellulose, pectin, ash content, and fiber density increased after chemical treatments. Following alkali treatment, cellulose, lignin, and moisture content increased, while wax content decreased. The vice versa took place post permanganate treatment. The density of Tc fibers is much less than many other natural fibers.
- The p-XRD analysis showed that following chemical treatments, the crystallinity index and % crystallinity of Tc fibers dropped. After being treated with permanganate, the crystallite size grew. Nevertheless, following alkali treatment, the crystallite's size shrank. The percentage crystallinity values are high for three samples when compared to many other natural fibers.
- All Tc fiber samples had the same peaks in their FTIR spectra, although the treatments had different intensities of the peaks. Treatments also caused some peaks to appear and vanish.
- The SEM data showed that the surface of Tc powder had been roughened by chemical treatments, which also produced micropores and microcracks. The particle size is also found from these images.
- Tc fibers contain higher surface concentrations of carbon and oxygen, according to the EDAX investigation. Having high carbon content helps in better bonding with matrix.
- TGA-DTA demonstrated that treatments increased the maximum temperature limit for Tc fibers. It has been observed that treatments with alkali and permanganate in Tc leaf fibers slightly reduce the activation energy values. The rate of weight loss is found from DTG curves. The phase transition of sample materials is explained using exo- and endothermic events using DSC.
- Tc leaf fibers had an impressive carbon content, according to the CHNS research. Next in order of importance is hydrogen, followed by nitrogen. Additionally, it has been noted that, after treatments, the concentrations of carbon and hydrogen increased.

These findings suggest that Tc fibers can be used as reinforcements in composite materials, whether they are left untreated or chemically modified. To benefit society, those composites can be suggested for structural applications such as floor mats, automotive interior panels, and seatbacks, whereby lightweight and medium strength are the main concerns. To look into the variations in fiber characteristics, a variety of further surface treatments can be applied. Also, Tc leaf fibers from various other districts of Tamil Nadu can be collected, and the difference in their qualities due to geographical locations can be analyzed. The development of composites based on Tc leaf fibers and their quality testing will be the next important stage. The product that is under consideration can be used as reinforcement in a variety of matrices, including concrete, epoxy, bioplastic, and natural rubber. Furthermore, in addition to Tc leaf fibers, any other natural fiber can be added during the fabrication process of composites to analyze the properties of hybrid composites.

The study can benefit from further application-focused research that uses BET for the same samples to determine the surface area of the powdered Tc fiber particles. Different milling techniques can be used to prepare Tc powders, and the differences in their properties can be investigated. The study of ultrafine natural fiber powder is still relatively new, with only a few research groups involved. Future studies in this area will primarily concentrate on reducing the energy and chemical needs for the production of fine particles. Their application in environmental products, like those that purify water and air, is also possible. To propel such breakthroughs, it would be necessary to attain precise control over particle morphology and size, as well as the capacity to functionalize and tailor the attributes of particle surfaces.

■ ASSOCIATED CONTENT

Data Availability Statement

All the data are available in the manuscript.

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Author Contributions

S.A.K.: conceptualization; methodology; investigation; validation; writing—original draft. R.K.P.: conceptualization; methodology; investigation; validation; writing—original draft. K.P.A.: conceptualization; methodology; investigation; validation; writing—original draft. M.A.: investigation; validation; writing—review draft. C.B.Á.: investigation; formal analysis. S.A.: validation; formal analysis investigation; validation. All authors have read and agreed to the published version of the manuscript.

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REFERENCES

(1) Selvaraj, M.; Chapagain, P.; Mylsamy, B. Characterization Studies on New Natural Cellulosic Fiber Extracted from the Stem of Ageratina Adenophora Plant. *J. Nat. Fibers* **2023**, *20* (1), No. 2156019.

(2) Ganapathy, T.; Sathiskumar, R.; Senthamaraikannan, P.; Saravanakumar, S. S.; Khan, A. Characterization of Raw and Alkali Treated New Natural Cellulosic Fibres Extracted from the Aerial Roots of Banyan Tree. *Int. J. Biol. Macromol.* **2019**, *138*, 573−581.

