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Effect of Alkaline Chemical Treatment on the Surface Morphology and Tensile Characteristics of Diospyros Ebenum fibers

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Abstract

This research examines the impact of alkaline treatment on tensile properties and surface morphology. Numerous studies have been conducted on chemically treating fibers to improve the bonding strength between the fibers and the matrix by altering the surface properties of the fibers. No research was conducted to examine the alterations in surface morphology and tensile properties of the fiber independently. Fibers of Diospyros ebenum were subjected to treatment with 2% concentrated sodium chloride (NaCl) for varying durations and subsequently evaluated for tensile strength and surface alterations pre- and post-treatment. The chemically treated single fibers exhibited an increasing trend in tensile strength. The fibers treated for up to 60 minutes exhibited maximal strength, while those treated for longer than 60 minutes shown diminished tensile strength. The fiber subjected to chemical treatment for 60 minutes had a maximum tensile strength of 9.54 MPa.

Keywords

Single fibril tensile strength, Sodium chloride, Diospyros ebenum, ASTM D3379-75 standard.

1. Introduction

It is not often recognized that chemical treatments directly and substantially improve the strength of an individual fiber in standalone form (Islam et al., 2024). The principal advantages of chemical treatments typically pertain to the interaction between the fiber and its surrounding matrix, rather than a significant enhancement of the fiber's intrinsic strength (Hosseini et al., 2023). Chemical treatments predominantly influence the fiber–matrix interaction. Chemical treatments can alter the surface of natural fibers, enhancing their compatibility with polymer matrices (Mohammed et al., 2022). This enhances the adhesion between the fiber and the matrix, resulting in a more durable composite material (Mylsamy et al., 2024). Certain treatments can eliminate contaminants or alter the surface chemistry of the fiber,

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thereby improving fiber–matrix adhesion. Chemical treatments can also modify the surface properties of fibers, rendering them either hydrophobic or hydrophilic depending on the treatment employed (Verma, 2021).

Although certain treatments may marginally influence the mechanical properties of the fiber, such as slightly increasing stiffness, they are not generally intended to directly improve the tensile strength of the individual fiber (Leites et al., 2024). If the objective is to enhance the strength of individual fibers, alternative approaches such as the use of synthetic fibers or the application of surface coatings may be more effective (Mohammed et al., 2023). Alkaline treatment can remove contaminants and lignin from natural fibers, thereby improving adhesion with the matrix (Begum et al., 2021). Silane treatment enhances the compatibility between natural fibers and polymer matrices, leading to improved interfacial bonding (Dharmakrishnan et al., 2022).

The application of graphene coatings can considerably enhance the tensile strength of fibers, particularly when combined with alkali treatment (da Silveira et al., 2022). A study reported that rotting hardwood in Sri Lanka provides a unique niche for the discovery of fungal species with biotechnological potential (Senthilkumar et al., 2018). A 5 w/v% sodium hydroxide solution has been used to modify the surface of Pongamia pinnata fiber by varying immersion durations between 15 and 75 minutes (Umashankaran and Gopalakrishnan, 2021). Another study showed that the cellulose content of waru tree trunk fiber increased after silane treatment, with a crystalline index of 63.02%, and the single fiber exhibited a tensile strength of 243.94 MPa (Wirawan et al., 2022). Basalt fibers treated with hybrid sizings consisting of a silane coupling agent and silicon dioxide nanoparticles showed uniform sizing distribution on the fiber surface, resulting in a significant improvement in tensile strength (Agrawal and Durai Prabhakaran, 2024).

This study analyzes the impact of chemical treatment on a standalone single ebony fiber. The investigation focused on the tensile properties and surface characteristics prior to and following the chemical treatment.

2. Materials and methods

2.1 Extraction and preparation of fibers

The process of extracting fiber from ebony bark employs a natural retting technique, which involves soaking in water to aid in the separation of fibers from non-fibrous components. The removal of ebony bark from the tree's stem initiates a traditional retting process, which effectively loosens the fibers for subsequent processing. The outer bark is first removed from the stem by hand, employing sharp instruments for precision. The bark is subsequently divided into smaller, more manageable sections and immersed in clean water for a duration of three days. The soaking process, referred to as retting, facilitates microbial activity and water in breaking down the pectins and other binding substances that connect the fibers with lignin and hemicellulose. Throughout this phase, it is crucial to maintain complete submersion of the bark to facilitate consistent retting and inhibit fungal development. Following a three-day period, the retted bark is extracted from the water. At this point, the fibers have been adequately loosened and can be separated by hand. The pliable bark is delicately struck with wooden mallets or stones to enhance the loosening of the fibers. The fibrous material undergoes a meticulous hand-stripping process, during which the long fibers are carefully teased out. The extracted fibers undergo a rigorous washing process with clean water to eliminate any remaining impurities, such as decomposed tissues and microbial remnants.

