Journal of Computers, Mechanical and Management
©Global Academy Digital Library (GADL)
Journal homepage: www.jcmm.co.in

Volume 2 Issue 2

Article Number: 23057

A Mini Review of Natural Cellulosic Fibers: Extraction, Treatment and Characterization Methods

Yashaarth Kaushik[1], Nilakshman Sooriyaperakasam[2], Udit Rathee[3], and Nithesh Naik*[1]

[1]Department of Mechanical and Industrial Engineering, Manipal Institute of Technology, Manipal Academy of Higher Education, Manipal, Karnataka, India 576104
[2]Department of Mechanical and Industrial Engineering, University of Moratuwa, Moratuwa, Sri Lanka 10400
[3]International Center for Automotive Research, Clemson University, Clemson, SC, USA 29634

Abstract

Natural cellulosic fibers have garnered significant attention in recent years due to their potential as eco-friendly and sustainable alternatives to synthetic materials in various industries, such as composite reinforcement, textiles, and packaging. The successful utilization of these fibers depends on understanding their properties, which can be achieved through comprehensive characterization methods. This mini-review discusses the extraction, treatment, and characterization of natural cellulosic fibers, focusing on the latest advancements in these areas. The extraction methods of natural cellulosic fibers, including chemical, physical, and biological processes, are discussed. The choice of extraction method impacts the fiber's morphology, crystallinity, and purity, which ultimately affects its performance in different applications. Surface modification methods, including chemical treatments like treatment, acetylation, silane treatment, grafting, bleaching, and physical treatments such as corona treatment, plasma treatment, and UV/ozone treatment, are also explored. These treatments can enhance the compatibility and performance of natural cellulosic fibers in various applications. Characterization techniques for natural cellulosic fibers are reviewed, including mechanical properties testing (tensile strength, flexural strength, and impact strength), morphological analysis (scanning electron microscopy (SEM) and transmission electron microscopy (TEM)), and advanced techniques such as atomic force microscopy (AFM), confocal Raman spectroscopy, and X-ray photoelectron spectroscopy (XPS). Furthermore, the application of the Weibull distribution for analyzing the tensile strength of natural cellulosic fibers is discussed, providing valuable information on the fiber's strength distribution and reliability. This review highlights the importance of comprehensive characterization methods in understanding the properties and potential applications of natural cellulosic fibers. The continued development of these techniques will be crucial for the advancement and commercialization of these sustainable materials as eco-friendly alternatives to traditional materials in a wide range of industries.

Keywords: Natural Fibers; Extraction Methods; Surface Modification; Characterization Techniques; Sustainable Materials

1 Introduction

The demand for sustainable, environmentally friendly materials has driven the exploration of natural fibers as potential alternatives to synthetic fibers in composite materials. Due to their biodegradability, abundance, low density, recyclability, sustainability, non-toxicity, non-corrosiveness, eco-friendliness, and low carbon emissions, natural fibers are identified as viable replacements for petroleum-based fiber-reinforced composites [1–4]. Natural fibers are obtained from various parts of plants, such as stems, leaves, bark, roots, fruits, and seeds. Researchers have examined different plant fibers like banana, hemp, sisal, coir, and bamboo for reinforcement in polymer composites. Recent studies have explored the suitability of fibers such as Sansevieria cylindrica, Sansevieria ehrenbergii, Prosopis juliflora, Indian mallow, Saharan Aloe vera, Furcraea foetida, Thespesia populnea, aerial roots of banyan trees, red banana peduncle, Calotropis gigantea, and Leucas Aspera [5–19].

*Corresponding author: nithesh.naik@manipal.edu
Received: 16 March 2023; **Revised:** 23 April 2023; **Accepted:** 28 April 2023

DOI: 10.57159/gadl.jcmm.2.2.23057.

The final properties of polymer composites depend on the resin's nature, fiber alignment, and bonding between the fiber and matrix [20]. Researchers have extracted fibers from various parts of the banana plant, including the stem, while banana peduncles are often discarded as waste. To utilize this waste more beneficially, recent studies have focused on extracting, treating, and characterizing natural fibers from the banana peduncle, particularly from the Nendran Banana Peduncle (NBP). The properties of natural fibers depend on their source, harvesting method, and processing techniques. Therefore, it is essential to characterize natural fibers and understand their properties to enhance their performance as a reinforcement in composite materials. Natural fibers have gained increasing interest as a reinforcement in composite materials due to their unique properties, such as biodegradability, low cost, low density, high strength, and renewability [1–3, 5]. Using natural fibers in composite materials can reduce the environmental impact by replacing synthetic fibers, which are derived from non-renewable resources and have a high carbon footprint. Various fibers, including those derived from plants like bamboo, hemp, sisal, coir, and jute, and those derived from animal sources such as wool and silk, have been investigated for potential use in composite materials. The mechanical properties of natural fibers vary depending on their source and processing methods [21, 8–10]. Despite the potential advantages of using natural fibers in composite materials, challenges exist. One main challenge is achieving good fiber-matrix adhesion, essential for transferring stresses between the fiber and matrix and ensuring the mechanical integrity of the composite material. Natural fibers also have inherent variability in their properties, which can make it difficult to achieve consistent properties in composite materials. Utilizing natural cellulosic fibers offers several advantages over synthetic fibers, including renewability, biodegradability, and low environmental impact, making them promising candidates for sustainable and eco-friendly composite materials. Recent advances in extraction, treatment, and characterization methods have enhanced the properties of natural fibers, leading to the production of eco-friendly composite materials. The appropriate utilization of natural cellulosic fibers can play a significant role in sustainable development by reducing the dependence on petroleum-based products and minimizing environmental pollution. THe various techniques employed in the extraction, treatment, and characterization methods for natural cellulosic fibers have led to the production of eco-friendly composite materials with improved properties. Researchers have explored various extraction methods, including enzymatic extraction, microwave-assisted extraction, and ultrasonic-assisted extraction. Similarly, recent advances in treatment methods, such as plasma treatment, nanocellulose modification, surface modification, and functionalization, have enhanced fiber-matrix adhesion and compatibility between fibers and the polymer matrix. Additionally, recent advances in characterization methods, including in-situ characterization, multiscale characterization, and computational modeling, have improved our understanding of the physical, chemical, and mechanical properties of natural fibers [22, 23]. Physical properties such as density, moisture content, and fiber diameter affect the composite material's overall properties. Chemical properties, including chemical composition, functional groups, and degree of polymerization, determine fiber-matrix adhesion and compatibility between fibers and the polymer matrix. Furthermore, mechanical properties such as tensile strength, stiffness, and elongation at break affect the composite material's strength and durability [22–25]. Table 1 shows the physical and mechanical properties of different natural fibers [26].

Table 1: Physical and Mechanical properties of natural fibers [26] .

Name of the fiber	**Diameter (μm)**	**Length (mm)**	**Density (kg/mm^3)**	**Moisture gain (%)**	**Tensile strength (MPa)**	**Young's modulus (GPa)**	**Failure strain (%)**
Abca	18.2	4.9	1500	14	12	41	3.4
Alfa	-	-	890	-	350	22	5.8
Bagasse	20	1.7	900	-	290	17	-
Banana	-	2.9	1325	-	721.5	29	2
Bamboo	25	2	1500	-	575	27	-
Coir	17.5	1.25	1250	13	140.5	6	27.5
Cotton	14.5	42	1550	8.59	500	8	7
Curaua	-	-	1400	-	825	9	7.5
Flax	20	31.75	1450	12	700	60	2.3
Hemp	19.9	11.2	1200	-	530	45	3
Isora	-	-	1200	1.2	550	-	5.5
Jute	18.4	2.55	1400	17	325	37.5	2.5
Kapok	25	20	384	10.9	93.3	4	1.2
Kenaf	19.8	2.35	1300	17	743	41	-
Piassava	-	-	1400	-	138.5	2.83	5
Pineapple	50	-	1540	-	1020	71	0.8
Ramie	31.55	160	1550	8.5	925	23	3.7
Sisal	21	2.5	1400	14	460	15.5	-

Characterizing natural fibers is crucial for understanding their properties and potential as reinforcement in composite materials. Various techniques have been developed for the characterization of natural fibers, including microscopy, spectroscopy, thermal analysis, and mechanical testing. Microscopy techniques such as scanning electron microscopy (SEM) and optical microscopy are commonly used to analyze fibers' morphological features, including fiber diameter, shape, and surface morphology. Spectroscopy techniques, such as Fourier transform infrared spectroscopy (FTIR) and nuclear magnetic resonance (NMR) spectroscopy, are used to identify fibers' functional groups and determine their chemical composition.

Thermal analysis techniques, such as thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), are employed to analyze fibers' thermal stability and degradation behavior. Mechanical testing techniques, including tensile testing and flexural testing, are used to determine fibers' mechanical properties [22, 24, 25, 27, 28]. Understanding natural fibers' properties and characteristics is essential for enhancing their performance as reinforcement in composite materials. Further research and development in this field will provide opportunities for creating sustainable and eco-friendly materials with improved properties. However, challenges associated with their use, such as achieving good fiber-matrix adhesion and dealing with inherent variability in fiber properties, must be addressed to fully realize their potential in composite materials. Thus, this mini-review article provides an overview of the techniques used in extraction, treatment, and characterization methods, while discussing the physical, chemical, and mechanical properties of various natural fibers.

