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A Review: Fracture Structure of Natural Fiber after Treatment with Various Alkali Chemicals

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Abstract. *Natural fibers, including both animal and plant fibers, have many applications in a variety of sectors, therefore knowing the effects of chemical treatments on their characteristics is critical. This study evaluates the impact of different chemical treatments, including silanes, acetylation, benzoylation, peroxides, and several coupling agents, including maleation. In addition, this paper discusses treatment using acrylation and acrylonitrile, stearic acid, sodium chlorite, oleoyl chloride, isocyanate, and permanganate. These treatments alter the surface structure of the fibers by changing their chemical composition and shape. Silane and coupling maleation chemicals typically increase fiber attachment to the polymer matrix, whereas acetylation and benzoylation frequently improve resistance to moisture and microbial attacks. Peroxide and permanganate can produce oxidation, which reduces fiber strength, but grafting acrylation, acrylonitrile, and isocyanate improves resistance and temperature stability. Stearic acid and oleoyl chloride increase hydrophobicity, whereas sodium chlorite treatment alters the lignocellulose structure, increasing density and strength. This study also examines the impacts of biological treatment with fungi, that may change fiber structure via biodegradation pathways. This study's findings shed light on how various chemical treatments might alter the durability and strength of natural fibers, and also their use in industry and material technology. A practical implication of this research is that optimizing chemical treatments can significantly enhance the performance of natural fibers in specific applications, such as improving moisture resistance, mechanical strength, and adhesion in composite materials, thereby expanding their potential in industries like automotive, construction, and packaging.*

Keywords: *Chemical treatment; silane agents; acetylation and benzoylation; fungal treatment; and fiber surface structure.*

Type of the Paper: Review Article.

1. Introduction

Natural fibers, including plant and animal fibers, play an important role in a wide range of sectors, from textiles to composite products and infrastructure. Natural fibers provide several advantages, including natural mechanical characteristics, moisture absorption, and biodegradability [1,2]. However, to suit the increasingly particular requirements of modern applications, these fibers are frequently modified. Chemical treatment has been the primary way for improving the properties and applications of natural fibers, enabling them to compete with

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more modern synthetic fibers [1,2].

As technology advances, numerous chemical treatment procedures have been developed to change the surface structure of fibers. Treatments that include silane agents, acetylation, benzoylation, and peroxides have been shown to improve fiber functional characteristics. Silane agents, for example, improve fiber adhesion to the polymer matrix by generating cross-links, whilst acetylation and benzoylation improve moisture and microbial resistance [3]. Recent research has revealed that these treatments have a considerable impact on the surface morphology of fibers, and that in turn impacts the performance of the produced materials. Other chemical treatments, like mediation linking agents, have a major influence on fiber structure. Maleation enhances the connection between fibers from nature and polymers, which is critical for composite components [4,5]. In addition, sodium chlorite is utilized to remove lignin from fibers, boosting fiber density and strength, while acrylation and acrylonitrile bonding enhance resistance to external influences and improve thermal stability [6,7]. Chemical treatments involving isocyanates, stearic acid, oleoyl chloride, and permanganate can also be used to affect fiber characteristics [8]. Isocyanates can improve fiber thermal stability, whereas stearic acid and oleoyl chloride boost hydrophobic characteristics and thus moisture resistance [9–11]. On the other hand, Permanganate, causes oxidation that affects fiber strength and surface morphology [12]. Research on these treatments reveals how chemical changes can improve fiber performance in different applications.

In addition to chemical treatments, current conventional research has looked into biological treatments using fungi as an alternative. This method has the potential to provide a more ecologically friendly solution by changing the fiber structure via biodegradation mechanisms. Fungi can alter the lignocellulosic structure of fibers, affecting fiber density and strength while boosting resistance to bacterial degradation [13]. In addition to chemical treatments, current conventional research has looked into biological treatments using fungi as an alternative. This method has the potential to provide a more ecologically friendly solution by changing the fiber structure via biodegradation mechanisms. Fungi can alter the lignocellulosic structure of fibers, affecting fiber density and strength while boosting resistance to bacterial degradation [14].

Therefore, the purpose of this review is to provide a thorough understanding of the numerous chemical treatment processes used on natural fibers, as well as their effects on the fibers' surface structure and performance. A deeper comprehension of the effectiveness and applicability of each treatment approach is envisaged as a result of gathering and assessing existing material. This review's findings will help researchers design more efficient and environmentally friendly natural fiber-based products. Furthermore, this information can help researchers and industry practitioners identify the best treatment approach for their particular applications. Overall, this literature strives to create a complete reference that not only describes the changes appearing on the fiber surface following chemical treatment, but also makes recommendations for further study and development in this sector.

2. Surface texture of natural fibers after treatment with various alkali chemicals

2.1. Treatment with silane agents

The morphological alterations of fiber surfaces after treatment with silane compounds have emerged as a significant topic in natural fiber-based composite material research. Silane agents operate as a linker between the fiber and the polymer matrix, increasing compatibility and adherence. The interaction between silane and natural fibers occurs in steps, beginning with the hydrolysis of the alkoxy group in silane ($R\text{- }Si(OR')_3$) to form a silanol group ($R\text{-}Si(OH)_3$). Through a condensation reaction, this silanol group links with the hydroxyl group (-OH) on the natural fiber's surface, forming a siloxane bond. Following that, the silane molecules linked to the fiber can connect to each other via interactions between the silanol groups, generating a three-dimensional siloxane a system (-Si-O-Si-), increasing the fiber's adhesion to the polymer matrix and improving the composite's mechanical properties [14]. This treatment usually consists of an interaction between functional elements on the fiber surface and silane, resulting in a very thin film of silane on the fiber, generally ranging from **1 to 10 nanometers (nm)**, depending on the concentration of the silane used, the fiber type, and the specific treatment conditions [8,15]. This layer can cover gaps, smooth the surface, and boost the wettability of the fiber thanks to the polymer matrix. Overall, these modifications help to increase the mechanical characteristics of the composite, including tensile strength, rigidity and resistance to delamination. Silane treatment has also been shown to alter fiber morphology and decrease microporosity and surface fractures [15]. When the composite is mechanically loaded, a smoother and more homogeneous surface results in a more even stress distribution, extending the material's service life [16]. Fig. 1 illustrates, for instance, how silane alters the surface structure of hemp fiber. The surface of hemp fiber is depicted in SEM micrographs in Fig. 1, both before and after silane treatments of 2%, 4%, and 6%. The surface structure of hemp fiber following silane agent treatment, as depicted in the above picture, reveals notable alterations in the fiber's surface structure. The hemp fiber in Fig. 1(a) has a surface that is visible to the naked eye, with visible fissures and tiny voids prior to being treated with silane. This can lower the mechanical qualities of the resultant composite since it shows that the fiber has low adherence to the matrix. The fiber surface appears smoother and more uniform following treatment with silane agents, as shown by Fig. 1(b), 1(c), and 1(d). There are fewer cracks and voids that virtually vanish. Increasing the binding between the hemp fiber and the matrix is one of the coupling agents that the silane agent does. Improved adhesion qualities from this treatment may lead to an increase in the composite's tensile strength and elastic modulus. The optimization of hemp fiber-based materials' performance in engineering applications is thus greatly aided by silane treatment.