(3) Anjum, N.; Suresha, B.; Prasad, S. L. A. Influence of Water Ageing on Mechanical Properties of $CaCO_3$ Filler Filled Epoxy Resin and Sansevieria /Carbon Fiber Reinforced Composites. *Open J. Compos. Mater.* **2019**, *09*, 1−20.

(4) Parusha, O. A.; Rozikin, M. N.; Laksono, A. D.; Tajalla, G. U. N.; Putri, N. A.; Rochim, F. M. F. Characterization of Bark-Midrib Fibers Using Chemical Treatment Variations as Reinforcement in Bark-Midrib Fiber Composites. *SPECTA J. Technol.* **2019**, *3*, 36−45.

(5) Sanjay, M. R.; Madhu, P.; Jawaid, M.; Senthamaraikannan, P.; Senthil, S.; Pradeep, S. Characterization and Properties of Natural Fiber Polymer Composites: A Comprehensive Review. *J. Clean. Prod.* **2018**, *172*, 566−581.

(6) Ding, L.; Han, X.; Cao, L.; Chen, Y.; Ling, Z.; Han, J.; He, S.; Jiang, S. Characterization of Natural Fiber from Manau Rattan (Calamus Manan) as a Potential Reinforcement for Polymer-Based Composites. *J. Bioresour. Bioprod.* **2022**, *7* (3), 190−200.

(7) Sahu, S.; Sahu, S. B. B. P. J.; Nayak, S.; Mohapatra, J.; Khuntia, S. K.; Malla, C.; Samal, P.; Patra, S. K.; Swain, S. Characterization of Natural Fiber Extracted from Bauhinia Vahlii Bast Subjected to Different Surface Treatments: A Potential Reinforcement in Polymer Composite. *J. Nat. Fibers* **2023**, *20* (1), No. 2162185.

(8) Ramasamy, S.; Kandasamy, J.; Samrot, A. V.; Vijayashree, T. Study of Various Properties of Chemically Treated Lignocellulosic Cissus Quadrangularis Stem Fiber for Composite Reinforcement. *J. Nat. Fibers* **2023**, *20* (1), No. 2161689.

(9) Selvaraj, M.; S, A.; Mylsamy, B. Characterization of New Natural Fiber from the Stem of Tithonia Diversifolia Plant. *J. Nat. Fibers* **2023**, *20* (1), No. 2167144.

(10) Bhunia, A. K.; Mondal, D.; Parui, S. M.; Mondal, A. K. Characterization of a New Natural Novel Lignocellulose Fiber Resource from the Stem of Cyperus Platystylis R.Br. *Sci. Rep.* **2023**, *13* (1), 9699.

(11) Gelaw, B. B.; Tesfaye, T. Characterization of a New Fiber From Cyperus Dichrostachus A. Rich Plant. *Preprint* **2021**, DOI: 10.21203/rs.3.rs-1201679/v1.

(12) Rangappa, S. M.; Siengchin, S.; Parameswaranpillai, J.; Jawaid, M.; Ozbakkaloglu, T. Lignocellulosic Fiber Reinforced Composites: Progress, Performance, Properties, Applications, and Future Perspectives. *Polym. Compos.* **2022**, *43* (2), 645−691.

(13) Udhayakumar, A.; Mayandi, K.; Rajini, N.; Devi, R. K.; Muthukannan, M.; Murali, M. Extraction and Characterization of Novel Natural Fiber from Cryptostegia Grandiflora as a Potential Reinforcement in Biocomposites. *J. Nat. Fibers* **2023**, *20* (1), No. 2159607.

(14) Vinod, A.; Sanjay, M. R.; Suchart, S.; Jyotishkumar, P. Renewable and Sustainable Biobased Materials: An Assessment on Biofibers, Biofilms, Biopolymers and Biocomposites. *J. Clean. Prod.* **2020**, *258*, No. 120978.