2 Extraction Methods of Natural Cellulosic Fibers

The extraction of natural cellulosic fibers from plant material is an important process for their characterization and utilization in various applications. Recent research has explored different extraction processes for natural cellulosic fibers. The extraction process for natural cellulosic fibers depends on the plant source and the desired fiber properties [29]. Here are some commonly used methods:

2.1 Chemical extraction

Chemical extraction involves the use of chemicals such as alkalis or acids to break down the plant material and isolate the fibers. The fibers are then washed and treated with bleaching agents to remove impurities. Chemical extraction is a common method for isolating cellulose fibers from various plant sources, such as cotton, flax, and hemp [30, 31].

2.2 Physical extraction

Physical extraction involves the mechanical separation of cellulose fibers from other plant components. This can be achieved through various methods such as retting, decortication, and steam explosion. Retting is a process in which plant material is soaked in water to allow microbial degradation of non-cellulosic components, followed by mechanical separation of the cellulose fibers. Decortication involves the removal of the outer layer of the plant material to expose the cellulose fibers, which are then mechanically separated. Steam explosion involves the use of high-pressure steam to break down the lignin and hemicellulose components of the plant material, leaving behind the cellulose fibers [32–35].

2.3 Biological extraction

Biological extraction involves the use of microorganisms to break down the non-cellulosic components of the plant material. This process is known as bio-pulping and has the potential to be a more environmentally friendly alternative to chemical pulping [36–39]. The microorganisms used for bio-pulping include fungi and bacteria. Fungi such as Trichoderma reesei and Phanerochaete chrysosporium are commonly used for bio-pulping. These microorganisms produce enzymes that break down the lignin and hemicellulose components of the plant material, leaving behind the cellulose fibers [40, 41].

3 Chemical and Physical Property Determination

3.1 Chemical properties

Various chemical methods can be used to analyze the chemical composition of natural cellulosic fibers and determine the amounts of lignin, hemicellulose, and cellulose. These include:

- **Acid-base titration:** This method involves the use of acid and base solutions to determine the amount of hemicellulose, lignin, and cellulose in the fibers. The fibers are first treated with a strong acid to remove hemicellulose, followed by treatment with a strong base to remove lignin. The remaining residue is cellulose, which is weighed to determine its amount [42].
- **Spectrophotometry:** This method involves the use of a spectrophotometer to determine the amount of lignin in the fibers. The fibers are treated with a reagent that reacts with lignin to form a colored compound. The color intensity is then measured using a spectrophotometer, and the lignin content is calculated from a standard calibration curve [43, 44].
- **Gravimetric analysis:** This method involves the determination of the weight of the fibers and the weight of the ash remaining after burning the fibers at high temperatures. The weight of the ash represents the amount of non-cellulosic components in the fibers, such as hemicellulose, lignin, and minerals [45].
- **Elemental analysis:** This method involves the determination of the elemental composition of the fibers using techniques such as X-ray fluorescence (XRF) or inductively coupled plasma-optical emission spectroscopy (ICP-OES). Elemental analysis can provide information on the number of minerals and trace elements present in the fibers [46–48].

- **High-performance liquid chromatography (HPLC):** This powerful analytical technique has been used to study the chemical composition of natural cellulosic fibers. HPLC separates and identifies individual chemical components present in a sample based on their chemical and physical properties. One primary application of HPLC in natural cellulosic fibers is to analyze the monosaccharide composition of the fibers. HPLC can be used to analyze the individual glucose units in the fiber and to quantify the percentage of different monosaccharides present. This information can be used to understand the chemical composition and properties of the fibers [49–52].
- **Wax content determination:** The wax content of the fiber can be evaluated using the Conrad method [53], which involves the following steps:
  - **Extraction of wax:** A known weight of the fiber is boiled in a mixture of equal parts of ethanol and ether for a specific duration. The solvent mixture dissolves the wax from the fiber and extracts it into the solvent.
  - **Filtration:** The solvent extract is then filtered through a filter paper to remove any insoluble impurities.
  - Evaporation: The filtered solvent extract is then evaporated in a pre-weighed dish under a vacuum to remove the solvent and leave behind the extracted wax.
  - **Weighing:** The dish with the extracted wax is then re-weighed to determine the weight of the extracted wax. The difference between the weight of the dish with extracted wax and the weight of the dish before extraction gives the weight of the extracted wax.
  - **Calculation of wax content:** The wax content of the fiber is calculated by dividing the weight of the extracted wax by the weight of the original fiber sample and multiplying the result by 100. This gives the percentage of wax in the fiber.
- **C (CP-MAS) nuclear magnetic resonance (NMR) spectroscopy analysis**: Solid-state nuclear magnetic resonance (NMR) spectroscopy is a powerful technique for studying the chemical structure and properties of natural cellulosic fibers. It can be used to analyze the degree of polymerization of cellulose chains, which is the number of repeating units in a polymer chain and can affect the physical and mechanical properties of the fibers. NMR spectroscopy can provide information about the average degree of polymerization of cellulose in the fibers, which can be used to estimate their properties. To perform solid-state NMR spectroscopy on the fibers, a magic angle sample spinner (MAS) is used, along with a 10 KHz rate and a frequency of 75.46 MHz, at room temperature. Here's a general procedure for analyzing natural fibers using NMR spectroscopy [54–57]:
  - **Sample preparation:** The natural fiber sample is first washed with a suitable solvent to remove any impurities and then dried and ground into a fine powder.
  - **Dissolution:** The fiber sample is then dissolved in a suitable solvent, such as dimethyl sulfoxide (DMSO) or deuterated dimethyl sulfoxide (DMSO-d6), to make a concentrated solution.
  - NMR spectrometer setup: The NMR spectrometer is calibrated and set up to analyze the sample. The sample is placed in an NMR tube and inserted into the spectrometer.
  - **Spectral acquisition:** The NMR spectrometer is used to acquire the spectrum of the fiber sample. The spectrum is recorded by varying the magnetic field strength and radio frequency and measuring the energy absorbed and emitted by the sample.
  - Data analysis: The NMR spectrum is analyzed to determine the chemical shifts and peak intensities of the various components of the sample. The chemical shifts can provide information about the molecular structure of the fiber, including the types of chemical bonds present and their spatial arrangements.
  - **Interpretation of results:** The NMR data is interpreted to gain insights into the molecular structure and properties of the natural fiber. For example, the degree of crystallinity, molecular orientation, and chemical modifications can be determined using NMR spectroscopy.

  Using NMR spectroscopy, researchers can gain a better understanding of the chemical structure and properties of natural cellulosic fibers, which can help in developing new applications and improving existing ones.

3.2 Physical properties

3.2.1 Fiber Diameter Measurement

The average diameter of the fiber is measured generally using an optical microscope [58]. The measurement is made for a minimum of 25 fiber samples. Measurements should be made randomly along the fiber at several locations. The Weibull distribution statistical analysis is mostly used to examine the likelihood of the fiber average diameter. [59–61].

3.2.2 Density Measurement

The density of the fiber is generally measured using a pycnometer [62, 63]. For the first five days, natural cellulosic fibers should be placed in silica-packed desiccators to remove the moisture from the fibers. Following that, they should be chopped into pieces no larger than 5 mm and put in the machine. Before starting the experiment, the fibers are to be immersed in a toluene-filled container for roughly 2 hours to remove any microbubbles from the fibers. The density of fibers (ρ) was obtained through the Eqs [1] and [2]:

$$\rho = \frac{m}{V} \tag{1}$$

where m is the mass of the fiber and V is the volume of the pycnometer.

$$\rho_{BF} = \frac{m_2 - m_1}{(m_3 - m_1) - (m_4 - m_2)}\rho_T \tag{2}$$

4 Surface Modification Methods

The natural cellulosic fibers extracted from plant material often require treatment to modify their surface properties and enhance their compatibility with various matrices for improved performance in different applications. The treatment could be chemical or physical.

4.1 Chemical treatment

Recent research has explored various chemical treatment processes for natural cellulosic fibers. Chemical treatments can modify the surface properties of natural cellulosic fibers to improve their performance in various applications [29]. Here are some commonly used chemical treatment processes:

4.1.1 Alkali treatment

This process involves treating the fibers with alkalis, such as sodium hydroxide or potassium hydroxide, to remove impurities and increase the fiber's surface area. The treatment also introduces hydroxyl groups on the fiber surface, which can improve the fiber's hydrophilicity and reactivity. Alkali chemical treatment is a common method used to modify the properties of natural cellulosic fibers. The treatment involves soaking the fibers in a solution of strong alkalies, such as sodium hydroxide (NaOH), for a specific period. The alkali breaks down the hemicellulose and lignin components in the fiber, leaving behind a more pure form of cellulose. This process is known as mercerization. The alkali treatment can result in various modifications to the properties of the natural cellulosic fibers. The treatment can lead to an increase in the fiber's tensile strength, elongation at break, and stiffness. The treatment can also result in changes in the fiber's surface morphology, as well as its chemical and physical properties. The extent of the modification can be controlled by adjusting the concentration and duration of the alkali treatment. Higher concentrations of alkali and longer treatment times result in more significant modifications to the fiber's properties [64, 65].