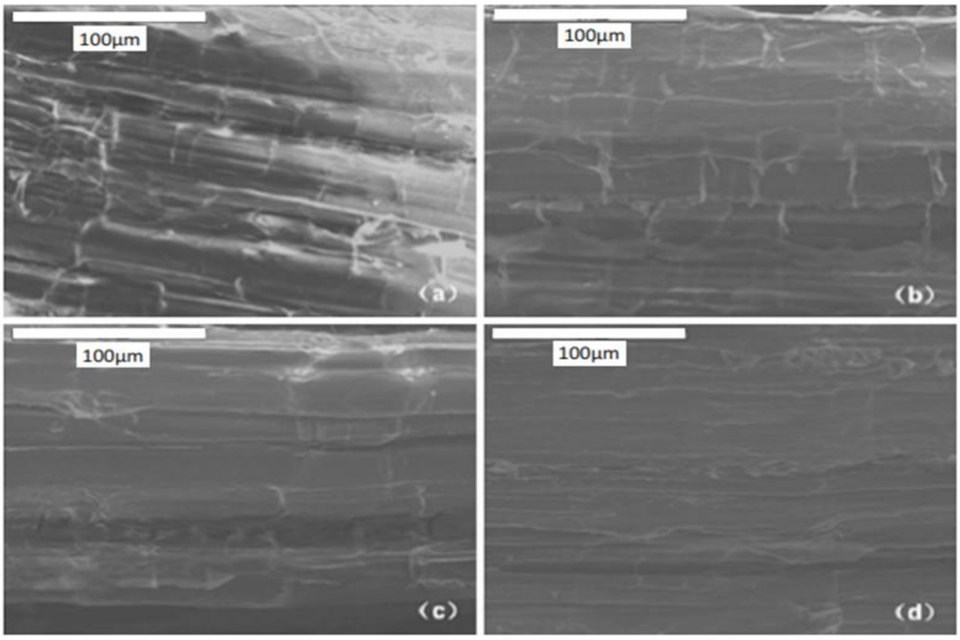
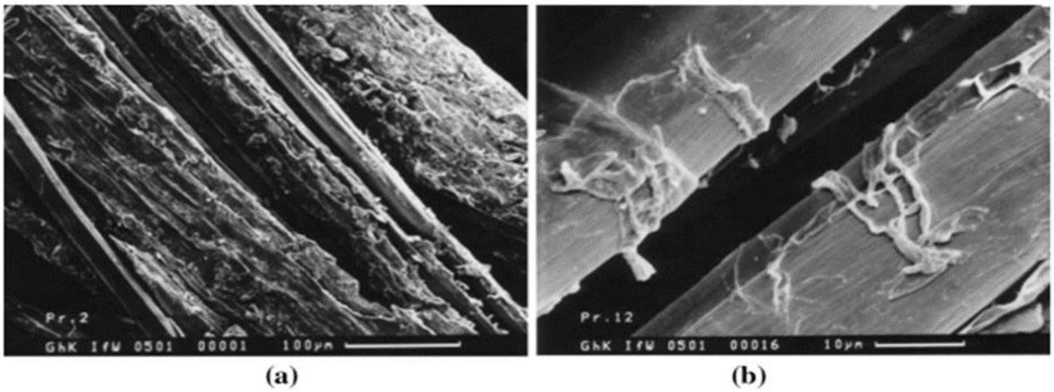


Fig. 1. SEM micrographs of the surface of hemp fiber (a) Untreated, (b) treated 2% silane, (c) treated 4% silane, and (d) treated 6% silane [17].

2.2. *Treatment with acetylation*

Grafting acetyl groups into the fiber's cellular structure is the goal of the acetylation process. The fiber bundle is first soaked in acetic acid to initiate the acetylation process. It then undergoes a high-temperature treatment with acetic anhydride for 1-3 hours. OH, and the arboxylic/anhydride bonds in plant fibers undergo an esterification reaction in this method. The acetylation process replaces hydroxyl groups (-OH) on the surface of fibers like cellulose with acetyl groups ($-COCH_3$) from acetic anhydride. In this process, the hydroxyl groups interact with acetic anhydride to form acetate groups that are linked to the fiber and release acetic acid as a byproduct. Acetylation reduces the fiber's affinity for water (hydrophobicity) through decreased the quantity of hydrophilic hydroxyl groups, boosting waterproofing and dimensional stability while also improving the rigidity and strength of composites derived from these natural fibers. The fiber surface may become smoother as a result of this interaction with acetylene, which may also produce wax and cuticles [8,18]. SEM images of the surface characteristics of hemp fibers treated with 18% acetylation and those left untreated are displayed in Fig. 2. The untreated hemp fibers' rough, multilayered surface is depicted in Fig. 2a, together with loose-looking microfibers from the main surface. The presence of impurities and uneven texture suggest that lignin, hemicellulose, and various non-cellulosic components are still present in these natural fibers, which may hinder their ability to interact with the polymer structure in the composite. The fiber surface appears significantly smoother and cleaner following acetylation treatment at a concentration of 18%, as demonstrated in Fig. 2b. By reducing the amount of lignin and hemicellulose on the fiber, acetylation treatment eliminates the majority of contaminants and loose microfibers [19]. By choosing 18%, the study likely aims to optimize fiber performance in terms of both mechanical properties and moisture resistance, making it a suitable concentration for improving interfacial bonding in composite materials while maintaining fiber strength. Contextually, this concentration offers a midpoint that enhances fiber characteristics without the adverse effects seen at higher treatment levels. Enhancing the compatibility between the fiber and the polymer matrix, the outcome is a more uniform surface. Reducing the amount of water absorbed by the fibers is a significant benefit for applications in humid regions, and it can also enhance the mechanical and thermal durability of hemp fiber-based composites [20,21].

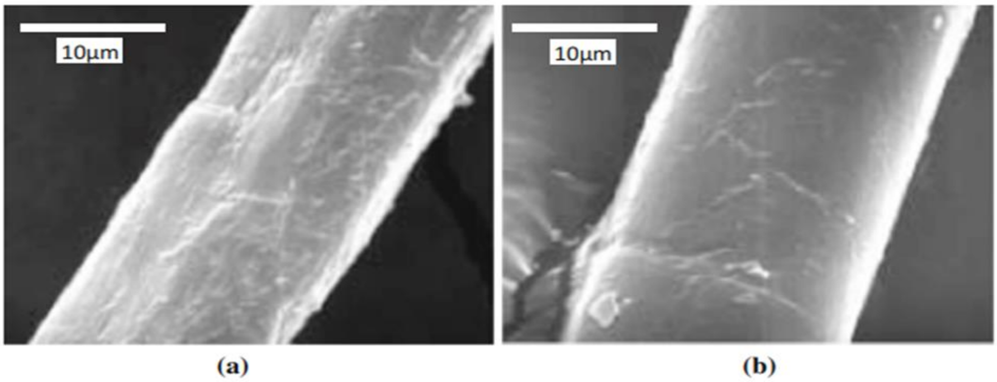


(a) (b)

Fig. 2. SEM micrographs of hemp fibers, (a) untreated hemp fiber surface and (b) hemp fiber treated with 18% acetylation [22].