(15) Ramesh, M.; Rajeshkumar, L.; Bhuvaneswari, V. Leaf Fibres as Reinforcements in Green Composites: A Review on Processing, Properties and Applications. *Emergent Mater.* **2022**, *5*, 833−857.

(16) Prayitno, P.; Abu Yazid, M. Z.; Seyajah, N.; Sudiro, S. A Review on the Potential Applications of the Ketapang Tree in Different Areas (Terminalia Catappa) BT - Design in Maritime Engineering; Ismail, A., Dahalan, W. M., Öchsner, A., Eds.; Springer International Publishing: Cham, 2022; pp 387−402.

(17) Paul, P. E. S.; Pitchipoo, P. Experimental Investigation on the Physicochemical Properties of Terminalia Catappa Fiber. *Biomass Convers. Biorefinery* **2024**, *14*, 10713−10721.

(18) Susilawati; Doyan, A.; Muliyadi, L. Mechanical Properties of Composite Board Based on Coconut Coir and Terminalia Catappa Fruit Fibers with Polyvinyl Acetate (PVAc) Matrix. *J. Phys. Conf. Ser.* **2021**, *1816* (1), 012013.

(19) Ramesh, M.; Deepa, C.; Niranjana, K.; Rajeshkumar, L.; Bhoopathi, R.; Balaji, D. Influence of Haritaki (Terminalia Chebula) Nano-Powder on Thermo-Mechanical, Water Absorption and Morphological Properties of Tindora (Coccinia Grandis) Tendrils Fiber Reinforced Epoxy Composites. *J. Nat. Fibers* **2022**, *19*, 6452.

(20) H. S. Souza Rosa, C.; Goncalves Mothe, M.; Vieira Marques, M. F.; Goncalves Mothe, C.; Neves Monteiro, S. Steam-Exploded Fibers of Almond Tree Leaves as Reinforcement of Novel Recycled Polypropylene Composites. *J. Mater. Res. Technol.* **2020**, *9* (5), 11791−11800.

(21) Vadwala, Y.; Kola, N. Natural Dyes Extracted from Waste Leaves of Terminalia catappa Locally Known As Tropical Almond and Its Application on Silk Fabrics Pretreated with Eco Friendly and Noneco-Friendly Mordants. *Int. J. Res. -Granthaalayah* **2017**, *5*, 125−137.

(22) Anand, A. V.; Divya, N.; Kotti, P. P. An Updated Review of Terminalia Catappa. *Pharmacogn. Rev.* **2015**, *9* (18), 93−98.

(23) Saravanakumar, K.; Srinivasan, M. Exploration of Mechanical, Physical & Chemical Properties of Terminalia Catappa. *Int. J. Res. Aero. Mech. Eng.* **2017**, *5* (11), 1−15.

(24) M. R., S.; Siengchin, S.; Parameswaranpillai, J.; Jawaid, M.; Pruncu, C. I.; Khan, A. A Comprehensive Review of Techniques for Natural Fibers as Reinforcement in Composites: Preparation, Processing and Characterization. *Carbohydr. Polym.* **2019**, *207*, 108−121.

(25) Ramesh, M.; Deepa, C.; Rajeshkumar, L.; Tamil Selvan, M.; Balaji, D. Influence of Fiber Surface Treatment on the Tribological

Properties of Calotropis Gigantea Plant Fiber Reinforced Polymer Composites. *Polym. Compos.* **2021**, *42*, 4308.

(26) Yao, Y.; Chen, S. The Effects of Fiber's Surface Roughness on the Mechanical Properties of Fiber-Reinforced Polymer Composites. *J. Compos. Mater.* **2013**, *47*, 2909−2923.

(27) Li, C.; Fei, J.; Zhang, T.; Zhao, S.; Qi, L. Relationship between Surface Characteristics and Properties of Fiber-Reinforced Resin-Based Composites. *Compos. Part B Eng.* **2023**, *249*, No. 110422.