4.1.2 Acetylation

Acetylation chemical treatment is a widely used method for modifying the properties of natural cellulosic fibers. The treatment involves the reaction of cellulose with an acetylating agent, such as acetic anhydride or acetic acid, in the presence of a catalyst. This results in the substitution of some of the hydroxyl groups on the cellulose chain with acetyl groups. The acetylation treatment can result in various modifications to the properties of natural cellulosic fibers. The treatment can lead to an increase in the fiber's water resistance, dimensional stability, and thermal stability. The treatment can also result in changes in the fiber's surface morphology, as well as its chemical and physical properties. The extent of the modification can be controlled by adjusting the concentration and duration of the acetylation treatment. Higher concentrations of acetylating agents and longer treatment times result in more significant modifications to the fiber's properties [66, 66].

4.1.3 Silane treatment

Silane treatment is a method used to modify the surface properties of natural cellulosic fibers. The treatment involves the reaction of the fiber with a silane coupling agent, such as 3-glycidoxypropyltrimethoxysilane (GPTMS), in a solvent, followed by drying and curing. The silane treatment can result in various modifications to the properties of natural cellulosic fibers. The treatment can lead to an increase in the fiber's hydrophobicity, as well as changes in its surface morphology and chemical properties. The coupling agent chemically bonds to the hydroxyl groups on the fiber surface, resulting in the formation of a siloxane layer on the fiber surface. The extent of the modification can be controlled by adjusting the concentration and duration of the silane treatment. Higher concentrations of coupling agents and longer treatment times result in more significant modifications to the fiber's properties [67, 68].

4.1.4 Grafting

Grafting treatment is a process used to modify the surface properties of natural cellulosic fibers by introducing polymer chains onto the fiber surface. The process involves the chemical bonding of monomers or polymers onto the fiber surface via covalent bonds, resulting in the formation of a polymer layer on the fiber surface. The grafting process can be carried out using various techniques, such as radiation-induced grafting, chemical grafting, and enzymatic grafting. The choice of technique depends on the specific properties required for the fibers and the intended application. The grafting process can lead to various modifications in the properties of natural cellulosic fibers, such as increased hydrophobicity, improved mechanical properties, and enhanced thermal stability. The properties of the grafting layer can be controlled by adjusting the type and concentration of monomers or polymers used, as well as the reaction conditions [69–71].

4.1.5 Bleaching

Bleaching is a common chemical treatment used to modify the properties of natural cellulosic fibers, such as cotton, bamboo, and hemp [72]. The process involves the removal of impurities, such as lignin and hemicellulose, and the brightening of the fiber's color [73, 74]. Bleaching treatment is carried out using various chemical agents, such as hydrogen peroxide, sodium hypochlorite, and chlorine dioxide [75]. The choice of bleaching agent depends on the specific requirements of the fiber and the intended application. The bleaching process can lead to various modifications in the properties of natural cellulosic fibers, such as an increase in brightness, improved dyeability, and enhanced mechanical properties. However, bleaching can also weaken the fibers if not carried out properly. The extent of the modification depends on various factors, such as the type and concentration of the bleaching agent, the duration of the treatment, and the temperature and pH of the treatment [76]. However, there are concerns about the environmental impact of the chemicals used in the process, and efforts are being made to develop more sustainable alternatives [77]. One alternative to traditional bleaching methods is the use of eco-friendly bleaching agents, which have a lower environmental impact. Another approach is the use of enzymatic bleaching, which utilizes enzymes to remove impurities from the fibers, without the need for harsh chemicals. Enzymatic bleaching has shown promising results in terms of improving the properties of natural cellulosic fibers while reducing the environmental impact of the process [78–81]. Overall, chemical treatment plays a critical role in modifying the properties of natural cellulosic fibers, making them more suitable for various applications. However, it is important to consider the environmental impact of the chemical processes used, and efforts should be made to develop more sustainable and eco-friendly methods.

4.2 Physical treatment

Physical treatments can also be used to modify the surface of natural cellulosic fibers to improve their compatibility with other materials or enhance their properties. These methods often provide an environmentally friendly alternative to chemical treatments [82]. Some common physical treatment methods include corona treatment, plasma treatment, and UV/ozone treatment [32].

4.2.1 Corona treatment

Corona treatment is an electrical discharge process that involves applying high voltage to an electrode placed near the surface of the fibers. This results in the formation of a corona discharge, which generates reactive species such as ozone and other oxygen-containing compounds. These reactive species modify the surface of the fibers by introducing polar groups, thus increasing their surface energy and enhancing their adhesion to polymer matrices. Corona treatment is a fast and effective method for improving the interfacial bonding between natural fibers and various polymer matrices [32, 83, 84].

4.2.2 Plasma treatment

Plasma treatment involves the use of low-pressure, non-thermal plasma to modify the surface of natural cellulosic fibers. The plasma is generated by applying an electric field to a gas (such as oxygen, nitrogen, or argon) at low pressure, which results in the formation of reactive species. These reactive species interact with the surface of the fibers, leading to the introduction of functional groups, surface etching, or crosslinking. Plasma treatment can significantly improve the surface properties of natural fibers, including wettability, adhesion, and chemical resistance. This method is considered environmentally friendly, as it does not require the use of hazardous chemicals [85–88].

4.2.3 UV/Ozone treatment

UV/ozone treatment is a photochemical process that utilizes ultraviolet (UV) radiation in the presence of ozone to modify the surface of natural cellulosic fibers [89, 90]. The UV radiation generates highly reactive ozone molecules, which react with the fiber surface, introducing polar groups such as hydroxyl, carbonyl, and carboxyl groups. These functional groups increase the surface energy of the fibers and improve their compatibility with polymer matrices. UV/ozone treatment is an environmentally friendly method that can be easily implemented for large-scale production [89, 91–94].

5 Characterization Techniques

5.1 Mechanical properties

Mechanical properties are crucial factors in determining the performance of natural cellulosic fibers in various applications, such as reinforcing composites, textiles, and packaging materials. These properties include tensile strength, flexural strength, and impact strength, which can be evaluated using standardized testing techniques [95–97].

5.1.1 Tensile strength testing

Tensile strength is a measure of a material's ability to withstand tension or pulling forces. Tensile strength testing involves applying a uniaxial tensile force to a fiber or a fiber bundle until it breaks. This test is typically performed using a universal testing machine (UTM) equipped with suitable grips and a load cell to measure the force applied. The tensile strength is calculated by dividing the maximum load by the initial cross-sectional area of the fiber or bundle. The results provide insights into the fiber's potential to reinforce composite materials and its performance in textile applications [98–101].

5.1.2 Flexural strength testing

Flexural strength, also known as bending strength, is an indicator of a material's ability to resist deformation under bending loads. Flexural strength testing is performed using a three-point or four-point bending test setup, in which a fiber-reinforced composite specimen is subjected to bending by applying a load at one or two points while supported at two or three points, respectively. The flexural strength is calculated by dividing the maximum bending load by the initial cross-sectional area of the specimen. This test is particularly useful for evaluating the performance of natural fiber-reinforced composites in applications where bending loads are expected [98–101].

5.1.3 Impact strength testing

Impact strength is a measure of a material's ability to absorb energy during sudden impacts or shocks. Impact strength testing is typically performed using an Izod or Charpy impact tester, in which a pendulum with a known mass and velocity strikes a notched specimen. The energy absorbed by the specimen during fracture is calculated from the difference in the pendulum's initial and final potential energy. Impact strength testing is critical for understanding the performance of natural fiber-reinforced composites in applications where they may be subjected to impact loads or dynamic forces [98–101].

5.2 Morphological analysis

Morphological analysis techniques, such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM), are vital for understanding the structure and surface characteristics of natural cellulosic fibers, which can greatly influence their properties and performance in various applications [102].

5.2.1 Scanning Electron Microscopy (SEM)

SEM is a widely used technique for investigating the surface morphology and microstructure of natural cellulosic fibers. SEM utilizes a focused electron beam to scan the fiber surface, generating secondary and back scattered electrons that are collected to form high-resolution images. The SEM technique not only allows for the visualization of fiber surfaces, defects, and the fiber-matrix interface in composite materials, providing valuable insights into the fiber's processing, treatment, and performance but also helps one to investigate the effect of morphology on the mechanical properties of the material. Several researchers to date have used SEM images for detailed learning of natural fiber's morphological structure. One such example is depicted in Figure 1, wherein the researchers utilized the obtained SEM images to comprehensively investigate the effect of morphological structure of various available natural fiber on their tensile properties [103].

5.2.2 Transmission Electron Microscopy (TEM)

TEM is a powerful technique for characterizing the internal structure of natural cellulosic fibers at the nanoscale. In TEM, a high-energy electron beam is transmitted through an ultra-thin specimen, and the resulting interactions between the electrons and the specimen's atoms are used to form high-resolution images. TEM enables the visualization of the internal structure of fibers, including the arrangement and orientation of cellulose microfibrils and the presence of other components such as hemicellulose and lignin. This information is essential for understanding the relationship between the fiber's internal structure and its mechanical, thermal, and chemical properties. An example of TEM image used by a set of researchers who investigated the effect of silver nanoparticles in cotton fiber is depicted in Figure 2 [104].