2.3. Treatment with benzoylation

The interfacial bonding and thermal stability of natural fibers have been found to be enhanced by the application of benzoyl chloride for a surface modification solution. Pretreatment exposes the reactive OH groups on the fiber surface and sets the stage for the rest of the treatment procedure. When the fiber is treated with benzoyl, its surface structure is altered and its OH groups are substituted with benzoyl groups [23,24]. The surface microstructure of both benzoyl-treated and untreated ramie fibers is depicted in SEM images in Fig. 3. With protrusions and microfibers still attached, the surface of the untreated ramie fibers in Fig. 3a appears uneven and rough. The abrasive surface of the fiber suggests the existence of lignin, hemicellulose, and additional constituents that may diminish its ability to adhere to the polymer matrix. The tensile strength and moisture resistance of the resultant composite may be decreased by the inclusion of these materials, among other mechanical effects. The shape of the fiber surface underwent a substantial alteration following the benzoylation treatment, as demonstrated in Fig. 3b. Protrusions and associated impurities decreased, and the fiber surface became more uniformly smooth. Adding benzoyl groups to the fiber's surface through benzoylation treatment can lessen the fiber's attraction for water and increase its compatibility with the polymer matrix [25]. Benzoylation-induced non-cellulosic material removal can also enhance the mechanical contact between the fibers and matrix, hence boosting the composite's strength and stability [23].



(a) (b)

Fig. 3. SEM micrographs of the hemp fiber surface, a) without treatment and b) after benzoylation treatment [26].

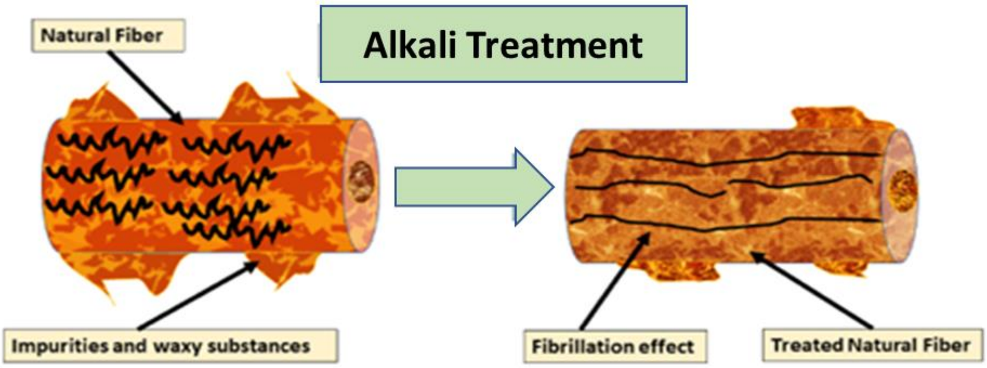


Fig. 4. Schematic of alkaline treatment [27].

Because of their smoother and chemically active surfaces, the treated fibers are better able to form strong bonds with the polymer matrix, leading to composite materials with improved mechanical properties and increased resilience to environmental deterioration. Fig. 4 presents the process scheme for alkali treatment. These modifications also suggest that benzoylation treatment is a useful method for enhancing ramie fiber qualities for composite applications [28,29]. To improve the performance of natural fibers in composites, more research could examine how different benzoylation conditions affect the final properties of fibers and composites. The results could then be compared with those of other fiber treatments, like acetylation or salinization.

2.4. Treatment with peroxide

Fibers treated with peroxide are meant to have three main effects: they should become more thermally stable, less capable of absorbing moisture, and more adherent to the matrix at the interface [30]. Peroxide treatment involves the incorporation of peroxide-induced polyethylene grafting into the fiber surface, whereby peroxide free radicals react with both the OH groups in the fiber and matrix. The surface appearance of ramie fibers treated with peroxide and those that are not is depicted in SEM micrographs in Fig. 5.

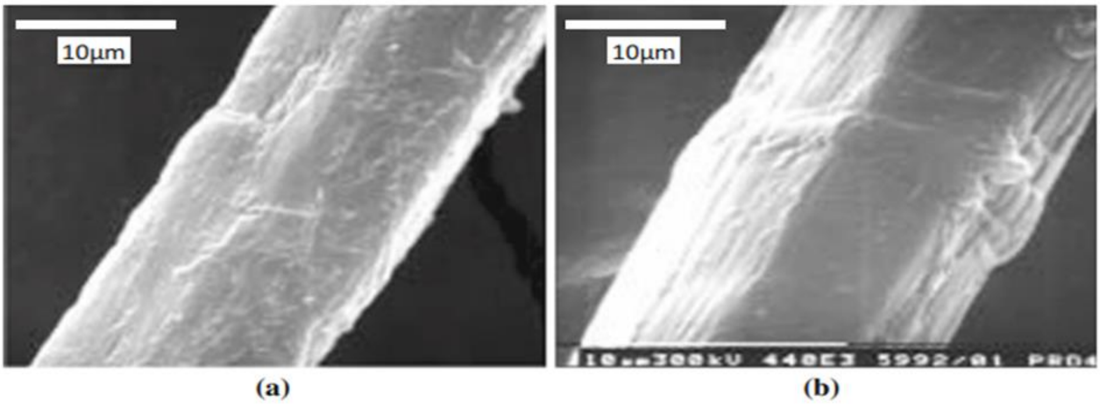


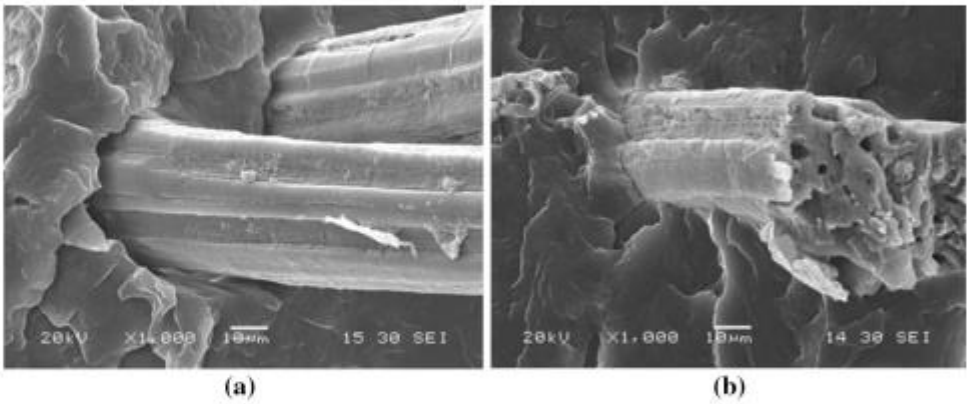
Fig. 5. SEM photos of the hemp fiber surface, a) untreated and b) treated benzoylation [26].

