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Characterization Methods of Natural Fibers (Cellulose Fibers) as Reinforcement for Polymer Matrix Composites: A Review

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Abstrak

Ketertarikan penggunaan serat alam sebagai penguat pada komposit matriks polimer semakin meningkat, hal ini dilatarbelakangi oleh ketersediaan serat yang melimpah, sifat biodegradabilitas dan biaya produksi yang jauh lebih murah dibandingkan dengan penggunaan serat sintetis. kandungan selulosa pada serat alam sangat berpengaruh terhadap sifat mekanik dan stabilitas termal serat, yang pada akhirnya memberi dampak terhadap kekuatan mekanik komposit. untuk meningkatkan kandungan selulosa pada serat, salah satu usaha yang dapat dilakukan adalah dengan perlakuan alkalisasi dengan larutan NaOH pada serat. Agar dapat mengetahui efektivitas perlakuan kimia dan mempelajari karakteristik fisik dan mekanik serat, serta melihat potensi dan kelayakan serat sebagai penguat pada komposit matriks polimer maka perlu dilakukan serangkaian karakterisasi atau pengujian pada serat. Pada review artikel ini akan diulas secara lengkap metode karakterisasi serat alam serta pengaruh sifat fisik dan mekanik serat terhadap kekuatan komposit. Sehingga diharapkan artikel ini dapat memberikan kontribusi positif terhadap perkembangan material komposit matriks polimer dengan penguat serat alam.

Kata kunci: Karakterisasi; komposit; polimer; selulosa; serat alam

Abstract

The interest in using natural fibers as reinforcement in polymer matrix composites is increasing, driven by their abundant availability, biodegradability, and significantly lower production costs compared to synthetic fibers. The cellulose content in natural fibers has a significant impact on the mechanical properties and thermal stability of the fibers, ultimately affecting the mechanical strength of the composites. To enhance the cellulose content in the fibers, one approach is to use alkali treatment with a NaOH solution on the fibers. To evaluate the effectiveness of the chemical treatment and study the physical and mechanical characteristics of the fibers, as well as to assess the potential and feasibility of the fibers as reinforcement in polymer matrix composites, a series of characterizations or tests on the fibers needs to be conducted. This review article will discuss in depth the methods of characterizing natural fibers and the influence of their physical and mechanical properties on composite strength. This article is expected to provide a positive contribution to the development of polymer matrix composite materials reinforced with natural fibers.

Keywords: *Cellulose; Characterization; Composite; Natural fiber; Polymer*

1. Introduction

Environmental awareness has been increasing over the past few decades, influencing the development and utilisation of more environmentally friendly materials. One of the materials currently being focused on is polymer matrix composites reinforced with natural fibers [1]. The benefits of using natural fibers in polymer matrix composites include their easy availability, natural breakdown, and lower production costs compared to some synthetic fibers, such as glass fiber composites [2]. The use of polymer matrix composites with natural fiber reinforcements in the industrial world has shown great potential due to their adequate mechanical properties for various technical applications. Natural fiber composites are expected to contribute to reducing the carbon footprint and preserving the environment [3].

The natural fiber consists of three main components: cellulose, lignin, and hemicellulose. Cellulose is the primary component that influences the quality and performance of natural fiber [4]. Natural fibers with high cellulose content can provide good mechanical strength and thermal stability to the composite, ultimately resulting in good mechanical

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properties [5]. One way to increase the cellulose content and reduce the lignin and hemicellulose levels in the fiber is by using alkali treatment, which involves soaking the fiber in a specific concentration of NaOH solution [6]. The characteristics of natural fibers significantly influence the properties of composites, including their mechanical strength, physical properties, and thermal properties [7]**.** Therefore, a comprehensive characterization is necessary to understand the characteristics and potential of fibers as reinforcements so that the resulting composites can achieve optimal mechanical strength and physical properties .