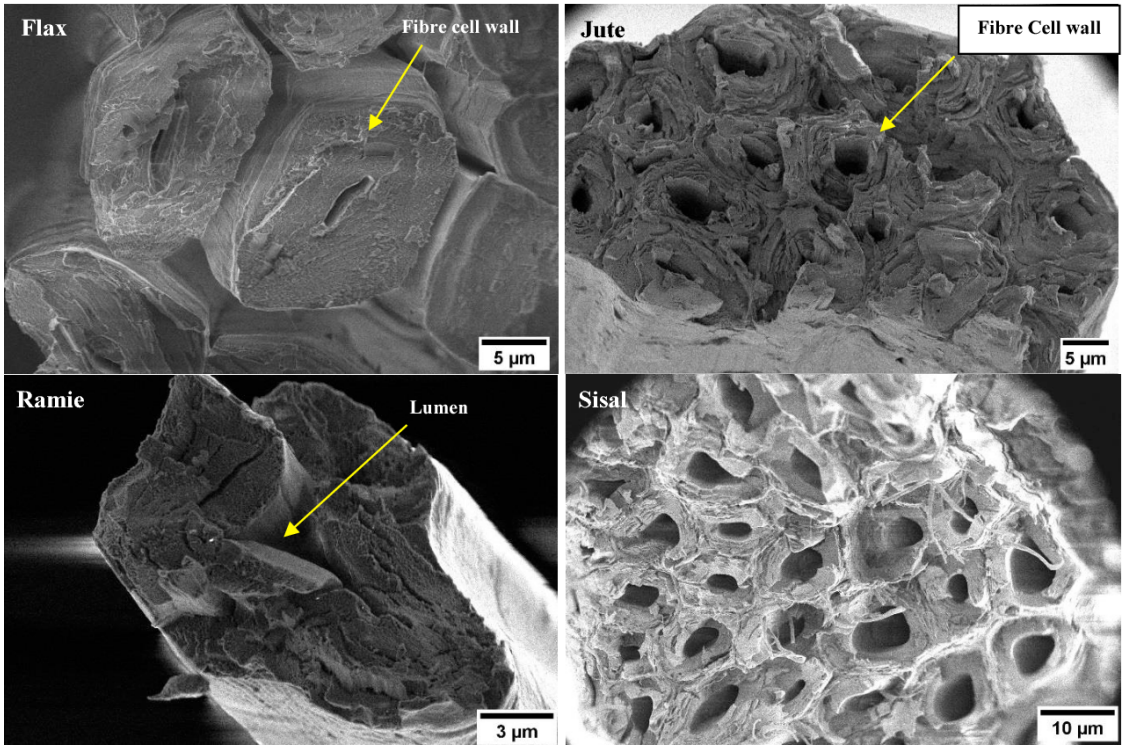

Figure 1: SEM images of the cross section of various natural fibers [103].

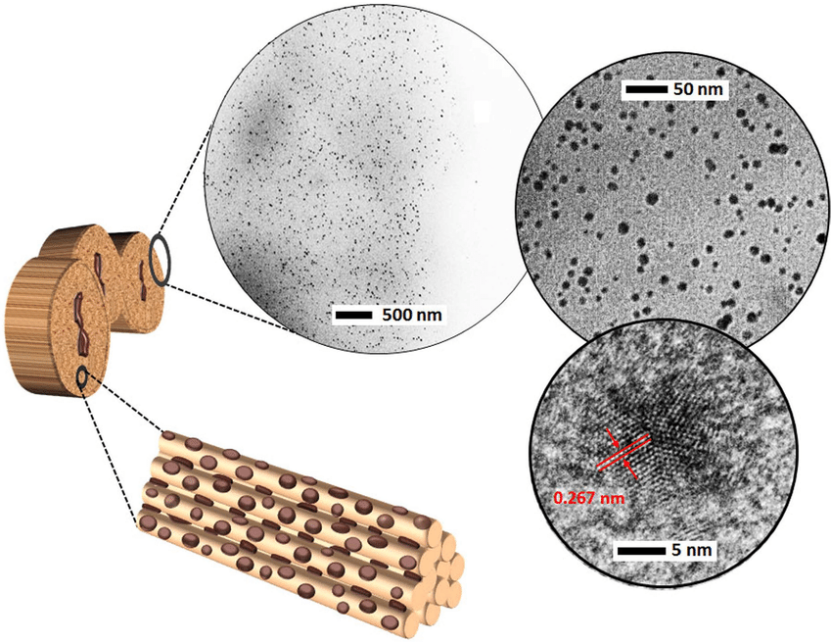

Figure 2: TEM image representing cross-section of silver-cotton nanocomposite fiber[104].

5.3 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

Fourier Transform Infrared Spectroscopy (FTIR) analysis is a powerful technique used to study the chemical structure and composition of natural cellulosic fibers. FTIR can detect the presence of chemical functional groups in fibers, such as hydroxyl, carbonyl, and ether groups, which can affect their properties, including their reactivity and adhesion to other materials. FTIR is commonly used in recent research papers to investigate the functional groups and molecular vibrations of various natural cellulosic fibers. An infrared spectrometer with a Fourier transform can be used to create an infrared absorption spectrum in the frequency range between 500 cm^{-1} and 4000 cm^{-1} in total reflectance mode. The FTIR spectrum is obtained by scanning the fibers at a resolution of 4 cm^{-1} and a scan rate of 32 scans. Figure 3 shows the FTIR spectra of some natural fibers as summarized by Guimares et al. [105]. The general procedure for FTIR analysis of natural fibers includes sample preparation, instrument setup, data acquisition, data analysis, and interpretation of results. The natural fiber sample is prepared by grinding it into a fine powder and pressing it into a thin, flat pellet. The pellet is then placed onto the sample holder for FTIR analysis.

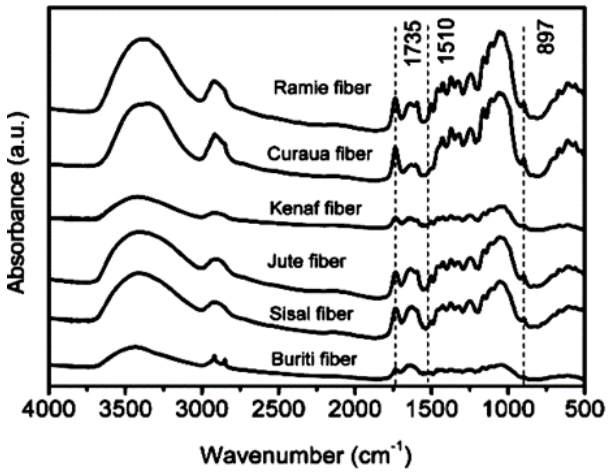

Figure 3: FTIR spectra of Ramie, Curaua, Kenaf, Jute, Sisal, and Buriti fibers [105].

The FTIR instrument is calibrated and set up for analysis of the fiber sample. The appropriate IR source is selected, and the instrument is adjusted to the desired resolution and range. The FTIR instrument is used to collect infrared spectra from the fiber sample. A background spectrum is recorded before the sample is analyzed. The sample is then scanned, and the absorbance or transmittance of the IR radiation is measured at each wavelength. The FTIR spectrum is analyzed to determine the chemical structure and composition of the natural fiber. The peaks in the spectrum are identified and compared to reference spectra to identify functional groups and chemical bonds present in the sample. The FTIR data is interpreted to gain insights into the chemical structure and composition of the natural fiber. For example, FTIR can be used to study the effects of chemical treatments or environmental conditions on the fiber structure and composition [106–108].

5.4 X-ray diffraction (XRD) analysis

X-ray diffraction (XRD) is a widely used technique for investigating the crystalline structure of natural cellulosic fibers. By measuring the diffraction of X-rays by the atoms in the fiber's crystal lattice, XRD can provide information on the degree of crystallinity, crystal size, and crystal structure of the fibers. To perform XRD analysis of natural fibers, the fibers are first ground into a fine powder and pressed into a thin, flat pellet. The pellet is then placed onto the sample holder of the XRD instrument, which is calibrated and adjusted to the desired resolution and range. The XRD instrument is then used to collect X-ray diffraction patterns from the fiber sample. The sample is scanned over a range of angles while the intensity of the diffracted X-rays is measured. The XRD pattern is then analyzed to determine the crystalline structure of the natural fiber. The diffraction peaks are identified and compared to reference patterns to determine the crystal structure and size of the fiber. XRD data can provide insights into the crystalline structure and properties of natural fibers, such as the effects of chemical treatments or environmental conditions on the fiber's crystal structure and degree of crystallinity. Figure 4 shows XRD patterns of fibers from banana, sugarcane bagasse, and sponge gourd obtained by a set of researchers who intended to investigate the structure and crystallinity indices of various plant fibers [105].

5.5 Evaluation of Crystalline Index (CI) and Crystallite Size (CS)

The degree of crystallinity in natural cellulosic fibers can be determined by calculating the crystalline index (CI) using the peak height method [109, 110]. The empirical equation [111] shown in Eq [3] can be used to calculate the CI.

$$\mathrm{CI} = \frac{I_{200} - I_{\mathrm{AM}}}{I_{200}} \tag{3}$$

Here, I_{200} refers to the maximum intensity of the 200 lattice plane at 2θ angle between 22° and 23°, and I_{AM} represents the amount of noncrystalline or amorphous material measured by the height of the valley of the minimum between the peaks, at an angle of 2θ about 18°. The crystallite size (CS) of natural cellulosic fibers can be determined using Scherrer's formula [71] as shown in Eq [4]:

$$\mathrm{CS}_{200} = \frac{k\lambda}{\beta_{200}\cos\theta} \tag{4}$$

Here, $k = 0.89$ is the Scherrer's constant, $\lambda = 0.1541$ nm is the wavelength of the radiation, β_{200} refers to the peak's full width at half-maximum in radians, and θ indicates the corresponding Bragg angle.