After the washing stage, the fibers undergo sun-drying on clean surfaces or drying racks to minimize moisture content and maintain their quality. Effective drying inhibits mold development and preserves fiber integrity. After achieving full dryness, the fibers can be collected and preserved for subsequent applications, including the production of bio-composites or traditional crafts. This water retting technique is environmentally sustainable and economically viable, utilizing natural microbial processes and requiring minimal chemical input, thereby making it appropriate for the sustainable production of fiber from ebony bark.

2.2 Fiber extraction, chemical treatment, processing

A sodium chloride (NaCl) solution was prepared for the chemical treatment of ebony fibers by dissolving 2 grams of NaCl in 100 millilitres of distilled water. This yields a 2% (w/v) concentration solution, appropriate for altering the surface characteristics of natural fibers, with a molar mass of NaCl being 58.44 g/mol and a molar concentration of 0.342 M. The solution was mixed meticulously to guarantee the complete dissolution of the salt. The ebony fibers were subsequently immersed in the NaCl solution for specified treatment durations of 30, 60, and 90 minutes. This concentration was chosen to improve fiber characteristics while preventing degradation, resulting in enhanced compatibility in composite applications.

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Following extraction, ebony fibers are subjected to a chemical treatment involving sodium chloride (NaCl) to enhance their physical properties and improve compatibility with matrix materials in composite applications. The procedure entails submerging the desiccated fibers in a NaCl solution for different time intervals: 30 minutes, 60 minutes, and 90 minutes. Initially, a 2% NaCl solution is created by dissolving the appropriate quantity of salt in distilled water. The ebony fibers, once extracted and sun-dried, are subsequently immersed in this solution for the designated time periods. This treatment effectively eliminates residual non-cellulosic components and improves the surface roughness of the fibers, leading to enhanced adhesion with polymers like lactic acid in bio-composite applications. Following each treatment duration of 30, 60, and 90 minutes, the fibers are extracted from the solution and thoroughly rinsed with distilled water to remove any residual salt. The extracted samples are subsequently air-dried or subjected to low-temperature oven drying to mitigate the risk of thermal degradation. The application of NaCl contributes to the stabilization of the fiber structure through the reduction of microbial content and enhancement of moisture absorption resistance. The differences in treatment duration enable the assessment of the ideal conditions that produce the most effective mechanical and bonding performance for subsequent composite fabrication investigations.

The chemical treatment of ebony fibers using sodium chloride (NaCl) was conducted under standard laboratory conditions. The procedure was carried out at room temperature, around $32 \pm 2°C$, and under standard atmospheric pressure. The treatment was conducted under natural daylight, ensuring ambient lighting without the emergence of extra heat or UV sources. The NaCl solution was formulated with distilled water, and the fibers were completely submerged in the solution for durations of 30, 60, and 90 minutes, respectively. During the treatment process, the environment was maintained in a stable condition, devoid of external contaminants, to guarantee reliable outcomes. Following treatment, the fibers underwent rinsing and were subsequently air-dried in consistent room conditions. Following the chemical treatment involving sodium chloride, the ebony fibers underwent a comprehensive rinsing process with distilled water to eliminate any residual salt and impurities. The purified fibers were subsequently distributed uniformly on pristine, dry surfaces and subjected to direct sunlight for a duration of two days. The natural drying process effectively lowered the moisture content without the use of artificial heat, thereby maintaining the structural integrity of the fiber. The fibers were flipped at regular intervals to guarantee consistent drying across all surfaces. The process of sun drying effectively contributed to the removal of any lasting odors or microbial activity, thereby rendering the fibers appropriate for subsequent testing or application in bio-composite production.

3. Experimentation

A single fiber tensile test evaluates the tensile characteristics of individual fibers by applying a tensile force until failure occurs (Mesquita et al., 2026). This method is widely used to determine strength and modulus, particularly for synthetic fibers, although its application to natural fibers may introduce certain complications. The procedure involves clamping a single fiber, applying a controlled tensile load, and recording the force and displacement until fracture (Žižek et al., 2024). The primary objective of the tensile test is to determine the tensile strength, modulus, and failure strain of individual fibers (Stanciu and Ioan, 2021).