This article presents a comprehensive review of the fundamental methods used to characterize fibers as reinforcement in composite materials, based on recent literature. However, further research is required to identify the most effective characterization methods tailored to the specific plant sources utilized as natural fibers. By fully understanding how to characterize fibers used as reinforcements in polymer matrix composites, it is expected that this knowledge will greatly help in creating natural fiber-based composite materials that are strong enough and environmentally friendly.

2. Material and Method

This study is a review of articles related to the characterization methods of natural fibers as reinforcement in polymer matrix composites. The research began by searching for and collecting reputable articles related to natural fiber characterization over the past five years. These articles were checked to see which ones focused on the methods used to study natural fibers, leading to the selection of about 70 articles that are useful for understanding the different ways natural fibers are characterized when used in polymer matrix composites. We then reviewed and analyzed the selected articles to draw conclusions about several key characterization methods for natural fibers.

3. Results and Discussion

3.1. Fiber Extraction

The extraction process is the initial step in obtaining fibers from plants, and it is conducted in various ways depending on the fiber source. One of the simplest and most commonly used extraction methods is water retting (soaking). Water retting is the biological decomposition of the fiber source, utilizing microbial activity to soften the plant tissue that is taken as the fiber source, making it easy to separate the fibers from the plant parts [8].

3.2. Chemical Treatment of Fibers

The chemical treatment of natural fibers used in composites aims to increase cellulose levels and reduce the amounts of lignin and hemicellulose in the fibers [9]. Increasing the cellulose content in the fibers can affect the increase in surface roughness, thermal stability, and mechanical properties of the fibers, as well as reduce water absorption. This, in turn, will provide the composite with good physical and mechanical properties [10]. One of the effective and widely used chemical treatments is alkalization using an NaOH solution. We carry out alkalization by soaking the fibers in a NaOH solution for a specific period and at a specified concentration. The optimal NaOH concentration is crucial for optimizing fiber properties without damaging the cellulose [11,12]. The effectiveness of alkalization with NaOH solution on several types of fibers from various studies is presented in Table 1.

3.3. Chemical Composition Analysis

Chemical composition testing of fibers is conducted to determine the proportions of cellulose, hemicellulose, lignin, and other materials present in the fibers, which helps evaluate their suitability for strengthening composites [18].

Table 1. The Effectiveness of Alkalization on Natural Fibers

No	Fiber Source	Best NaOH Concentration	Best Soaking Time	Effect on Fiber	Reference
1	Dichrostachys Cinerea Bark	5%	90 Minutes	▪ Increase in cellulose content from 72.4% to 78.4%. ▪ Decrease in hemicellulose from 13.08% to 4.6%. ▪ Decrease in lignin from 16.89% to 9.12%.	[13]
2	Furcraea Foetida Leaves	9%	3 Hours	▪ Tensile strength reaches 241.75 MPa due to increased cellulose concentration. ▪ Thermal stability improves up to 385˚C. ▪ Surface roughness increases.	[14]
3	Banyan Aerial	10%	2 Hours	▪ Thermal stability increased by 13% compared to untreated fiber. ▪ Fiber diameter decreased by 16% from untreated fiber. ▪ Cellulose content increased from 43.2% to 55.1%.	[15]
4	Ficus Macrocarpa Bark	5%	4 Hours	▪ Increase in cellulose content from 48.4% to 59.7%. ▪ Crystallinity index increased from 80.2% to 84.75%. ▪ Thermal stability enhancement up to 378.87 ˚C. ▪ Decrease in fiber diameter from 253.80 ± 15 μm to 223.27 ± 12 μm.	[16]
5	Kenaf fiber	6%	1 hour	▪ Cellulose content 78.12% ▪ Lignin content 7.45% ▪ Hemicellulose 21.38%	[17]

The chemical composition of fibers greatly influences their physical, mechanical, and thermal properties [19]. One of the standard methods used in chemical composition testing is the TAPPI (Technical Association of the Pulp and Paper Industry) method [20].

a. Cellulose Composition

To calculate the cellulose composition in fibers, the TAPPI T203 method is used. The basic principle of this method is to remove non-cellulose content from the fiber by soaking it in a concentrated NaOH solution at room temperature. The concentrated NaOH solution dissolves the non-cellulose components, leaving only α-cellulose. The α-cellulose residue is then dried and weighed to determine the cellulose content of the fiber, after comparing it to the initial weight of the fiber [21].