Fig. 5a depicts the surface roughness of the untreated hemp fiber, exhibiting uneven layers, protrusions, and microfibers. The texture of the fiber suggests the presence of non-cellulose constituents like lignin and hemicellulose, as well as other elements that may impact the adhesion characteristics between the fiber and matrix. The mechanical qualities of the resultant composite may also be diminished by this structure's potential to produce less-than-ideal interactions between the fiber and matrix. The morphological structure of the fiber surface underwent a substantial alteration following treatment with peroxide, as seen in Fig. 5b. The removal of hemicellulose, lignin, and other non-cellulose constituents from the fiber by peroxide treatment is known to be effective, leaving the fiber surface cleaner and more structured. The fiber has a smoother, more uniform surface and fewer protrusions and microfibers than before the peroxide treatment, as seen in Fig. 5b. These findings suggest that peroxide works well to open up a more pristine cellulose structure, which can enhance the fiber's interface characteristics with the matrix in composite applications.

To elaborate, the use of peroxide treatment not only leaves the fiber surface clean but also has the ability to alter the functional groups present there, so enhancing the fiber's polarity and bonding with the polymer matrix. Tensile strength and elastic modulus, two of the composite's mechanical qualities, may be enhanced as a result [30,31]. Additionally, the fiber's resistance to deterioration at high temperatures can be increased by applying a peroxide treatment. Peroxide-treated ramie fibers have a smoother surface and more consistent structure, which can improve performance in many composite applications, particularly in sectors that need materials with greater mechanical qualities and resistance to the environment.

2.5. *Treatment with maleated anhydride*

In order to improve the interfacial characteristics between natural fibers and the polymer matrix in composites, maleated coupling agents, such as maleated anhydride, have been frequently employed to treat natural fibers. Maleic anhydride modifies the functional groups on the fiber surface when it connects to them through the maleation procedure, which is how these coupling agents work. This approach improves the interface with the polymer matrix by increasing the polarity and reactivity of the fiber surface, especially for non-polar polymers like polypropylene. The end result is a more homogeneous and chemically reactive fiber surface structure, as well as an increased number of functional groups which may bind to the matrix. This immediately enhances the composite's adhesion and stress distribution. In addition to forming carbon-carbon bonds with the matrix's polymer chains, maleated coupling agents serve to sufficiently interact with the functional surface of the fibers. When maleic anhydride reacts with the OH groups in the amorphous area of the fiber cellulose structure, long-chain polymers are coated on the fiber surface [32].



(a) (b)

Fig. 6. SEM micrographs of jute fiber surfaces, (a) untreated and (b) treated with maleic anhydride [32].

The removal of OH groups from the fiber cells by this coating decreases the hydrophilic capacity of the fibers, which in turn causes the OH groups of the fibers to form covalent bonds with the anhydride groups of maleic anhydrides, resulting in the formation of link interfaces and effective interlocking between the fibers and matrix. Maleic anhydride (MA) treated and untreated ramie fiber surfaces are depicted in SEM micrographs in Fig. 6. Fibre failure via tearing instead of interfacial failure was observed in the MA-treated fibers due to improved fiber-resin adhesive at the interface.

As mentioned earlier, the amount of lignin and hemicellulose that might obstruct fiber-matrix interactions is decreased when fiber surfaces are modified using maleated coupling agents.

The altered fibers' surface tends to be smoother and more impure-free, which improves the fibers' capacity to disperse the load uniformly throughout the matrix. Along with improving the composites' mechanical strength, this treatment also makes the fibers more thermally stable and resistant to moisture. The mechanical qualities and durability of composites manufactured using maleated fibers have been significantly improved in a number of studies, making them more appropriate for structural applications in a variety of industries, including consumer products, construction, and automotive. Maleated anhydride treatment of natural fibers improves the durability of fiber-reinforced composites, allowing them to be used in a variety of practical applications. Malleated fiber composites, for example, are utilized in interior panels, door trims, and dashboards because of their increased mechanical strength, lightweight characteristics, and moisture resistance, which contribute to fuel efficiency and longevity. In construction, these composites are used in non-load-bearing constructions like wall panels, insulating materials, and decking boards, where improved thermal stability and moisture resistance are critical for long-lasting performance. Maleated fiber composites are used in consumer products such as furniture, athletic equipment, and packaging materials because of their higher strength-to-weight ratio and ecological responsibility, which provide practical advantages over conventional materials.

Maleated anhydride treatment improves fiber-matrix adhesion, resulting in more uniform stress distribution and prevents delamination in composites. It also improves mechanical and thermal properties. This increases the material's strength as well as its durability in a variety of environments. Overall, maleated anhydride treatment greatly expands the application potential of natural fiber composites, enabling them to be used in a wide range of challenging industries where performance, sustainability, and efficiency are important factors.

2.6. Treatment with sodium chlorite ($NaClO_2$)

Sodium chlorite ($NaClO_2$) treatment is generally used to delignify natural fibers for composite material applications. The primary goal of this treatment is to eliminate lignin, a non-cellulose element that contributes to the fibers' stiffness and dark color. Delignification is an important process because lignin can hinder fiber-matrix interaction in composites, lowering overall performance. Sodium chlorite acts by oxidizing and degrading lignin, effectively removing it from the fiber structure. As a result, the cellulose within the fiber is more exposed, and the fiber surface becomes smoother and cleaner, enhancing its potential to interact with polymer matrices. This chemical activity not only improves the fibers' aesthetic features by making them whiter and more polished, but it also improves their strength characteristics by lowering hemicellulose concentration, which can lead to fiber breakdown. By eliminating lignin and lowering hemicellulose concentration, the sodium chlorite treatment produces more structurally homogeneous fibers that can transmit loads more effectively when used in composites. These changes improve the interfacial adhesion between fibers and the matrix, hence increasing the final composite material's mechanical strength, thermal properties, and moisture resistance. In conclusion, sodium chlorite treatment improves the durability of natural fibers in composite applications, leaving them more suitable for use in industries like automotive, building, and consumer goods [6,33].

During the discussion, it was noted that the fiber surface treated with $NaClO_2$ had improved adhesion to the polymer matrix. Reducing hemicellulose concentration with sodium chlorite ($NaClO_2$) treatment may enhance fiber performance as it is less crystalline, more hydrophilic, and more thermally unstable than cellulose. Hemicellulose absorbs moisture quickly, which can decrease fiber-matrix adhesion in composites, resulting in lower mechanical strength and durability. limiting the hemicellulose percentage makes the fibers more resistant to moisture absorption, enhancing the integrity of the fiber-polymer interface and limiting the possibility of swelling or degradation over time. $NaClO_2$ treatment increases the cellulose-to-hemicellulose ratio of fibers. This results in a more crystalline and robust fiber structure, which improves mechanical qualities such as tensile strength and stiffness. Furthermore, a decrease in hemicellulose reduces the fibers' susceptibility to heat breakdown, enhancing their thermal durability when employed in high-temperature composite materials. Overall, lignin removal and hemicellulose reduction produce a cleaner, more crystalline fiber surface, making it better suitable for high-performance composites, particularly in moisture-sensitive applications like construction and automotive components.