The tensile strength of single filament materials was determined in accordance with the ASTM D3379-75 standard (Musanif and Thomas, 2024). In this method, a single fiber is mounted on a slotted paper or cardboard tab using an adhesive to ensure proper alignment during testing (Bhakta et al., 2023). The tab ends are secured within the grips of a tensile testing apparatus. The central section of the tab is either ruptured or charred and the fiber is exposed to a steady loading rate until it ultimately fails. The tensile strength is determined by taking the force at failure and dividing it by the average cross-sectional area of the fiber. The accuracy of SFTT may be lower and exhibit greater variability when assessing natural fibers in comparison to synthetic fibers. Selection and preparation of fibers can significantly influence outcomes and contribute to variability. Elements such as the measurement of fiber diameter and strain can lead to inaccuracies.

4. Results and Discussion

Observations indicated that the tensile strength of individual fibers was low during the initial testing phase prior to chemical treatment. The tensile strength values of the individual fibers following the chemical treatment process, conducted over varying durations, are presented in Table 1. Figure 1 presents a bar chart illustrating the average tensile strength values of individual fibers following different durations of chemical treatment. Three sets of fibers were utilized for each duration of chemical treatment.

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Table 1. Single fiber tensile strength before and after chemical treatment at different durations

Fiber specimen no.	Fiber tensile strength in MPa			
	Without chemical treatment	After 30 minutes of chemical treatment	After 60 minutes of chemical treatment	After 90 minutes of chemical treatment
1	4.67	6.51	9.54	7.76
2	3.32	4.99	7.47	6.24
3	4.85	6.52	6.53	5.07

The results indicated that the tensile strength of individual fibers improves following chemical treatment, with variations in tensile strength observed depending on the duration of the treatment. The average single fiber tensile strength was observed to be 4.28 MPa. Following 30 and 60 minutes of NaCl chemical treatment, the average tensile strength surged to 6.01 MPa and 7.85 MPa, respectively. It was observed that when the duration of fiber treatment exceeds 60 minutes, the average tensile strength of the individual fiber diminishes to 6.36 MPa.

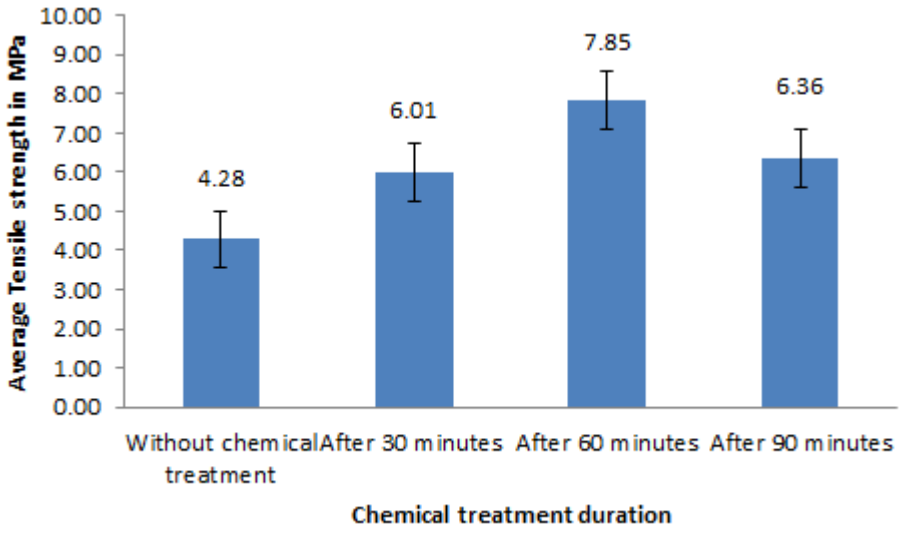

Figure 1. Average tensile strength of single fibers after different durations of chemical treatment

Figure 2 illustrates the variations in single fiber tensile strength across different durations of chemical treatment. After 30 minutes of NaCl chemical treatment, there was a 28.75% increase in the tensile strength of the fibers. The fibers subjected to chemical treatment for 60 minutes exhibited a 45.45% enhancement in tensile strength when compared to the untreated fibers. Upon extending the chemical treatment duration to 90 minutes, a decline in the tensile strength of the fibers was observed, decreasing from 45.45% to 32.67%.