b. Hemicellulose Composition

To calculate the hemicellulose content in the fiber, the holocellulose value must first be determined using the TAPPI T249-75 standard. Holocellulose is the carbohydrate contained in the fiber composed of cellulose and hemicellulose. The hemicellulose content in the fiber is obtained by subtracting the cellulose value from the holocellulose value [22,23] .

c. Lignin Composition

To calculate the lignin composition in fibers, the TAPPI T222 method is used. The testing procedure involves hydrolyzing the fibers with concentrated sulfuric acid. The sulfuric acid dissolves the carbohydrate components in the fibers, while the lignin remains as an insoluble solid residue, which is then weighed to determine the lignin content [24,25].

3.4. Fiber Density Analysis

The density of natural fiber as a reinforcement significantly affects the final performance of the composite. For some applications, it is highly desirable to have a composite with a low density but good mechanical strength [26]. By knowing the fiber density and consistency, it is possible to predict the quality of the composite and control the fabrication process, ensuring the resulting composite meets the desired standard [27].

The pycnometer method, as outlined in ASTM D2320, serves as one of the testing methods for measuring fiber density [28]. The procedure for fiber density testing is as follows:

1. Prepare dry and clean samples.
2. Fill the pycnometer with distilled water at a specific volume to obtain the measured water density, then weigh the pycnometer filled with water.
3. Put the fiber into the pycnometer containing the liquid, then weigh the pycnometer, water, and fiber together.
4. Weigh the pycnometer with only water.
5. Weigh the empty pycnometer.

After the measurement data have been collected, the fiber density can be calculated . The basic principle for calculating material density can be applied using Equation 1.

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

Where ρ Density of natural fibers (g/cm^3); *m* Fiber Mass (g); *V* Fiber Volume (cm^3)

Then, to calculate the fiber density using the pycnometer method, it can be done with equation 2 [29].

$$\rho = \rho_t \frac{m_2 - m_0}{(m_1 - m_0) - (m_3 - m_2)} \tag{2}$$

Where

ρ : Fiber Density (g/cm^3)

ρ_t : Water Density (g/cm^3)

m_2 : Weight of Pycnometer and Powder (g)

m_0 : Weight of Empty Pycnometer (g)

m_3 : Weight of Pycnometer, Powder and Water (g)

m_1 : Weight of Pycnometer and Water (g)

3.5. Single Fiber Tensile Strength Testing

Every natural fiber has different mechanical properties that are influenced by biological and environmental factors, as well as the type of fiber source plant [30]. Single-fiber tensile strength testing on natural fibers used as composite reinforcement is a step to identify the mechanical strength of the fibers, allowing for optimization in their utilization as reinforcement, as well as providing a basis for fiber treatment [31]. The single-fiber tensile strength analysis value can be used as the basis for arranging the volume fraction mixture between the fiber and matrix, using the rule of mixtures principle, so that the resulting composite has optimal mechanical strength [32]. The procedure for testing the single fiber tensile strength refers to the ASTM D 3379–75 standard [33].

The results of the single-fiber tensile test will be presented in the form of a stress-strain graph that illustrates the stress condition on the fiber during the test. The stress-strain graph can be used to interpret the mechanical properties of the fiber as follows:

a. Ultimate Tensile Strength

Ultimate tensile strength refers to the maximum stress that a natural fiber material can withstand before it fails or breaks [34]. To calculate the tensile strength of natural fibers, equation 3 can be used [35].

$$\sigma = \frac{F_{max}}{A} \tag{3}$$

Where

σ : Tensile strength (MPa)

F_{max} : Maximum force at break (N)

A : Fiber cross-sectional area (mm^2)

b. Strain

Strain is the change in the length of a fiber when a force is applied, followed by a return to its original size or the occurrence of failure. To calculate strain, equation 4 can be used [36].