When coupled with the inclusion of coupling agents or polymer matrices, the removal of lignin not only enhances the fiber's physical appearance but also its chemical reactivity on the surface. More equal stress distribution in the composite material results from a cleaner fiber surface, which enhances mechanical qualities like tensile strength and fracture resistance. Furthermore, fibers treated with sodium chlorite shown enhanced resilience against hydrolytic and thermal degradation, rendering them more robust under a range of environmental circumstances. Because of this alteration, natural fibers are now more competitive in industrial settings where high-performance composite materials are needed. Sodium chlorite treatment involves bleaching the fibers with $NaClO_2$ in an acidic solution. By increasing the fibers' hydrophobicity and eliminating moisture, this method makes the fibers more flexible [34]. SEM micrographs of the surface characteristics of flax fibers treated with $NaClO_2$ and those that are not are displayed in Fig. 7.

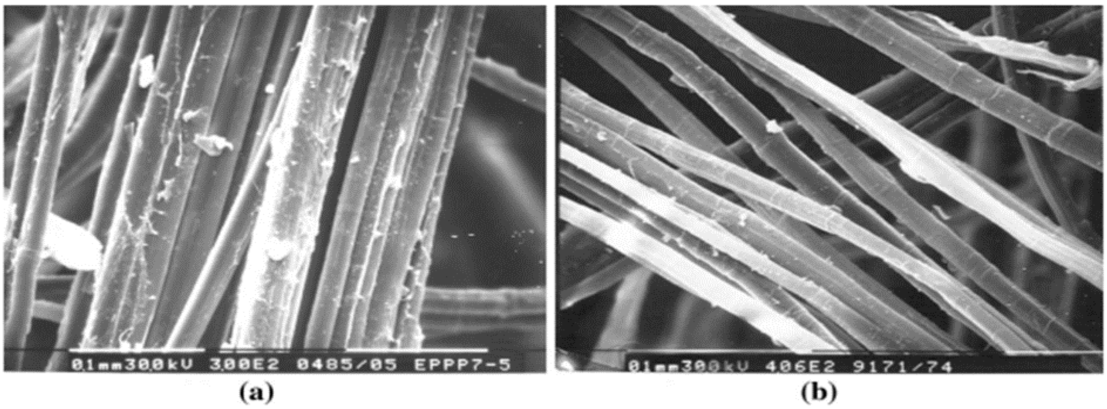


Fig. 7. SEM photos of the surface of hemp fibers a). untreated, b) treated sodium chlorite [35].

2.7. treatment with acrylation and acrylonitrile grafting

Acrylation and acrylonitrile grafting are common chemical modification techniques for improving the compatibility and mechanical endurance of natural fibers in composites. These treatments are important because untreated natural fibers often have poor adherence to polymer matrix, limiting the mechanical capabilities and stability of fiber-reinforced composites. By altering the fiber surface, acrylation and acrylonitrile grafting improve interfacial interaction between fibers and a range of polymer matrices, both polar and nonpolar, hence increasing composite performance. The acrylation procedure involves adding acrylate groups to the fiber via a polymerization reaction in which acrylate monomers create covalent connections with the hydroxyl groups in the cellulose. Acrylonitrile grafting involves creating polymer linkages with nitrile groups (-C≡N) on the fiber surface. Both treatments provide a more reactive fiber surface with increased polarity, which improves the fiber-matrix interaction. Furthermore, the creation of a polymer layer over the fibers creates a more complex surface structure, allowing for further property tweaking to fulfill specific composite use demands such as improved strength, thermal properties, and moisture resistance [8]. After grafting, a polymer layer covers the fiber, giving it a more complicated surface structure and new properties that can be adjusted to meet the requirements of a composite application.

Acrylation and acrylonitrile modification offers many benefits in composite applications. Fibers having polymer matrices, particularly polar polymers like polyester or polyurethane, can have their adhesion qualities improved by acrylate grafting. Acrylonitrile grafting enhances moisture barrier qualities and chemical resistance in the meantime. Combining these two methods yields fibers with exterior structures that are more amenable to different kinds of polymers, improving the mechanical qualities of the final composites, including wear resistance and tensile strength. But in order for this treatment to continue to benefit the fiber without lessening its inherent benefits, the grafting procedure must be adjusted to avoid sacrificing the structural integrity of the fiber. For usage in a variety of industrial applications that call for high-performance composite materials, natural fibers can thus be made more versatile and functional through the processes of acrylation and acrylonitrile grafting.

Acrylic acid ($CH_2 = CHCOOH$) reacts with the cellulose OH groups on the fiber in the acrylation and acrylonitrile grafting processes, allowing reactive cellulose macro radicals more access to the polymerization media. The hydrophilic OH groups of the structure of the fiber are reduced to create an ester bond with the cellulose OH groups, and the carboxylic acid of the coupling agent eliminates the moisture from the fiber [13]. When peroxide radicals attach themselves to acrylic acid in the matrix, they take hydrogen atoms out of the polymer chains and form oxygen-oxygen bonds [8]. Fiber and matrix integration therefore increases the bonding capacity and interfaces stress transmission mechanism. Acrylation and acrylonitrile grafting can significantly improve the performance of natural fibers in composite applications. These treatments enhance fiber-matrix adhesion by decreasing hydrophilic hydroxyl groups and generating strong covalent connections between the fiber surface and polymer matrices, resulting in enhanced stress transmission and overall mechanical qualities. The modified fibers' higher moisture resistance and thermal stability make them ideal for application in high-demand industries including automotive, construction, and consumer goods, where durability and performance are crucial. Finally, these chemical alterations allow for the creation of stronger, more dependable, and adaptable natural fiber composites, broadening their application across multiple industries. Surface appearance of palm fibers treated with acrylate, acrylonitrile, and untreated is depicted in SEM micrographs in Fig. 8.

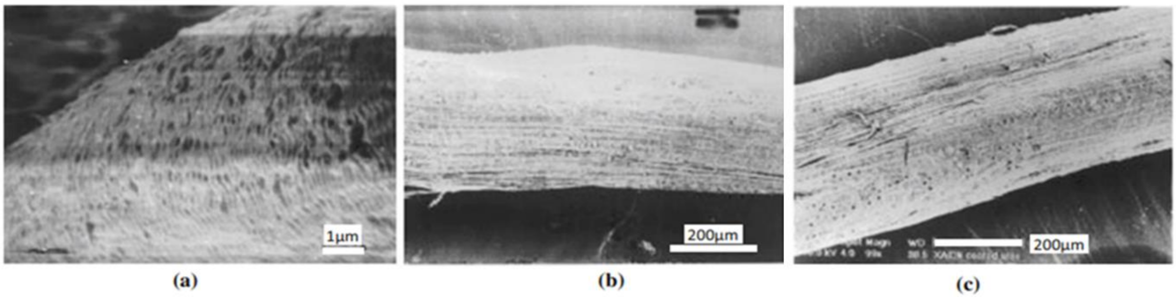
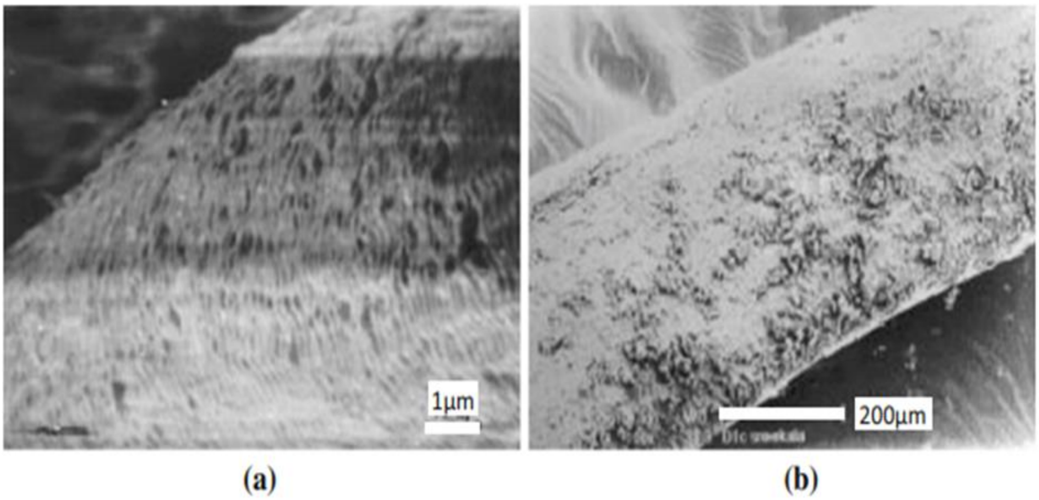