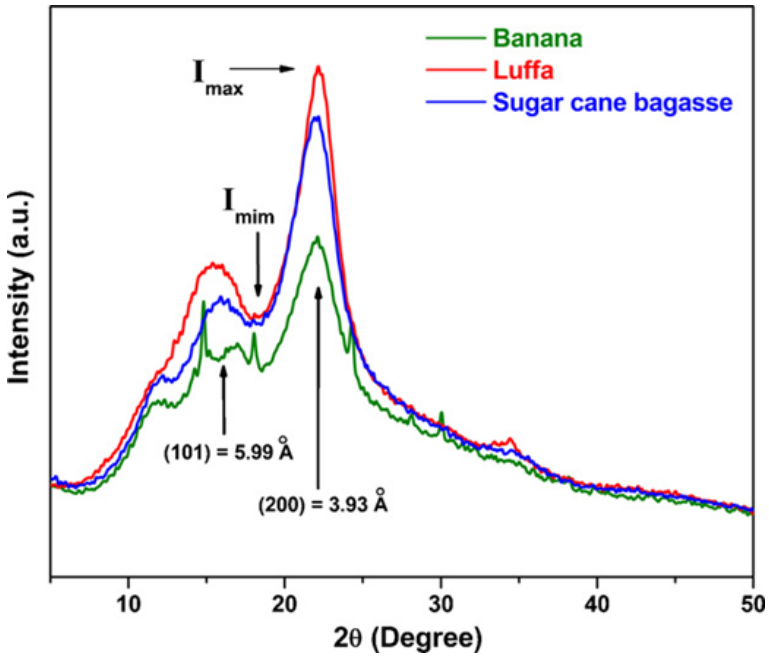

Figure 4: X-ray diffraction patterns of various plant fibers [105].

5.6 Atomic force microscopy (AFM) analysis

Atomic force microscopy (AFM) is a powerful technique used to study the morphology and physical properties of natural cellulosic fibers at the nanoscale. AFM analysis can be performed using an AFM instrument with image processing software to assess the roughness of natural cellulosic fibers. The surface roughness parameters, such as average surface roughness (S_{a}), Root mean square roughness (S_{q} or S_{rms}), Ten points average roughness (S_{z}), skewness (S_{sk}), and kurtosis (S_{ku}), can be determined. The resolution range of the scanner scale in the x, y, and direction is set to 10 μm $\times$ 10 μm $\times$ 70 μm. AFM has been used to study the surface morphology, topography, and mechanical properties of cellulosic fibers in recent research papers. The high-resolution images of the fiber surface provided by AFM can reveal details such as fiber diameter, roughness, and surface defects. These features can affect the fiber's mechanical and physical properties, and studying them can help to understand the performance of the fibers in various applications [23]. To perform AFM microscopy analysis of natural fibers, the sample is first washed with a suitable solvent to remove any impurities and then mounted onto a suitable substrate for AFM imaging. The AFM is calibrated and set up for imaging of the sample. The AFM probe is brought into contact with the sample, and the tip-sample interaction is measured to provide a topographic image of the sample surface. The AFM tip is scanned across the sample surface to obtain a topographic image of the fiber. The AFM can provide information on the surface roughness, fiber diameter, fiber cross-section, and surface morphology of the fiber. In addition to topographic imaging, AFM can also be used to measure the mechanical properties of the fiber, such as stiffness, adhesion, and elasticity. This is achieved by applying a known force to the AFM tip and measuring the resulting deformation of the fiber. The AFM images and force measurements are analyzed to determine the morphology and physical properties of the natural fiber. For example, the fiber diameter, length, and orientation can be determined from the AFM images, while the mechanical properties can be determined from the force measurements. AFM data is interpreted to gain insights into the morphology and physical properties of the natural fiber. For example, AFM can be used to study the effects of chemical treatments or environmental conditions on fiber morphology and properties. Figure represents an AFM image used by set of researchers to investigate the effect of chemical (NaOH) treatment on the sisal fibers at different scan sizes [26].

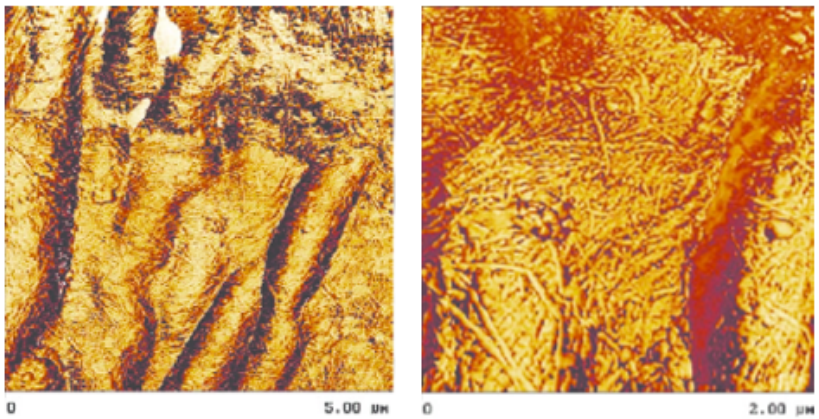

Figure 5: AFM images of NaOH treated sisal fiber at scan size of 5 and 2 microns [26].

6 Conclusion

In conclusion, the methods and techniques for characterizing natural cellulosic fibers have developed substantially, offering a more comprehensive understanding of their properties and potential uses. The extraction processes play a crucial role in obtaining fibers with desired properties, impacting their morphology, crystallinity, and purity. Morphological and structural analysis through scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are widely employed, along with X-ray diffraction (XRD) and Fourier-transform infrared (FTIR) spectroscopy for analyzing crystallinity and chemical composition. Mechanical properties are assessed through tensile testing, flexural strength testing, and impact strength testing. Advanced techniques, such as atomic force microscopy (AFM), confocal Raman spectroscopy, and X-ray photoelectron spectroscopy (XPS), provide deeper insights into the surface chemistry and interactions of natural cellulosic fibers. The ongoing refinement of these techniques will be crucial for the advancement and commercialization of natural cellulosic fibers as sustainable and eco-friendly alternatives to conventional materials. Despite the progress in characterizing natural cellulosic fibers, there is still a need for further development in this field. Future researchers should focus on exploring innovative extraction and surface modification methods to improve the properties and performance of these fibers in various applications.

Additionally, the development of new characterization techniques and the refinement of existing methods will enable a more in-depth understanding of the relationships between fiber properties, processing conditions, and performance in end-use applications. The exploration of new applications and markets for natural cellulosic fibers, such as in energy storage, filtration, and biodegradable materials, will also open up new research areas and commercial opportunities. By advancing the knowledge and technology surrounding natural cellulosic fibers, researchers can help promote the transition to a more sustainable and eco-friendly future.

Declaration of Competing Interests

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.

Funding Declaration

This research did not receive any grants from governmental, private, or nonprofit funding bodies.

Author Contribution

Yashaarth Kaushik: Conceptualization, Investigation, Methodology, Writing - original draft, Writing - review and editing; **Suraparb Keawsawasvong**: Data curation, Formal analysis, Investigation, Methodology, Writing - review and editing; **Nilakshman Sooriyaperakasam**: Data curation, Formal analysis, Investigation, Methodology, Writing - review and editing; **Udit Rathee**: Investigation, Methodology, Validation, Writing - review and editing; **Nithesh Naik**: Conceptualization, Methodology, Project administration, Supervision, Writing - original draft, Writing - review and editing.

References

[1] M. P. Dicker, P. F. Duckworth, A. B. Baker, G. Francois, M. K. Hazzard, and P. M. Weaver, "Green composites: A review of material attributes and complementary applications," *Composites Part A: Applied Science and Manufacturing*, vol. 56, pp. 280–289, jan 2014.

[2] H. S. S. Shekar and M. Ramachandra, "Green composites: a review," *Materials Today: Proceedings*, vol. 5, no. 1, pp. 2518–2526, 2018.

[3] L. Y. Mwaikambo and M. P. Ansell, "Chemical modification of hemp, sisal, jute, and kapok fibers by alkalization," *Journal of Applied Polymer Science*, vol. 84, no. 12, pp. 2222–2234, 2002.

[4] K. L. Pickering, M. G. Efendy, and T. M. Le, "A review of recent developments in natural fibre composites and their mechanical performance," *Composites Part A: Applied Science and Manufacturing*, vol. 83, pp. 98–112, apr 2016.

[5] A. K. Mohanty, M. Misra, and L. T. Drzal, *Natural fibers, biopolymers, and biocomposites*. CRC press, 2005.

[6] A. R. Bunsell, *Handbook of properties of textile and technical fibres*. Woodhead Publishing, 2018.

[7] S. Rwawiire and B. Tomkova, "Morphological, Thermal, and Mechanical Characterization of Sansevieria trifasciata Fibers," *Journal of Natural Fibers*, vol. 12, pp. 201–210, may 2015.