$$\varepsilon = \frac{\Delta L}{L_0} \tag{4}$$

Where

ε : *Strain*

ΔL : Change in fiber length (mm)

L_0 : Initial fiber length (mm)

c. Young's Modulus

Modulus of elasticity is the stiffness value of a fiber against elastic deformation when the fiber is subjected to tensile force. The modulus of elasticity value represents how much strain occurs in the fiber when a force is applied

and returns to its original shape when the force is removed. To calculate the modulus of elasticity, equation 5 is used.

$$E = \frac{\sigma}{\varepsilon} \tag{5}$$

Where

E : Young's Modulus

ε : Strain

σ : Tensile strength (MPa)

3.6. X-Ray Diffraction (XRD)

The crystal structure in natural fibers used as composite reinforcements can be analyzed through XRD testing. The purpose of XRD testing is to obtain the crystallinity index value, which represents the proportion of crystalline to amorphous regions in the natural fiber [37]. The crystallinity index influences the mechanical properties of the fiber; fibers with high crystallinity indices tend to have better mechanical strength and elastic modulus [38,39].

Natural fibers have a semi-crystalline structure consisting of crystalline and amorphous parts. Sharp diffraction peaks come from the crystalline regions, while broad, low diffraction peaks appear from the amorphous parts. The XRD test results are presented in a graph illustrating the diffraction patterns [40,41].

The XRD data were obtained by scanning the fiber sample over a 2θ angle range from 5° to 40°. The scanning results are presented in a graph with an X-axis and a Y-axis, where the X-axis represents the diffraction angle 2θ and the Y-axis represents the X-ray intensity. The central peak that represents the crystalline part in the graph is at 2θ = 22°–23°, while the peak representing the amorphous (disordered) part is at 2θ = 18°. The crystallinity index of the fiber can be determined using the Segal method by applying equation 6 [42].

$$CI\ \% = \frac{I_{002} - I_{am}}{I_{002}}\ X\ 100\% \tag{6}$$

Where

CI : Crystallinity Index

I_{002} : Maximum intensity of the crystalline peak.

I_{am} : Minimum intensity of the amorphous peak

Figure 1 shows one example of an XRD test result graph that can be analyzed on natural fibers

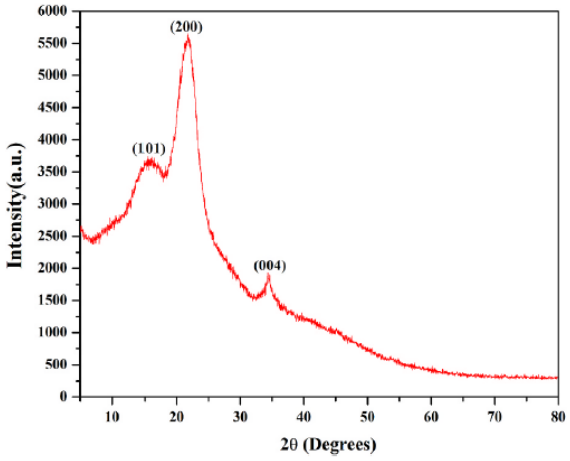

Figure 1 XRD Spectrograph of Calamus Tenuis Cane Fiber [43]

Analysis of the XRD spectrograph results for Calamus Tenuis Cane fiber, as shown in Figure 1, is summarized as follows:

1. The spectrum displays three principal peaks (101, 200, and 004) at 2θ angles of 16°, 22°, and 34.5°, respectively, corresponding to diffraction from crystal planes.
2. The (200) peak at 2θ = 22° with an intensity of 5800 a.u. Represents the crystalline phase.
3. The (101) peak at 2θ = 16° with an intensity of 3630 a.u. is indicative of the amorphous phase. This peak is classified as amorphous because it is located adjacent to the central crystalline peak but is less intense and lacks sharpness.
4. The (004) peak corresponds to a crystalline phase; however, as it is not a primary crystalline peak, it is excluded from the amorphous region determination.