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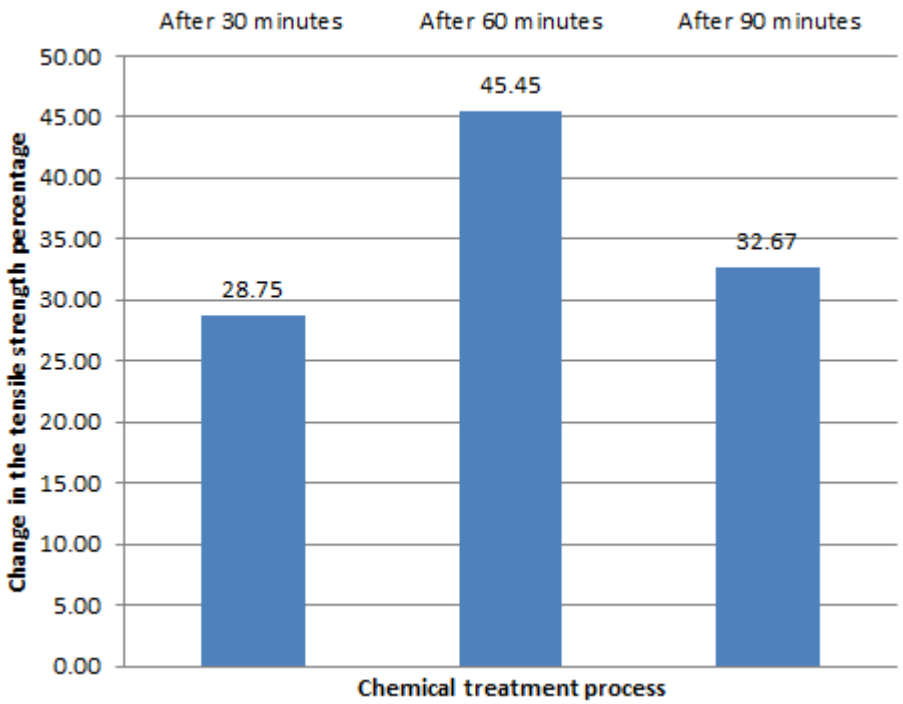

Figure 2. Variations in the tensile strength percentages with respect to chemical treatment time duration

Following a 90-minute chemical treatment, a notable decrease in the tensile strength of the single fiber was observed, influenced by various factors and reasons that impact fiber strength. Chemical treatments such as alkalization or bleaching serve the main purpose of eliminating non-cellulosic components, including lignin, hemicellulose, waxes, and pectin from the fiber surface. This treatment was typically implemented to enhance the adhesion between the fiber and matrix in polymer composites. Cellulose microfibrils, which are the primary load-bearing components, are bound by lignin and hemicellulose, which also serve as a protective sheath. Extended exposure to chemicals can lead to the breakdown and, in some cases, the dissolution of the essential structural matrix located deep within the fiber. With the extension of the treatment duration, an increased quantity of material was dissolved and removed from both the surface of the fibers and their internal structure. Intensive and extended chemical treatment can interfere with the structured arrangement of microfibrils, which are aligned at a particular angle relative to the fiber axis. The degradation of the interfibrillar region, through the removal of lignin and hemicellulose, can lead to misalignment of the fibrils, thereby diminishing the efficiency of load transfer along the fiber length while under tension.

5. Conclusions

Thus, this study examined the impact of the duration of chemical treatment on the tensile strength of individual natural fibers. It was observed that the tensile strength increased with the duration of the chemical treatment. The strength of the fibers treated for up to 60 minutes shows an increase, while a decline in strength is observed when the treatment duration exceeds 60 minutes. Following a 90-minute chemical treatment, a reduction of 32.67% in tensile strength was observed when compared to the fibers subjected to a 60-minute treatment. Initially, the tensile strength of the raw fibers measured 4.28 MPa, which surged to a peak of 7.85 MPa following 60 minutes of chemical treatment. A 45.45% increase in tensile strength was observed after 60 minutes of treatment when compared to the strength of the raw fibers. The main factors contributing to the reduction in tensile strength of the fibers were identified as the degradation of non-cellulosic components and the removal of the protective sheath from the micro fibrils, which serve as a load-bearing element. Extended exposure to chemicals disrupts the alignment of the fibrils, leading to the formation of micro-cracks and voids within the fiber structure. The presence of these defects serves as points of stress concentration, resulting in a notable decrease in tensile strength and a higher probability of premature fiber failure.

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