[8] P. Wambua, J. Ivens, and I. Verpoest, "Natural fibres: Can they replace glass in fibre reinforced plastics?," *Composites Science and Technology*, vol. 63, pp. 1259–1264, jul 2003.

[9] V. Raghunathan, J. D. J. Dhilip, G. Subramanian, H. Narasimhan, C. Baskar, A. Murugesan, A. Khan, and A. A. Otaibi, "Influence of Chemical Treatment on the Physico-mechanical Characteristics of Natural Fibers Extracted from the Barks of Vachellia Farnesiana," *Journal of Natural Fibers*, vol. 19, pp. 5065–5075, dec 2022.

[10] S. Manivel, N. Pannirselvam, R. Gopinath, and T. P. Sathishkumar, "Physico-mechanical, Chemical Composition and Thermal Properties of Cellulose Fiber from Hibiscus vitifolius Plant Stalk for Polymer Composites," *Journal of Natural Fibers*, vol. 19, pp. 6961–6976, dec 2022.

[11] P. Venkateshwar Reddy, P. Rajendra Prasad, D. Mohana Krishnudu, and P. Hussain, "Influence of fillers on mechanical properties of prosopis juliflora fiber reinforced hybrid composites," *Materials Today: Proceedings*, vol. 19, pp. 384–387, 2019.

[12] P. Madhu, S. Mavinkere Rangappa, A. Khan, A. Al Otaibi, S. A. Al-Zahrani, S. Pradeep, M. K. Gupta, P. Boonyasopon, and S. Siengchin, "Experimental investigation on the mechanical and morphological behavior of Prosopis juliflora bark fibers/E-glass/carbon fabrics reinforced hybrid polymeric composites for structural applications," *Polymer Composites*, vol. 41, pp. 4983–4993, dec 2020.

[13] V. Vignesh, A. N. Balaji, N. Nagaprasad, M. R. Sanjay, A. Khan, A. M. Asiri, G. M. Ashraf, and S. Siengchin, "Indian mallow fiber reinforced polyester composites: Mechanical and thermal properties," *Journal of Materials Research and Technology*, vol. 11, pp. 274–284, mar 2021.

[14] B. A.N. and N. K.J., "Characterization of alkali treated and untreated new cellulosic fiber from Saharan aloe vera cactus leaves," *Carbohydrate Polymers*, vol. 174, pp. 200–208, oct 2017.

[15] A. S. Madival, D. Doreswamy, S. Maddasani, M. Shettar, and R. Shetty, "Processing, Characterization of Furcraea foetida (FF) Fiber and Investigation of Physical/Mechanical Properties of FF/Epoxy Composite," *Polymers*, vol. 14, p. 1476, apr 2022.

[16] S. D. S. Kopparthy and A. N. Netravali, "Review: Green composites for structural applications," *Composites Part C: Open Access*, vol. 6, p. 100169, oct 2021.

[17] D. Malathi, A. K. Pulikkal, M. T. Ramesan, and S. Nagarajan, "Bio-fiber recovery from industrially discarded shoot waste (Cymbopogon nardus): a comprehensive study for reinforcement in sustainable composites," *Biomass Conversion and Biorefinery*, pp. 1–12, mar 2023.

[18] E. W. Gaba, B. O. Asimeng, E. E. Kaufmann, S. K. Katu, E. J. Foster, and E. K. Tiburu, "Mechanical and structural characterization of pineapple leaf fiber," *Fibers*, vol. 9, p. 51, aug 2021.

[19] P. Manimaran, G. P. Pillai, V. Vignesh, and M. Prithiviraj, "Characterization of natural cellulosic fibers from Nendran Banana Peduncle plants," *International Journal of Biological Macromolecules*, vol. 162, pp. 1807–1815, nov 2020.

[20] N. Saba, M. Jawaid, O. Y. Alothman, and M. T. Paridah, "A review on dynamic mechanical properties of natural fibre reinforced polymer composites," *Construction and Building Materials*, vol. 106, pp. 149–159, mar 2016.

[21] V. S. Sreenivasan, S. Somasundaram, D. Ravindran, V. Manikandan, and R. Narayanasamy, "Microstructural, physico-chemical and mechanical characterisation of Sansevieria cylindrica fibres - An exploratory investigation," *Materials and Design*, vol. 32, pp. 453–461, jan 2011.

[22] S. Kalia, B. S. Kaith, and I. Kaur, "Pretreatments of natural fibers and their application as reinforcing material in polymer composites-a review," *Polymer Engineering and Science*, vol. 49, pp. 1253–1272, jul 2009.

[23] S. Arora, M. Kumar, and G. P. Dubey, "Thermal decomposition kinetics of rice husk: Activation energy with dynamic thermogravimetric analysis," *Journal of the Energy Institute*, vol. 82, no. 3, pp. 138–143, 2009.

[24] N. E. Zafeiropoulos, *Interface engineering of natural fibre composites for maximum performance*. Woodhead Publishing Limited, 2011.

[25] N. I. N. Haris, M. Z. Hassan, R. A. Ilyas, M. A. Suhot, S. M. Sapuan, R. Dolah, R. Mohammad, and M. R. Asyraf, "Dynamic mechanical properties of natural fiber reinforced hybrid polymer composites: a review," *Journal of Materials Research and Technology*, vol. 19, pp. 167–182, jul 2022.

[26] F. L. Leite, P. S. P. Herrmann, A. L. Da Róz, F. C. Ferreira, A. A. S. Curvelo, and L. H. C. Mattoso, "Investigation of Sisal Fibers by Atomic Force Microscopy: Morphological and Adhesive Characteristics," *Journal of Nanoscience and Nanotechnology*, vol. 6, pp. 2354–2361, aug 2006.

[27] M. Asim, K. Abdan, M. Jawaid, M. Nasir, Z. Dashtizadeh, M. R. Ishak, M. E. Hoque, and Y. Deng, "A review on pineapple leaves fibre and its composites," *International Journal of Polymer Science*, vol. 2015, pp. 1–16, 2015.

[28] V. G. Geethamma, R. Joseph, and S. Thomas, "Short coir fiber-reinforced natural rubber composites: Effects of fiber length, orientation, and alkali treatment," *Journal of Applied Polymer Science*, vol. 55, pp. 583–594, jan 1995.

[29] L. Segal, J. J. Creely, A. E. Martin, and C. M. Conrad, "An Empirical Method for Estimating the Degree of Crystallinity of Native Cellulose Using the X-Ray Diffractometer," *Textile Research Journal*, vol. 29, pp. 786–794, oct 1959.

[30] S. Sathish, N. Karthi, L. Prabhu, S. Gokulkumar, D. Balaji, N. Vigneshkumar, T. S. Ajeem Farhan, A. Akilkumar, and V. P. Dinesh, "A review of natural fiber composites: Extraction methods, chemical treatments and applications," *Materials Today: Proceedings*, vol. 45, pp. 8017–8023, 2021.

[31] P. Jagadeesh, M. Puttegowda, S. Mavinkere Rangappa, and S. Siengchin, "A review on extraction, chemical treatment, characterization of natural fibers and its composites for potential applications," *Polymer Composites*, vol. 42, pp. 6239–6264, dec 2021.

[32] S. Mukhopadhyay and R. Fangueiro, "Physical modification of natural fibers and thermoplastic films for composites - A review," *Journal of Thermoplastic Composite Materials*, vol. 22, pp. 135–162, mar 2009.

[33] J. Biagiotti, D. Puglia, and J. M. Kenny, "A review on natural fibre-based composites - Part I: Structure, processing and properties of vegetable fibres," *Journal of Natural Fibers*, vol. 1, pp. 37–68, oct 2004.

[34] M. Zimniewska, "Hemp Fibre Properties and Processing Target Textile: A Review," *Materials*, vol. 15, p. 1901, mar 2022.

[35] P. Lyu, Y. Zhang, X. Wang, and C. Hurren, "Degumming methods for bast fibers—A mini review," *Industrial Crops and Products*, vol. 174, p. 114158, dec 2021.

[36] A. P. Singh and T. Singh, "Biotechnological applications of wood-rotting fungi: A review," *Biomass and Bioenergy*, vol. 62, pp. 198–206, mar 2014.

[37] D. Mboowa, "A review of the traditional pulping methods and the recent improvements in the pulping processes," *Biomass Conversion and Biorefinery*, pp. 1–12, jan 2021.

[38] K. P. Kumar and A. S. J. Sekaran, "Some natural fibers used in polymer composites and their extraction processes: A review," *Journal of Reinforced Plastics and Composites*, vol. 33, pp. 1879–1892, oct 2014.

[39] A. Karimah, M. R. Ridho, S. S. Munawar, D. S. Adi, Ismadi, R. Damayanti, B. Subiyanto, W. Fatriasari, and A. Fudholi, "A review on natural fibers for development of eco-friendly bio-composite: characteristics, and utilizations," *Journal of Materials Research and Technology*, vol. 13, pp. 2442–2458, jul 2021.

[40] O. D. Biko, M. Viljoen-Bloom, and W. H. van Zyl, "Microbial lignin peroxidases: Applications, production challenges and future perspectives," *Enzyme and Microbial Technology*, vol. 141, p. 109669, nov 2020.