Then, the crystallinity index from the spectrography results, when substituted into the Segal formula, will yield the crystallinity index value of the fiber as follows:

$$CI\ \% = \frac{5800 - 3630}{5800}\ X\ 100\%$$

$$CI\ = 37{,}41\%$$

3.7. Fourier Transform Infrared Spectroscopy Analysis (FTIR)

FTIR testing is an analytical method used to identify the functional groups of fibers, such as -OH (hydroxyl), -C=O (carbonyl), and -CH (methylene/methyl). From the fiber's functional groups, the effectiveness of chemical treatment can be determined by comparing the fiber's functional groups before and after the treatment [44].

The working principle of FTIR is to measure the absorption of infrared radiation by compound molecules. Each molecule in a specific functional group has a characteristic vibration expressed in wave numbers cm^{-1}. The result of FTIR testing is a graph of absorption intensity against wave numbers [45].

In the FTIR spectrum graph, there are horizontal and vertical axes. The horizontal axis represents the wave number (cm^{-1}) with a range from 4000 cm^{-1} to 400 cm^{-1}, while the vertical axis represents the absorption value (transmittance). The wave number range on the FTIR spectrum graph indicates the functional groups present in the fiber [46].

Figure 2 shows one example of a spectrum graph from FTIR testing on natural fibers that can be analyzed.

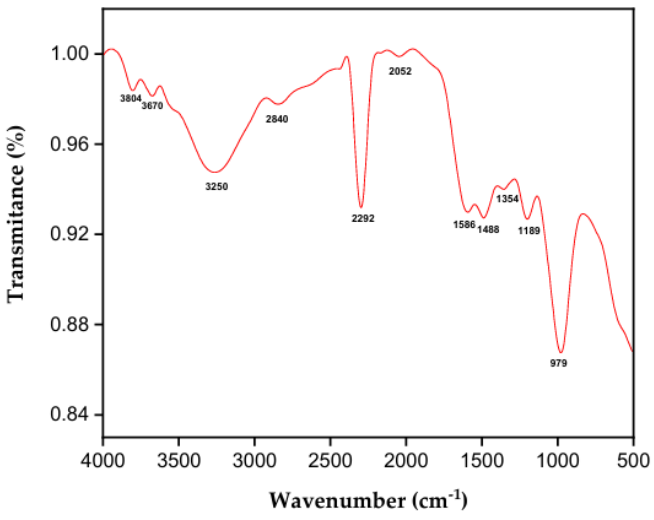

Figure 2 FTIR Testing Spectrum of Manicaria Saccifera Fiber [47].

The analysis results from the FTIR spectrum in Figure 2 are as follows:

1. In the wavenumber range of 3600 to 3100 cm^{-1}, two vibration bands are observed, indicating the stretching of OH groups (hydroxyls).
2. Vibrations of CH and CH_2 bonds occur at a wavenumber of 2840 cm^{-1}, indicating the presence of cellulose and hemicellulose.
3. The stretching of the benzene C=C bonds and the presence of wax are observed at a wavenumber of 2355 cm^{-1}.
4. The absorption band at 2052 cm^{-1} corresponds to the stretching of C≡N and C≡C bonds.
5. The stretching of the aromatic lignin ring bonds is observed at the peak of 1586 cm^{-1}.
6. The frequency of cellulose C-H bonds is observed at the peak of 1488 cm^{-1}. The stretching of lignin acetyl groups appears at the peak of 1354 cm^{-1}.
7. At a wavenumber of 1189 cm^{-1}, there is a stretching of the C-O-C groups, originating from the lignin and hemicellulose content.
8. The peak at 979 cm^{-1} is related to cellulose crystallinity.

In the utilization of natural fibers as composite reinforcements, each functional group on the fiber has the following roles:

1. -OH (Hydroxyl)
   The hydroxyl groups on the fibers serve as adhesion points between the fibers and the matrix, allowing the hydroxyl groups to form hydrogen bonds with the resin [48].
2. -C=O (Carbonyl)
   The carbonyl groups in the fibers influence their mechanical and chemical properties; however, high carbonyl values can also impact their thermal stability [49].
3. -CH (Methyl/Methylene)
   The methyl/methylene groups in natural fibers play a role in determining the hydrophobic properties of the fibers [50].
4. -COO- (Ester)
   Hemicellulose and lignin contain ester groups, which influence the stiffness and flexibility of the fibers [51].