Fig. 8. SEM micrographs of the surfaces of oil palm fibers, (a) untreated, (b) treated acrylate, and (c) treated acrylonitrile graft [36].

2.8. *Treatment with isocyanate*

One of the most difficult issues in natural fiber-reinforced composites is generating strong fiber-matrix bonding, as natural fibers' hydrophilic nature can cause weak bonding with hydrophobic polymer matrices. This inadequate fiber-matrix interaction might result in insufficient load transfer, decreased mechanical strength, and moisture absorption, compromising the composite's performance. Isocyanate treatment is a chemically modified process that is specifically intended to address these concerns. The reactive -NCO groups in isocyanates can generate urethane linkages (-NHCOO-) with hydroxyl groups on cellulose fibers, effectively attaching the isocyanates to the fiber surface. This treatment improves the fiber's interaction with polymer matrices, primarily those based on polyester or polyurethane, by generating functional structures which encourage better adhesive properties. Furthermore, the creation of a urethane layer makes the fiber surface more hydrophobic, improving fiber-matrix adhesion while also increasing the composite's resistance to water uptake and dimensional stability [37].

As discussed, surface modification of fibers using isocyanates provides multiple benefits for the creation of robust and long-lasting composites. The urethane groups' formation of chemical connections between the fibers and matrix boosts the composites' mechanical strength, including their elastic modulus and tensile strength. Furthermore, this treatment's hydrophobic qualities lessen the effects of moisture on fiber deterioration, which is crucial for applications in moist or humid conditions [38]. Isocyanate treatment needs to be carefully controlled, though, to avoid overreactions that could break the fiber or make it brittle. To balance enhancing mechanical qualities with maintaining the original fiber structure, it is therefore essential to optimize reaction conditions, such as isocyanate percentage and reaction duration. Through this process, natural fibers become more appropriate for usage in a range of industrial applications, such as consumer goods, automotive, and construction, where high-performance, long-lasting composite materials are needed. Strong covalent bonds between the fiber and matrix are created during isocyanate treatment, which is known to enhance the bonding qualities between the two. Additionally, this strengthens the fiber's ability to withstand moisture. By reacting with the OH groups of the cellulose and lignin component of the fiber, the functional groups (-N=C=O) of isocyanate form strong chemical bonds and moisture resistance [39]. The surface behavior of palm fibers treated with isocyanate and those that are not are displayed in Fig. 9.



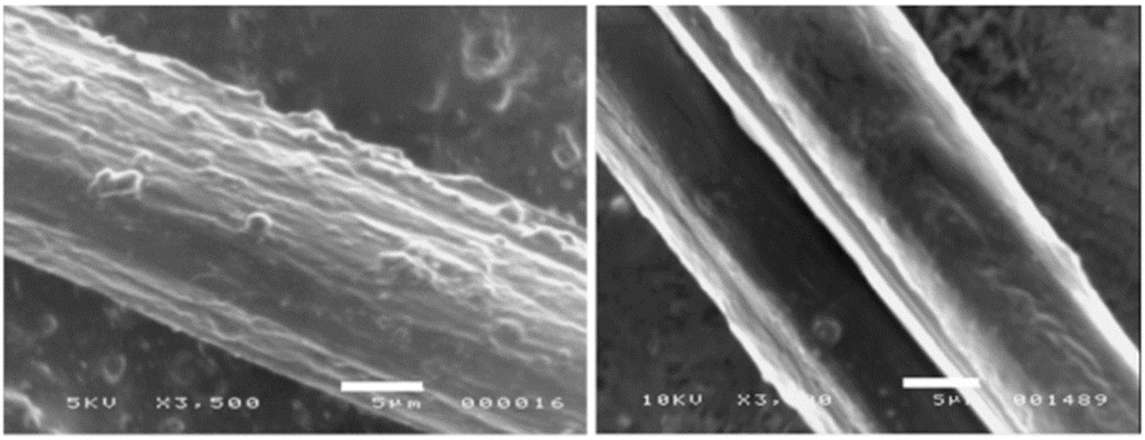
(a) (b)

Fig. 9. SEM micrographs of the surface of palm fiber, (a) untreated, and (b) isocyanate treated [36]

2.9. Treatment with stearic acid

Applying a fatty acid layer to natural fibers with stearic acid treatment enhances the fibers' hydrophobic qualities. Through hydrogen bonding or more potent chemical processes like esterification, stearic acid—a saturated fatty acid with a lengthy carbon chain—can interact with the hydroxyl groups on the exterior of cellulose fibers. Following this treatment, the fiber's surface structure changes significantly as a hydrophobic layer made of long stearic acid alkyl chains forms. This layer boosts the fiber's resistance to moisture and decreases its attraction for water, making the fiber more stable in damp environments [40]. Stearic acid treatment smoothes and coats the fiber surface morphologically, improving its resilience to chemical and physical deterioration.

Stearic acid modification of fiber surfaces has been discussed as having various significant advantages, particularly in the creation of polymer composites. Natural fibers modified by stearic acid exhibit increased interaction with hydrophobic polymer matrices like polypropylene or polyethylene due to their more hydrophobic fiber surface. In the end, this increases the mechanical qualities of the composite, including its tensile strength and moisture resistance, by enhancing the compatibility between the fiber and the matrix. Additionally, in applications where resistance to damp or corrosive conditions is required, the stearic acid coating serves as a barrier towards water penetration, lowering the risk of fiber damage [41,42]. Stearic acid treatment can improve the suitability of natural fibers for use in composite materials that need to have strong hydrophobic and durable qualities.



(a) (b)

Fig. 10. SEM micrographs of the surface of hemp fibers (a) untreated, (b) treated with stearic acid [43].