[41] Z. Wu, K. Peng, Y. Zhang, M. Wang, C. Yong, L. Chen, P. Qu, H. Huang, E. Sun, and M. Pan, "Lignocellulose dissociation with biological pretreatment towards the biochemical platform: A review," *Materials Today Bio*, vol. 16, p. 100445, dec 2022.

[42] M. Tesfa, A. Dia, M. Ollivier, I. D. Osorio-Leon, and R. Marsac, "An easy spectrophotometric acid-base titration protocol for dissolved organic matter," *MethodsX*, vol. 9, p. 101721, 2022.

[43] L. Qiu, S. Wang, Y. Ma, and F. Li, "Nanofluid and nanopowders," in *Micro and Nano Thermal Transport: Characterization, Measurement, and Mechanism*, pp. 247–284, Elsevier, 2022.

[44] Y. Assadi, M. A. Farajzadeh, and A. Bidari, "Dispersive liquid–liquid microextraction," 2012.

[45] E. Lau, "Preformulation studies," *Separation Science and Technology*, vol. 3, no. C, pp. 173–233, 2001.

[46] O. D. Neikov, D. V. Lotsko, and V. G. Gopienko, "Powder characterization and testing," *Handbook of Non-Ferrous Metal Powders: Technologies and Applications*, pp. 5–44, 2009.

[47] M. M. Alam, M. Maniruzzaman, and M. M. Morshed, "Application and Advances in Microprocessing of Natural Fiber (Jute)-Based Composites," *Comprehensive Materials Processing*, vol. 7, pp. 243–260, 2014.

[48] T. Mudalige, H. Qu, D. Van Haute, S. M. Ansar, A. Paredes, and T. Ingle, "Characterization of Nanomaterials: Tools and Challenges," *Nanomaterials for Food Applications*, pp. 313–353, 2018.

[49] C. Horváth, "High-performance liquid chromatography: advances and perspectives," 2013.

[50] A. M. Siouffi, "High performance liquid chromatography," *Handbook of Food Science, Technology, and Engineering - 4 Volume Set*, vol. 75, no. Sup2, pp. 859–900, 2005.

[51] A. B. Kanu, "Recent developments in sample preparation techniques combined with high-performance liquid chromatography: A critical review," *Journal of Chromatography A*, vol. 1654, p. 462444, 2021.

[52] M. Kašpar and P. Česla, "Characterization of Balsamic Vinegars Using High-Performance Liquid Chromatography and Gas Chromatography," *Applied Sciences (Switzerland)*, vol. 12, no. 18, p. 8946, 2022.

[53] C. M. Conrad, "Determination of Wax in Cotton Fiber a New Alcohol Extraction Method," *Industrial and Engineering Chemistry - Analytical Edition*, vol. 16, pp. 745–748, dec 1944.

[54] S. Park, D. K. Johnson, C. I. Ishizawa, P. A. Parilla, and M. F. Davis, "Measuring the crystallinity index of cellulose by solid state 13C nuclear magnetic resonance," *Cellulose*, vol. 16, no. 4, pp. 641–647, 2009.

[55] M. E. H. E. Nokab, M. H. Habib, Y. A. Alassmy, M. M. Abduljawad, K. M. Alshamrani, and K. O. Sebakhy, "Solid State NMR a Powerful Technique for Investigating Sustainable/Renewable Cellulose-Based Materials," *Polymers*, vol. 14, no. 5, p. 1049, 2022.

[56] R. S. Dassanayake, N. Dissanayake, J. S. Fierro, N. Abidi, E. L. Quitevis, K. Boggavarappu, and V. D. Thalangamaarachchige, "Characterization of cellulose nanocrystals by current spectroscopic techniques," *Applied Spectroscopy Reviews*, vol. 58, no. 3, pp. 180–205, 2023.

[57] W. Zhao, L. D. Fernando, A. Kirui, F. Deligey, and T. Wang, "Solid-state NMR of plant and fungal cell walls: A critical review," *Solid State Nuclear Magnetic Resonance*, vol. 107, p. 101660, 2020.

[58] P. A. Baron, "Measurement of airborne fibers: A review," *Industrial Health*, vol. 39, no. 2, pp. 39–50, 2001.

[59] J. Summerscales, A. Virk, and W. Hall, "A review of bast fibres and their composites: Part 3 - Modelling," *Composites Part A: Applied Science and Manufacturing*, vol. 44, no. 1, pp. 132–139, 2013.

[60] L. C. Barbosa, C. Lorrette, S. Le Bras, E. Baranger, and J. Lamon, "Tensile strength analysis and fractography on single nuclear grade SiC fibers at room temperature," *Journal of Nuclear Materials*, vol. 576, p. 154256, 2023.

[61] Y. Swolfs, I. Verpoest, and L. Gorbatikh, "A review of input data and modelling assumptions in longitudinal strength models for unidirectional fibre-reinforced composites," *Composite Structures*, vol. 150, pp. 153–172, 2016.

[62] A. Arul Marcel Moshi, D. Ravindran, S. R. Sundara Bharathi, V. Suganthan, and G. Kennady Shaju Singh, "Characterization of New Natural Cellulosic Fibers-A Comprehensive Review," in *IOP Conference Series: Materials Science and Engineering*, vol. 574, p. 12013, IOP Publishing, 2019.

[63] I. P. Lokantara, N. P. G. Suardana, I. W. Surata, and I. N. S. Winaya, "a Review on Natural Fibers: Extraction Process and Properties of Grass Fibers," *International Journal of Mechanical Engineering and Technology (Ijmet)*, vol. 11, no. 1, pp. 84–91, 2020.

[64] S. I. Magagula, M. J. Mochane, G. G. Lenetha, J. S. Sefadi, T. H. Mokhothu, and T. C. Mokhena, "The effect of alkaline treatment on natural fibers/biopolymer composites," in *Surface Treatment Methods of Natural Fibres and their Effects on Biocomposites*, pp. 19–45, Elsevier, 2022.

[65] D. P. Ferreira, J. Cruz, and R. Fangueiro, "Surface modification of natural fibers in polymer composites," in *Green Composites for Automotive Applications*, pp. 3–41, Elsevier, 2018.

[66] S. Parveen, S. Rana, and R. Fangueiro, "Macro- and nanodimensional plant fiber reinforcements for cementitious composites," in *Sustainable and Nonconventional Construction Materials using Inorganic Bonded Fiber Composites*, pp. 343–382, Elsevier, 2017.

[67] S. A. Roslan, Z. A. Rasid, and M. Z. Hassan, "Bamboo reinforced polymer composite - A comprehensive review," in *IOP Conference Series: Materials Science and Engineering*, vol. 344, p. 12008, IOP Publishing, 2018.

[68] B. D. Ratner, A. S. Hoffman, and S. L. McArthur, "Physicochemical Surface Modification of Materials Used in Medicine," in *Biomaterials Science: An Introduction to Materials in Medicine*, pp. 487–505, Elsevier, 2020.

[69] X. Li, L. G. Tabil, and S. Panigrahi, "Chemical treatments of natural fiber for use in natural fiber-reinforced composites: A review," *Journal of Polymers and the Environment*, vol. 15, no. 1, pp. 25–33, 2007.

[70] M. M. Kabir, H. Wang, K. T. Lau, and F. Cardona, "Chemical treatments on plant-based natural fibre reinforced polymer composites: An overview," *Composites Part B: Engineering*, vol. 43, no. 7, pp. 2883–2892, 2012.

[71] R. Latif, S. Wakeel, N. Zaman Khan, A. Noor Siddiquee, S. Lal Verma, and Z. Akhtar Khan, "Surface treatments of plant fibers and their effects on mechanical properties of fiber-reinforced composites: A review," *Journal of Reinforced Plastics and Composites*, vol. 38, pp. 15–30, jan 2019.

[72] S. Venkatarajan and A. Athijayamani, "An overview on natural cellulose fiber reinforced polymer composites," *Materials Today: Proceedings*, vol. 37, no. Part 2, pp. 3620–3624, 2020.

[73] R. Khan, R. Jolly, T. Fatima, and M. Shakir, "Extraction processes for deriving cellulose: A comprehensive review on green approaches," *Polymers for Advanced Technologies*, vol. 33, no. 7, pp. 2069–2090, 2022.

[74] R. Z. Khoo, W. S. Chow, and H. Ismail, "Sugarcane bagasse fiber and its cellulose nanocrystals for polymer reinforcement and heavy metal adsorbent: a review," *Cellulose*, vol. 25, no. 8, pp. 4303–4330, 2018.

[75] N. Sharma, N. K. Bhardwaj, and R. B. P. Singh, "Environmental issues of pulp bleaching and prospects of peracetic acid pulp bleaching: A review," *Journal of Cleaner Production*, vol. 256, p. 120338, 2020.

[76] T. A. Khattab, M. S. Abdelrahman, and M. Rehan, "Textile dyeing industry: environmental impacts and remediation," *Environmental Science and Pollution Research*, vol. 27, no. 4, pp. 3803–3818, 2020.

[77] N. M. Stark and L. M. Matuana, "Trends in sustainable biobased packaging materials: a mini review," *Materials Today Sustainability*, vol. 15, p. 100084, 2021.

[78] M. K. Zahran and H. B. Ahmed, "A greener approach for full flax bleaching," *Journal of the Textile Institute*, vol. 101, no. 7, pp. 674–678, 2010.