(28) Kurpińska, M.; Pawelska-Mazur, M.; Gu, Y.; Kurpiński, F. The Impact of Natural Fibers' Characteristics on Mechanical Properties of the Cement Composites. *Sci. Rep.* **2022**, *12* (1), 20565.

(29) Dahlan, I.; Doyan, A.; Kosim. Tensile Test of Terminalia Catappa Fruit Fiber Composite Material. *IOSR Journal of Applied Physics* **2018**, *10* (3), 63−67.

(30) Duppala Ajay Kumar, D. A. K. A Study on Different Chemical Treatments for Natural Fiber Reinforced Composites. *Int. J. Mech. Prod. Eng. Res. Dev.* **2018**, *8*, 143−152.

(31) Kavitha, S. A.; Priya, R. K.; Arunachalam, K. P.; Avudaiappan, S.; Saavedra Flores, E. I.; Blanco, D. Experimental Investigation on Strengthening of Zea Mays Root Fibres for Biodegradable Composite Materials Using Potassium Permanganate Treatment. *Sci. Rep.* **2024**, *14* (1), 12754.

(32) Arulselvan, S.; Manikandan, D. A. S. P. Effects of Various Chemical Treatments on Physicochemical Properties of Cotton Shell Fibers − A Study; **2020**.

(33) Madival, A. S.; Maddasani, S.; Shetty, R.; Doreswamy, D. Influence of Chemical Treatments on the Physical and Mechanical Properties of Furcraea Foetida Fiber for Polymer Reinforcement Applications. *J. Nat. Fibers* **2023**, *20* (1), No. 2136816.

(34) Zhao, X.; Oyedeji, O.; Webb, E.; Wasti, S.; Bhagia, S.; Hinton, H.; Li, K.; Kim, K.; Wang, Y.; Zhu, H.; Vaidya, U.; Labbé, N.; Tekinalp, H.; Gallego, N. C.; Pu, Y.; Ragauskas, A. J.; Ozcan, S. Impact of Biomass Ash Content on Biocomposite Properties. *Compos. Part C Open Access* **2022**, *9*, No. 100319.

(35) Sheeba, K. R.; Priya, R. K.; Arunachalam, K. P.; Avudaiappan, S.; Maureira-Carsalade, N.; Roco-Videla, Á. Characterisation of Sodium Acetate Treatment on Acacia Pennata Natural Fibres. *Polymers.* **2023**, *15*, 1996.

(36) Amroune, S.; Bezazi, A.; Belaadi, A.; Zhu, C.; Scarpa, F.; Rahatekar, S.; Imad, A. Tensile Mechanical Properties and Surface Chemical Sensitivity of Technical Fibres from Date Palm Fruit Branches (Phoenix Dactylifera L.). *Composites Part A: Applied Science and Manufacturing* **2015**, *71*, 95−106.

(37) Subramanian, S. G.; Rajkumar, R.; Ramkumar, T. Characterization of Natural Cellulosic Fiber from Cereus Hildmannianus. *J. Nat. Fibers* **2021**, *18* (3), 343−354.

(38) Manimaran, P.; Pillai, G. P.; Vignesh, V.; Prithiviraj, M. Characterization of Natural Cellulosic Fibers from Nendran Banana Peduncle Plants. *Int. J. Biol. Macromol.* **2020**, *162*, 1807−1815.

(39) Kavitha, S. A.; Priya, R. K.; Arunachalam, K. P.; Avudaiappan, S.; Maureira-Carsalade, N.; Roco-Videla, Á. Investigation on Properties of Raw and Alkali Treated Novel Cellulosic Root Fibres of Zea Mays for Polymeric Composites. *Polymers.* **2023**, *15*, 1802.

(40) Tamanna, T. A.; Belal, S. A.; Shibly, M. A. H.; Khan, A. N. Characterization of a New Natural Fiber Extracted from Corypha Taliera Fruit. *Sci. Rep.* **2021**, *11* (1), 7622.