Since the hydrophilic OH groups of the natural fibers react with the carboxyl groups of stearic acid, which are derived from ethyl alcohol solution, non-crystalline components of the fiber structure are eliminated, increasing the water resistance of the natural fibers treated with stearic acid solution. This results in increased fibrillation disrupting the fiber bonds and improving the dispersion of the fibers inside the matrix. Fig. 10 shows how the surfaces of ramie fibers treated with stearic acid and those that are not changed.

2.10. Treatment with oleoyl chloride

One chemical technique for altering the surface structure of natural fibers to enhance their hydrophobic qualities and interactions with polymer matrix is the use of oleoyl chloride. When long-chain fatty acids found in oleoyl chloride react with hydroxyl groups on the fiber surface, esters are produced and nonpolar alkyl chains are introduced. In nonpolar polymer constructions like polypropylene, this procedure boosts the fiber's hydrophobic characteristics and decreases its affinity for water. After this treatment, the fibers' surface structure smoothes out, exhibiting increased hydrophobic qualities and the capacity to interface with nonpolar polymers [8].

These surface structure modifications also help to improve the compatibility of the fiber with the polymer matrix, which produces composites with improved mechanical qualities. Additionally, a more hydrophobic fiber surface can greatly minimize the amount of water absorbed by the fibers, improving the composites' resilience to environmental deterioration and dimensional stability. In addition to improving fiber distribution in the polymer matrix, the use of oleoyl chloride as a modifying agent also lessens the creation of cavities and other flaws that could damage the composite. Because of this, natural fibers are now better suited for usage in high-performance composite that are utilized in many different industries, such as consumer products, construction, and automotive. The OH groups on the fibers react with a fatty acid derivative called oleoyl chloride, which enhances the wettability and adhesive characteristics of the fibers. The fibers are made more hydrophobic by eliminating the hydrophilic OH groups from their outside, which results in this enhancement [11]. The surface appearance of hemp fiber both before and after it was treated with oleoyl chloride in pyridine for a duration of 24 hours [44], as shown in Fig. 11.

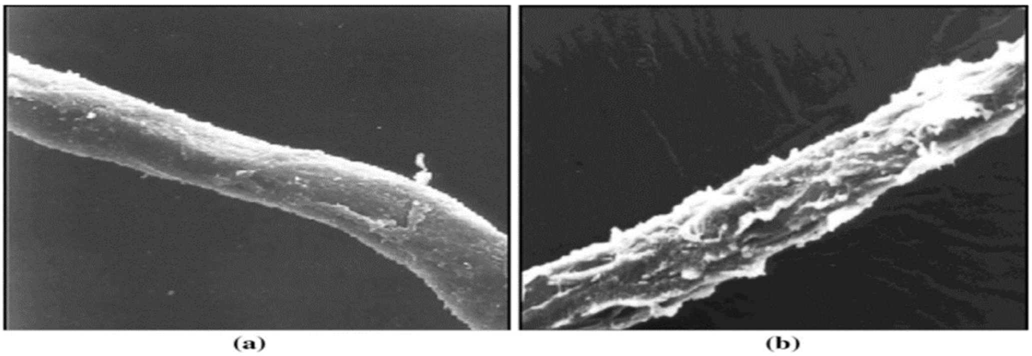


Fig. 11. SEM micrographs of the surface of hemp fiber (a) untreated, (b) treated with oleoyl chloride in pyridine for 24 hours [44].

2.11. Treatment with permanganate

An oxidation technique used to alter the surface structure of natural fibers is permanganate treatment, such as using potassium permanganate ($KMnO_4$). In this procedure, hemicellulose and lignin on the fiber surface are oxidized by permanganate, which functions as an oxidizing agent. This is a strong oxidative reaction. After this treatment, the fiber surface is cleaner, has less lignin, and has more hydroxyl and carbonyl groups that were created as a result of oxidation. Following permanganate treatment, the fiber's surface structure becomes rougher and more chemically reactive, which enhances its capacity to interact with the polymer matrix through both chemical and physical bonding. $KMnO_4$ interacts with natural fibers through an oxidative process. $KMnO_4$ is an oxidizing compound that targets hydroxyl groups (-OH) in natural fibers' cellulose structure. This contact alters the fiber surface, frequently producing carbonyl groups (-C=O) or carboxyl groups (-COOH) by partial oxidation. The process reduces Mn^{7+} from $KMnO_4$ to Mn^{2+}, producing manganese dioxide (MnO_2). The oxidation process adds polar groups to the fiber surface, increasing surface roughness and improving interfacial adhesion between the fiber and polymer matrix in composite materials. However, if not managed appropriately, excessive oxidation can weaken the fiber structure, resulting in a loss of mechanical strength [45].

Within the framework of composite applications, these surface structure modifications offer some advantages. Tensile strength and elastic modulus of the composite can be enhanced by a more reactive and rougher surface, which promotes better adhesion between the fibers and the matrix. Nevertheless, if improperly managed, this heightened reactivity may also raise the possibility of oxidative fiber breakdown, diminishing the composite's long-term endurance. To balance enhanced adhesion and fiber stability, it is crucial to optimize the permanganate treatment conditions. This processing improves the suitability of natural fibers for use in polymer composites when improved fiber-matrix interactions are needed, particularly in structural applications where good mechanical performance is required. Applying permanganate to natural fibers improves their chemical interlocking at the matrix-natural fiber interface [46]. This method increases the thermal stability of the fiber by allowing potassium permanganate (KMnO4) to react with the OH groups of the cellulose and lignin elements of the fiber to improve adhesion between the fiber and the matrix. Fig. 12 depicts the surface characteristics of untreated and permanganate-treated hemp fibers.

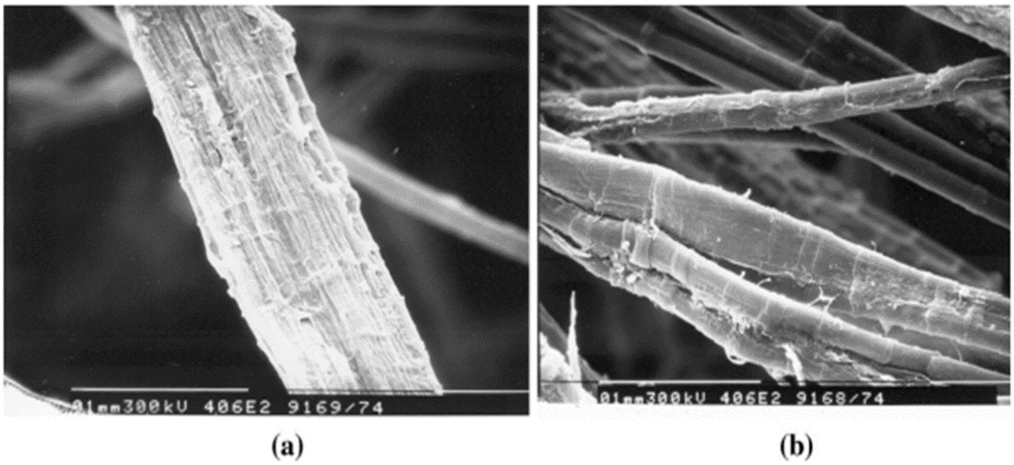


Fig. 12. SEM micrographs of the surface of flax fibers, (a) untreated and (b) permanganate treated [47].