3.8. Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) is a method used to analyze the thermal resistance of natural fibers by measuring the changes in mass of the fibers when heated at a constant rate, thereby determining the temperature at which the fiber undergoes degradation [52,53].

The primary goal of TGA testing on natural fibers is to determine the temperature at which the fibers begin to decompose. How they break down, so we can understand their temperature limits and how well they resist heat when used in composites, as well as how chemical treatments affect their heat stability. By understanding the thermal characteristics of natural fibers, the appropriate applications for the resulting composites can be identified [54].

In TGA testing, a gradual weight change in the fiber occurs due to chemical and physical processes that take place during heating. The initial weight loss occurs due to the release of water content in the fiber, which typically occurs at temperatures below 150°C [55]. After the weight loss due to water loss, the next stage is the degradation of hemicellulose at temperatures around 200°C to 300°C [56]. After hemicellulose degradation, the next stage is lignin degradation, which occurs over a wider temperature range, from 200°C to 500°C [57]. The subsequent stage is cellulose degradation. Cellulose is the main component of natural fibers, so its degradation causes significant weight loss in the fibers and marks the thermal stability limit of the fibers. Cellulose degradation occurs in the temperature range of 300°C to 400°C [58].

The final stage in TGA testing is the formation of residue. After the organic components (water, hemicellulose, cellulose, and lignin) of the natural fibers degrade, non-organic components that do not undergo chemical reactions or evaporation remain, forming residue. Residue formation occurs at temperatures above 500°C[59].

One example of a graph resulting from TGA testing on natural fibers that can be analyzed is shown in Figure 3.

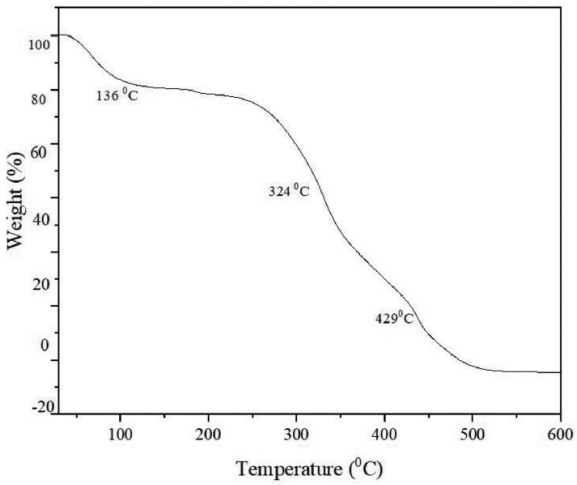

Figure 3 TGA Curve of Sirisha Bark Fiber [60].

From the TGA result graph, the fiber degradation can be analyzed to occur in four main phases as follows:

1. The evaporation of water content in the fiber causes weight loss from room temperature to 136°C.
2. Weight loss, which reaches 55% at a temperature of 324°C, is caused by the degradation of cellulose and hemicellulose.
3. Lignin degradation increases the decomposition temperature up to 429°C.
4. The final stage is the formation of char.

3.9. Morphological Characterization of Fibers / Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is a method used in the characterization of natural fibers to visualize the microstructure of the fibers with high resolution, which cannot be achieved with conventional microscopes [61]. SEM testing is conducted to identify and study the morphology of natural fibers, including their texture and defects, which can affect the interfacial bonding between the fibers and the matrix [62].

One example of an SEM image that can be analyzed is shown in Figure 4.