(41) Madhu, P.; Sanjay, M. R.; Senthamaraikannan, P.; Pradeep, S.; Siengchin, S.; Jawaid, M.; Kathiresan, M. Effect of Various Chemical Treatments of Prosopis Juliflora Fibers as Composite Reinforcement: Physicochemical, Thermal, Mechanical, and Morphological Properties. *J. Nat. Fibers* **2020**, *17* (6), 833−844.

(42) Ezeamaku, U. L.; Ezekannagha, C.; Eze, O. I.; Odimegwu, N.; Nwakaudu, A.; Okafor, A.; Ekuma, I.; Onukwuli, O. D. The Impact of Potassium Permanganate (KMnO) Treatment on the Tensile Strength of Pineapple Leaf Fiber Reinforced with Tapioca-Based Bio Resin. *Arab Gulf J. Sci. Res.* **2023**, *41* (3), 416−426.

(43) Sheeba, K. R. J.; Alagarasan, J. K.; Dharmaraja, J.; Kavitha, S. A.; Shobana, S.; Arvindnarayan, S.; Vadivel, M.; Lee, M.; Retnam, K. P. Physico−Chemical and Extraction Properties on Alkali−Treated Acacia Pennata Fiber. *Environ. Res.* **2023**, *233*, No. 116415.

(44) Rashid, M. M.; Samad, S.; Gafur, M.; Chowdhury, A. M. S. Study of Different Chemical Treatments for the Suitability of Banana (Musa Oranta) Fiber In Composite Materials. *Int. J. Sci. Eng. Res.* **2015**, *6*, 1870−1875.

(45) Huzaifah, M. R. M.; Sapuan, S. M.; Leman, Z.; Ishak, M. R. Comparative Study on Chemical Composition, Physical, Tensile, and Thermal Properties of Sugar Palm Fiber (Arenga Pinnata) Obtained from Different Geographical Locations. *BioResources* **2017**, *12*, 9366−9382.

(46) Madhu, P.; Sanjay, M. R.; Jawaid, M.; Siengchin, S.; Khan, A.; Pruncu, C. I. A New Study on Effect of Various Chemical Treatments on Agave Americana Fiber for Composite Reinforcement: Physico-Chemical, Thermal, Mechanical and Morphological Properties. *Polym. Test.* **2020**, *85*, No. 106437.

(47) Dhanalakshmi, S.; Ramadevi, P.; Basavaraju, B. A Study of the Effect of Chemical Treatments on Areca Fiber Reinforced Polypropylene Composite Properties. *SciEng. Compos Mater.* **2017**, *24* (4), 501−520.

(48) Abisha, M.; Priya, R. K.; Arunachalam, K. P.; Avudaiappan, S.; Saavedra Flores, E. I.; Parra, P. F. Biodegradable Green Composites: Effects of Potassium Permanganate (KMnO4) Treatment on Thermal, Mechanical, and Morphological Behavior of Butea Parviflora (BP) Fibers. *Polymers.* **2023**, *15*, 2197.

(49) Roodbar Shojaei, T.; Soltani, S.; Derakhshani, M. Chapter 6 - Synthesis, Properties, and Biomedical Applications of Inorganic Bionanomaterials. In *Micro and Nano Technologies*; Barhoum, A., Jeevanandam, J., Danquah, M. K. B. T.-F. B., Eds.; Elsevier, 2022; pp 139−174. DOI: 10.1016/B978-0-12-824147-9.00006-6.

(50) Rajkumar, R.; Manikandan, A.; Saravanakumar, S. Physicochemical Properties of Alkali Treated New Cellulosic Fiber from Cotton Shell. *Int. J. Polym. Anal. Char.* **2016**, *21*, 359.

(51) Nayak, S.; Mohanty, J. Influence of Chemical Treatment on Tensile Strength, Water Absorption, Surface Morphology, and Thermal Analysis of Areca Sheath Fibers. *J. Nat. Fibers* **2019**, *16*, 589.