3. Surface structure of natural fibers after being treated with fungi

Fungal treatment of natural fibers employs specialized enzymes—lignin, cellulose, and hemicellulose—that target the principal fiber elements: lignin, cellulose, and hemicellulose, respectively. Lignin is essential for breaking down lignin, a complex and hard polymer found in plant fibers that offers structural integrity and resists degradation. Lignin catalyzes lignin's oxidative breakdown by cleaving its phenol units, resulting in the network's depolymerization. This drop in lignin content exposes the cellulose microstructures beneath, resulting in a porous and rougher fiber surface. The removal of lignin not only improves the interaction between the fiber and polymer matrix in composites, but it also increases the accessibility of other enzymatic treatments, causing the fiber surface more reactive for later processing. Cellulase and hemicellulase are enzymes that work on natural fibers' secondary components. Cellulase breaks down β-1,4-glycosidic connections between glucose units in cellulose, the most prevalent polymer found in natural fibers. This partial hydrolysis of cellulose opens up the crystalline structure of the fibers, exposing additional hydroxyl groups that may form bonds with polymer matrices in composites applications, improving fiber-matrix adhesion. Hemicellulase, on the opposite hand, destroys hemicellulose, a branching polysaccharide that surrounds cellulose and serves as a filler material between it and lignin. Hemicellulase breaks down the β-1,4-linkages in hemicellulose, decreasing its presence in the fiber. Because hemicellulose is more hydrophilic and porous than cellulose, degradation reduces the fiber's susceptibility to moisture absorption, enhancing dimensional stability and lowering the chance of fiber swelling in composite products. Together, these enzymatic processes change the surface properties of natural fibers, increasing their compatibility with polymer matrices and boosting mechanical qualities and endurance in composites [8].

There are many benefits and difficulties to using fungi to change the surface structure of natural fibers. Given that they don't use dangerous chemicals and can operate at comparatively moderate temperatures and pressures, fungal biochemical processes are, on the one hand, environmentally beneficial. As a result, they can be used to produce composite materials that are environmentally friendly and sustainable. However, managing these deterioration processes is a significant task because too much degradation can destroy the fibers' mechanical integrity and make the composites that result weaker. To obtain the best surface modification without causing undue damage to the fibers, it is crucial to regulate the fungi's development parameters, including the incubation period, temperature, and humidity. Fungal fiber treatment is a more sustainable modification method than standard chemical procedures, and it can enhance the adherence and compatibility of fibers in composites under the correct conditions. Using certain enzymes, the novel biological therapy known as "fungal treatment" eliminates non-cellulosic elements from the fiber surface [48]. The lignin peroxidase enzyme in white rot fungi combines with extracellular oxidase enzymes to extract lignin from the fiber. Production of hyphae results in fine pores being created on the fiber surface by improved interaction between the fiber and the matrix. A surface comparison of treated and untreated ramie fibers is presented in Fig. 13. Fungal treatment offers an environmentally friendly alternative for surface modification of natural fibers by using enzymes to degrade lignin, cellulose, and hemicellulose, making it less harmful but slower and less controllable compared to chemical methods, which are highly effective at improving fiber-matrix bonding through aggressive reactions but pose significant environmental concerns due to hazardous waste. Physical treatments like plasma or mechanical abrasion increase surface roughness without chemicals, offering cleaner processes but with limited effectiveness in altering deeper fiber properties. While fungal treatment is ideal for sustainable applications, chemical and physical methods are more suitable for industries needing faster processing and higher performance.

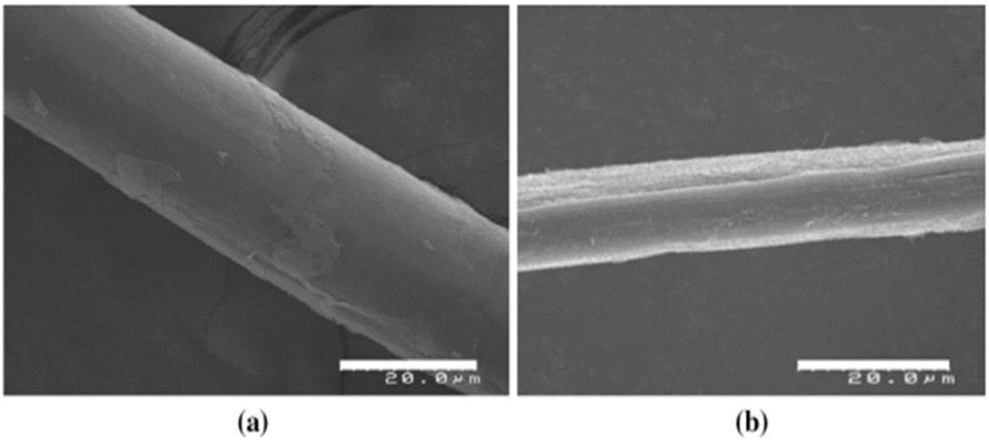


Fig. 13. SEM micrographs of the hemp fiber surface, (a) untreated, and (b) after fungal treatment [49]

4. Conclusions

This analysis of the literature demonstrates that natural fibers' surface structure and functional qualities can be greatly impacted by chemical treatments. Several techniques are available to enhance the strength, toughness, and compatibility of fibers with polymer matrix, including acetylation, benzoylation, silane agents, and peroxides. Improved fiber density, stability, and heat resistance have been demonstrated by treatments such acrylation, acrylonitrile grafting, sodium chlorite, and maleating agent coupling. Moreover, stearic acid, isocyanates, oleoyl chloride, and permanganate have all helped to increase hydrophobicity and heat stability. Studies on fungal biological treatments provide an eco-friendly substitute for fiber modification and could lead to more sustainable processing. Changes in the fiber surface structure have an impact on how well fiber-based materials function in a variety of industrial applications, according to microscopic inspection and chemical characterization of different treatments. Ultimately, a better comprehension of the effects of these different chemical treatments will offer helpful direction for the creation and use of natural fiber-based materials that are more efficient and sustainable. The results of this research underscore how crucial it is to choose the appropriate processing technique to enhance natural fiber quality and performance in accordance with particular application requirements. Modifying fiber surfaces with acetylation and $KMnO_4$ can enhance natural fiber composites' adhesion, moisture resistance, and mechanical qualities. Further research could focus on improving modification methods to strike a compromise between boosting mechanical qualities and reducing fiber deterioration, as well as investigating sustainable modification processes. Furthermore, research can lead to the creation of multifunctional composites with unique properties such as antibacterial, flame retardant, or increased conductivity, as well as the tailoring of fiber surface modifications for specific applications in diverse industries.

CRediT authorship contribution statement

Sari, N.H.: Conceptualization, Writing – original draft, review & editing, Supervision, Funding acquisition. Suteja, S and Sutaryono, Y.A. Moonish Ali Khan: Resources, Investigation. Data curation, Visualization.

Declaration of Competing Interest

The authors declare that they have no known competing interests that could have influenced the literature reported in this paper.

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