[79] J. G. March and B. M. Simonet, "A green method for the determination of hypochlorite in bleaching products based on its native absorbance," *Talanta*, vol. 73, no. 2, pp. 232–236, 2007.

[80] S. Dursun and S. Z. Yıldız, "Eco-Friendly Bleaching of Cotton Fabrics Without Heating Using Direct Process Water in the Presence of Sodium Chlorite and Phosphonate," *Journal of Natural Fibers*, pp. 1–14, 2022.

[81] A. Kumar, "Biobleaching: An eco-friendly approach to reduce chemical consumption and pollutants generation," *Green Pulp and Paper Industry: Biotechnology for Ecofriendly Processing*, vol. 6, no. 4, pp. 93–128, 2021.

[82] O. Faruk Dr and M. S. Ain, "Biofiber reinforced polymer composites for structural applications," in *Developments in Fiber-Reinforced Polymer (FRP) Composites for Civil Engineering*, pp. 18–53, Elsevier, 2013.

[83] Q. C. Sun, D. D. Zhang, and L. C. Wadsworth, "Corona treatment on polyolefin films," *Tappi Journal*, vol. 81, no. 8, pp. 177–183, 1998.

[84] M. Dalvi-Isfahan, M. Havet, N. Hamdami, and A. Le-Bail, "Recent advances of high voltage electric field technology and its application in food processing: A review with a focus on corona discharge and static electric field," *Journal of Food Engineering*, vol. 353, p. 111551, 2023.

[85] A. Zille, F. R. Oliveira, and P. A. P. Souto, "Plasma treatment in textile industry," *Plasma Processes and Polymers*, vol. 12, no. 2, pp. 98–131, 2015.

[86] S. Kalia, K. Thakur, A. Celli, M. A. Kiechel, and C. L. Schauer, "Surface modification of plant fibers using environment friendly methods for their application in polymer composites, textile industry and antimicrobial activities: A review," *Journal of Environmental Chemical Engineering*, vol. 1, no. 3, pp. 97–112, 2013.

[87] J. Šimončicová, S. Kryštofová, V. Medvecká, K. Ďurišová, and B. Kaliňáková, "Technical applications of plasma treatments: current state and perspectives," *Applied Microbiology and Biotechnology*, vol. 103, no. 13, pp. 5117–5129, 2019.

[88] J. Profili, S. Asadollahi, P. Vinchon, A. Dorris, S. Beck, A. Sarkassian, and L. Stafford, "Recent progress on organosilicon coatings deposited on bleached unrefined Kraft paper by non-thermal plasma process at atmospheric pressure," *Progress in Organic Coatings*, vol. 147, p. 105865, 2020.

[89] S. K. Nemani, R. K. Annavarapu, B. Mohammadian, A. Raiyan, J. Heil, M. A. Haque, A. Abdelaal, and H. Sojoudi, "Surface modification of polymers: methods and applications," *Advanced Materials Interfaces*, vol. 5, no. 24, p. 1801247, 2018.

[90] R. N. Marcus, C. G. Joseph, Y. H. Taufiq-Yap, S. Soloi, M. H. A. Majid, Z. Jamain, S. A. M. Ali, V. Vijayan, and C. K. Pang, "Current Trends on the Utilization of Ozonation Treatment Process for the Remediation of Dye Wastewater: A Short Review," *Malaysian Journal of Chemistry*, vol. 24, no. 3, pp. 113–124, 2022.

[91] C. Gleissner, J. Landsiedel, T. Bechtold, and T. Pham, "Surface Activation of High Performance Polymer Fibers: A Review," *Polymer Reviews*, vol. 62, no. 4, pp. 757–788, 2022.

[92] D. Hetemi and J. Pinson, "Surface functionalisation of polymers," *Chemical Society Reviews*, vol. 46, no. 19, pp. 5701–5713, 2017.

[93] X. Cao, C. Tan, X. Zhang, W. Zhao, and H. Zhang, "Solution-processed two-dimensional metal dichalcogenide-based nanomaterials for energy storage and conversion," *Advanced Materials*, vol. 28, no. 29, pp. 6167–6196, 2016.

[94] S. Purwar, "Application of natural dye on synthetic fabrics," *International Journal of Home Science*, vol. 2, no. 2, pp. 283–287, 2016.

[95] A. Zaman, F. Huang, M. Jiang, W. Wei, and Z. Zhou, "Preparation, Properties, and Applications of Natural Cellulosic Aerogels: A Review," *Energy and Built Environment*, vol. 1, no. 1, pp. 60–76, 2020.

[96] S. Venkatarajan, C. Subbu, A. Athijayamani, and R. Muthuraja, "Mechanical properties of natural cellulose fibers reinforced polymer composites - 2015-2020: A review," *Materials Today: Proceedings*, vol. 47, pp. 1017–1024, 2021.

[97] G. Rajeshkumar, K. Naveen Kumar, M. Aravind, S. Santhosh, T. K. Gowtham Keerthi, and S. Arvindh Seshadri, "A Comprehensive Review on Mechanical Properties of Natural Cellulosic Fiber Reinforced PLA Composites," *Lecture Notes in Mechanical Engineering*, pp. 227–237, 2021.

[98] T. P. Sathishkumar, P. Navaneethakrishnan, S. Shankar, R. Rajasekar, and N. Rajini, "Characterization of natural fiber and composites - A review," *Journal of Reinforced Plastics and Composites*, vol. 32, no. 19, pp. 1457–1476, 2013.

[99] P. Jagadeesh, M. Puttegowda, P. Boonyasopon, S. M. Rangappa, A. Khan, and S. Siengchin, "Recent developments and challenges in natural fiber composites: A review," *Polymer Composites*, vol. 43, no. 5, pp. 2545–2561, 2022.

[100] C. S. Malalli and B. R. Ramji, "Mechanical characterization of natural fiber reinforced polymer composites and their application in Prosthesis: A review," *Materials Today: Proceedings*, vol. 62, pp. 3435–3443, 2022.

[101] L. Prabhu, V. Krishnaraj, S. Sathish, S. Gokulkumar, N. Karthi, L. Rajeshkumar, D. Balaji, N. Vigneshkumar, and K. S. Elango, "A review on natural fiber reinforced hybrid composites: Chemical treatments, manufacturing methods and potential applications," *Materials Today: Proceedings*, vol. 45, pp. 8080–8085, 2021.

[102] I. Chakraborty, S. Rongpipi, I. Govindaraju, B. Rakesh, S. S. Mal, E. W. Gomez, E. D. Gomez, R. D. Kalita, Y. Nath, and N. Mazumder, "An insight into microscopy and analytical techniques for morphological, structural, chemical, and thermal characterization of cellulose," *Microscopy Research and Technique*, vol. 85, no. 5, pp. 1990–2015, 2022.

[103] S. F. Hamad, N. Stehling, C. Holland, J. P. Foreman, and C. Rodenburg, "Low-Voltage SEM of Natural Plant Fibers: Microstructure Properties (Surface and Cross-Section) and their Link to the Tensile Properties," *Procedia Engineering*, vol. 200, pp. 295–302, 2017.

[104] S. Nam, B. D. Condon, C. D. Delhom, and K. R. Fontenot, "Silver-cotton nanocomposites: Nano-design of microfibrillar structure causes morphological changes and increased tenacity," *Scientific Reports*, vol. 6, p. 37320, nov 2016.

[105] J. L. Guimarães, E. Frollini, C. G. da Silva, F. Wypych, and K. G. Satyanarayana, "Characterization of banana, sugarcane bagasse and sponge gourd fibers of Brazil," *Industrial Crops and Products*, vol. 30, no. 3, pp. 407–415, 2009.

[106] J. L. Xu, K. V. Thomas, Z. Luo, and A. A. Gowen, "FTIR and Raman imaging for microplastics analysis: State of the art, challenges and prospects," *TrAC - Trends in Analytical Chemistry*, vol. 119, p. 115629, 2019.

[107] Y. Liu, "Recent progress in Fourier transform infrared (FTIR) spectroscopy study of compositional, structural and physical attributes of developmental cotton fibers," *Materials*, vol. 6, no. 1, pp. 299–313, 2013.

[108] A. Geremew, P. De Winne, T. Adugna, and H. De Backer, "An overview of the characterization of natural cellulosic fibers," *Key Engineering Materials*, vol. 881 KEM, pp. 107–116, 2021.

[109] V. Jeyabalaji, G. R. Kannan, P. Ganeshan, K. Raja, B. NagarajaGanesh, and P. Raju, "Extraction and Characterization Studies of Cellulose Derived from the Roots of Acalypha Indica L," *Journal of Natural Fibers*, vol. 19, no. 12, pp. 4544–4556, 2022.

[110] Y. Seki, F. Selli, Ü. H. Erdoğan, M. Atagür, and M. Ö. Seydibeyoğlu, "A review on alternative raw materials for sustainable production: novel plant fibers," *Cellulose*, vol. 29, no. 9, pp. 4877–4918, 2022.

[111] S. K. Ramamoorthy, D. Åkesson, M. Skrifvars, and B. Baghaei, "Preparation and characterization of biobased thermoset polymers from renewable resources and their use in composites," in *Handbook of Composites from Renewable Materials* (G. P. Chaudhary and M. K. Sharma, eds.), vol. 1-8, pp. 425–457, Wiley, jun 2017.