(52) De Rosa, I. M.; Kenny, J. M.; Maniruzzaman, M.; Moniruzzaman, M.; Monti, M.; Puglia, D.; Santulli, C.; Sarasini, F. Effect of Chemical Treatments on the Mechanical and Thermal Behaviour of Okra (Abelmoschus Esculentus) Fibres. *Compos. Sci. Technol.* **2011**, *71* (2), 246−254.

(53) Oza, S.; Ning, H.; Ferguson, I.; Lu, N. Effect of Surface Treatment on Thermal Stability of the Hemp-PLA Composites: Correlation of Activation Energy with Thermal Degradation. *Compos. Part B Eng.* **2014**, *67*, 227−232.

(54) Jaiswal, D.; Devnani, G.L.; Rajeshkumar, G.; Sanjay, M.R.; Siengchin, S. Review on Extraction, Characterization, Surface Treatment and Thermal Degradation Analysis of New Cellulosic Fibers as Sustainable Reinforcement in Polymer Composites. *Curr. Res. Green Sustain. Chem.* **2022**, *5*, No. 100271.

(55) Moliner, C.; Badia, J. D.; Bosio, B.; Arato, E.; Kittikorn, T.; Strömberg, E.; Teruel-Juanes, R.; Ek, M.; Karlsson, S.; Ribes-Greus, A. Thermal and Thermo-Oxidative Stability and Kinetics of Decomposition of PHBV/Sisal Composites. *Chem. Eng. Commun.* **2018**, *205* (2), 226−237.

(56) Bharath, K. N.; Madhu, P.; Gowda, T. G. Y.; Sanjay, M. R.; Kushvaha, V.; Siengchin, S. Alkaline Effect on Characterization of Discarded Waste of Moringa Oleifera Fiber as a Potential Eco-Friendly Reinforcement for Biocomposites. *J. Polym. Environ.* **2020**, *28* (11), 2823−2836.

(57) Sheeba, K. R. J.; Priya, R. K.; Arunachalam, K. P.; Shobana, S.; Avudaiappan, S.; Flores, E. S. Examining the Physico-Chemical, Structural and Thermo-Mechanical Properties of Naturally Occurring Acacia Pennata Fibres Treated with KMnO4. *Sci. Rep.* **2023**, *13* (1), 20643.

(58) Reddy, N.; Yang, Y. Extraction and Characterization of Natural Cellulose Fibers from Common Milkweed Stems. *Polym. Eng. Sci.* **2009**, *49* (11), 2212−2217.

(59) Jones, D.; Ormondroyd, G. O.; Curling, S. F.; Popescu, C.-M.; Popescu, M.-C. *Chemical Compositions of Natural Fibres*; Fan, M., Fu, F. B. T.-A. H. S. N. F. C. C., Eds.; Woodhead Publishing, 2017; pp 23−58. DOI: 10.1016/B978-0-08-100411-1.00002-9.

(60) Ramesh, M.; Palanikumar, K.; Reddy, K. H. Plant Fibre Based Bio-Composites: Sustainable and Renewable Green Materials. *Renew. Sustain. Energy Rev.* **2017**, *79*, 558−584.

(61) Rao, H. J.; Singh, S.; Janaki Ramulu, P. Characterization of a Careya Arborea Bast Fiber as Potential Reinforcement for Light Weight Polymer Biodegradable Composites. *J. Nat. Fibers* **2023**, *20* (1), 71−87.

(62) Madhu, P.; Sanjay, M. R.; Senthamaraikannan, P.; Pradeep, S.; Saravanakumar, S. S.; Yogesha, B. A Review on Synthesis and Characterization of Commercially Available Natural Fibers: Part II. *J. Nat. Fibers* **2019**, *16* (1), 25−36.

(63) Avudaiappan, S.; Cuello Moreno, P. I.; Montoya R, L. F.; Chávez-Delgado, M.; Arunachalam, K. P.; Guindos, P.; Marzialetti B, T.; Fernando Parra, P.; Saavedra Flores, E. I.; Flores Arrey, J. I. Experimental Investigation on the Physical, Microstructural, and Mechanical Properties of Hemp Limecrete. *Sci. Rep.* **2023**, *13* (1), 22650.