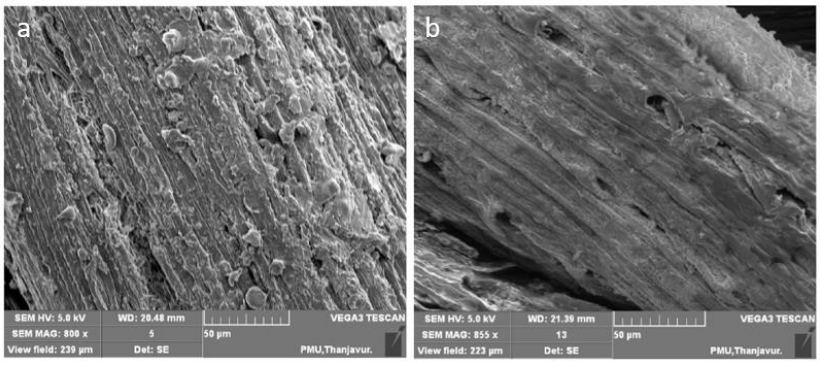

Figure 4 SEM micrographs of Pennisetum Orientale fiber [63]

From the results of SEM testing on Pennisetum orientale fibers, the analysis can be made as follows:

1. SEM observations were made with a magnification of 800x for image (a) and 855x for image (b).
2. The scale bar in the pictures indicates 50 µm.
3. The fiber surface in image (a) is untreated fiber; the fiber surface appears to have many impurities and holes.
4. The fiber surface in image (b) is a chemically treated fiber; the fiber surface appears cleaner.

4.1. Contact Angle Analysis

The natural fiber's vulnerability to liquids can be categorized into two properties: hydrophilic and hydrophobic. Hydrophilic is the property of natural fibers that tend to absorb liquids (attract liquids), whereas hydrophobic is the property of natural fibers that tend to resist liquid absorption [64,65].

The hydroxyl groups on natural fibers make them susceptible to water absorption, or hydrophilic. The ability of the fibers to absorb water can make them swell, which weakens the bond between the fibers and the water-repellent matrix. As a result, the hydrophilic nature of the fibers tends to reduce the mechanical strength of the composite [66,67]. The hydrophilic nature of fibers can make them susceptible to moisture, which affects the composite's resistance to environmental conditions. Fibers with hydrophobic properties have good bonding with polymer matrices that are also hydrophobic, so a good interfacial bond between the fiber and the matrix can produce a homogeneous composite with good mechanical properties [68,69].

Contact angle testing is conducted on natural fibers to analyze their characteristics concerning water, determining whether they are hydrophilic or hydrophobic. Contact angle testing is performed by dropping liquid onto the fibers, then analyzing the angle (contact angle) formed by the liquid drop to determine the fiber's wettability properties. The hydrophilic and hydrophobic nature of natural fibers can be identified from the contact angle values of the fibers, where a contact angle value of less than 90° indicates hydrophilic properties, and greater than 90° indicates hydrophobic properties [70].

Figure 5 shows one example of an image that can be analyzed, resulting from the contact angle test on natural fibers.

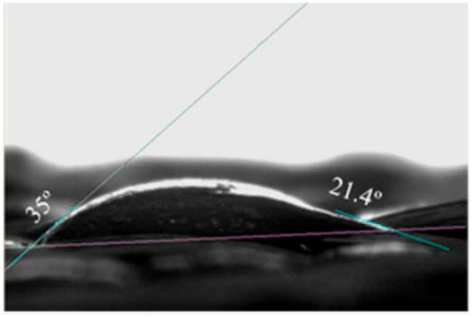

Figure 5 Contact Angle on Natural Fibers [71]

From Figure 5, it can be analyzed that:

1. The fiber contact angles are 35° and 21.4°.
2. Contact angle values below 90° indicate that the fibers are hydrophilic.
3. The two sides of the water droplet have different angles, indicating differences in the fiber surface or non-uniform chemical properties of the fibers.

5. Conclusion

Each natural fiber has distinct characteristics, and to utilize natural fibers as reinforcement for polymer matrix composites, it is crucial to understand the fiber's properties so that the resulting composite can meet the feasibility standards when used in various applications. The fiber characteristics that need to be known include the fiber's cellulose content or chemical composition, single fiber tensile strength, fiber density, fiber crystallinity index, fiber functional groups, fiber thermal stability, and fiber morphology. Knowing the properties of a fiber can help determine whether it is suitable for use as reinforcement in composites for specific applications.

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