(64) Rekha, M. S.; Sumathy, S. R.; Arunachalam, K. P.; Avudaiappan, S.; Abbas, M.; Fernande, D. B. Effects of Alkaline Concentration on Workability and Strength Properties of Ambient Cured Green Geopolymer Concrete. *Asian J. Civ. Eng.* **2024**, *25*, 4893.

(65) Syafri, E.; Jamaluddin; Harmailis; Umar, S.; Mahardika, M.; Amelia, D.; Mayerni, R.; Mavinkere Rangappa, S.; Siengchin, S.; Sobahi, T. R.; Khan, A.; Asiri, A. M. Isolation and Characterization of New Cellulosic Microfibers from Pandan Duri (Pandanus Tectorius) for Sustainable Environment. *J. Nat. Fibers* **2022**, *19* (16), 12924−12934.

(66) Bar, G.; Chaudhary, K. Characterization of Textile Grade Novel Bauhinia Vahlii Fiber. *J. Nat. Fibers* **2023**, *20* (1), No. 2143464.

(67) Khenblouche, A.; Bechki, D.; Gouamid, M.; Charradi, K.; Segni, L.; Hadjadj, M.; Boughali, S. Extraction and Characterization of Cellulose Microfibers from Retama Raetam Stems. *Polímeros Ciencia Tecnologia* **2019**, *29*, e2019011.

(68) Ramesh, M.; Rajeshkumar, L.; Deepa, C.; Tamil Selvan, M.; Kushvaha, V.; Asrofi, M. Impact of Silane Treatment on Characterization of Ipomoea Staphylina Plant Fiber Reinforced Epoxy Composites. *J. Nat. Fibers* **2022**, *19* (13), 5888−5899.

(69) Jayanudin, J.; Kurniawan, T.; Kustiningsih, I. Phenolic Analysis and Characterization of Palm Sugar (Arenga Pinnata) Produced by The Spray Dryer. *Orient. J. Chem.* **2019**, *35*, 150−156.

(70) Kavitha, D. S. A Study on the Bioefficacy of Caryota Urens L. *World J. Pharm. Res.* **2017**, 1381−1398.

(71) C, I. P.; R, S. Characterization of a New Natural Cellulosic Fiber Extracted from Derris Scandens Stem. *Int. J. Biol. Macromol.* **2020**, *165*, 2303−2313.

(72) Manimaran, P.; Saravanan, S. P.; Sanjay, M. R.; Siengchin, S.; Jawaid, M.; Khan, A. Characterization of New Cellulosic Fiber: Dracaena Reflexa as a Reinforcement for Polymer Composite Structures. *J. Mater. Res. Technol.* **2019**, *8* (2), 1952−1963.

(73) Chikkol Venkateshappa, S. Natural Areca Fiber: Surface Modification and Spectral Studies. *J. Adv. Chem.* **2014**, *10*, 3263−3273.

(74) Siddiqui, M.; Prasad, N.; Tewari, J. Physicochemical Properties and Toxicity Test of Prosopis Juliflora (Sw.) DC. and Balanites Aegyptiaca Del Gum Exudates from Rajasthan. *Indian J. Exp. Biol.* **2017**, *55*, 782−788.

(75) A. N., B.; K. J., N. Characterization of Alkali Treated and Untreated New Cellulosic Fiber from Saharan Aloe Vera Cactus Leaves. *Carbohydr. Polym.* **2017**, *174*, 200−208.

(76) Alkaron, W. A.; Hamad, S. F.; Sabri, M. M. Studying the Fabrication and Characterization of Polymer Composites Reinforced with Waste Eggshell Powder. *Adv. Polym. Technol.* **2023**, *2023* (1), No. 7640478.

(77) Wen, G.; Rippon, J. A.; Brady, P. R.; Wang, X. G.; Liu, X.; Cookson, P. G. The Characterization and Chemical Reactivity of Powdered Wool. *Powder Technol.* **2009**, *193* (2), 200−207.

(78) Sabarinathan, P.; Rajkumar, K.; Annamalai, V. E.; Vishal, K. Characterization on Chemical and Mechanical Properties of Silane Treated Fish Tail Palm Fibres. *Int. J. Biol. Macromol.* **2020**, *163*, 2457−2464.

(79) Rajkumar, S.; Tjong, J.; Nayak, S. K.; Sain, M. Wetting Behavior of Soy-Based Resin and Unsaturated Polyester on Surface-Modified Sisal Fiber Mat. *J. Reinf. Plast. Compos.* **2015**, *34* (10), 807−818.

(80) Sgriccia, N.; Hawley, M. C.; Misra, M. Characterization of Natural Fiber Surfaces and Natural Fiber Composites. *Compos. Part A Appl. Sci. Manuf.* **2008**, *39* (10), 1632−1637.

(81) Rajkhowa, R.; Wang, X. 16 - Properties and Applications of Ultrafine Powders Produced from Natural Fibres. In *The Textile Institute Book Series*, second ed.; Kozłowski, R. M., Mackiewicz-Talarczyk, M. B. T.-H. N. F., Eds.; Woodhead Publishing, 2020; pp 531−556. DOI: 10.1016/B978-0-12-818782-1.00016-X.

(82) Chenrayan, V.; Shahapurkar, K.; Kiran, M. C.; Ngarajan, B.; Arunachalam, K. P.; Weiss, A. D.; Fouad, Y.; Almehmadi, F. A.; Soudagar, M. E. M. Leverage of Aluminium Oxynitride on the Impact Resistance of Kevlar-Impregnated Epoxy Composites: Experimental and Numerical Evaluation under Low-Velocity Impact. *Polym. Compos.* **2024**, *45*, 17061.

(83) Gagic, T.; Perva-Uzunalic, A.; Knez, Z.; Skerget, M. Hydrothermal Degradation of Cellulose at Temperature from 200 to 300 °C. *Ind. Eng. Chem. Res.* **2018**, *57*, 6576.

(84) Waters, C. L.; Janupala, R. R.; Mallinson, R. G.; Lobban, L. L. Staged Thermal Fractionation for Segregation of Lignin and Cellulose Pyrolysis Products: An Experimental Study of Residence Time and Temperature Effects. *J. Anal. Appl. Pyrolysis* **2017**, *126*, 380−389.

(85) Díez, D.; Urueña, A.; Piñero, R.; Barrio, A.; Tamminen, T. Determination of Hemicellulose, Cellulose, and Lignin Content in Different Types of Biomasses by Thermogravimetric Analysis and Pseudocomponent Kinetic Model (TGA-PKM Method). *Processes.* **2020**, *8*, 1048.

(86) Alwani, M. S.; Khalil, H. P. S. A.; Sulaiman, O.; Islam, M. N.; Dungani, R. An Approach to Using Agricultural Waste Fibres in Biocomposites Application: Thermogravimetric Analysis and Activation Energy Study. *BioResources* **2013**, *9*, DOI: 10.15376/biores.9.1.218-230.

(87) Belaadi, A.; Bezazi, A.; Bourchak, M.; Scarpa, F.; Zhu, C. Thermochemical and Statistical Mechanical Properties of Natural Sisal Fibres. *Compos. Part B Eng.* **2014**, *67*, 481−489.

(88) Been Seok, Y.; Wee, F.; Muhamad, M. Effect of Biomass Waste Filler on the Dielectric Properties of Polymer Composites. *EnvironmentAsia* **2016**, *11*, 134−139.

(89) Tesfaye, T.; Sithole, B.; Ramjugernath, D.; Chunilall, V. Valorisation of Chicken Feathers: Characterisation of Chemical Properties. *Waste Manag.* **2017**, *68*, 626−635.