Int. J. Curr. Res. Chem. Pharm. Sci. (2022). 9(6): 78-88

INTERNATIONAL JOURNAL OF CURRENT RESEARCH IN CHEMISTRY AND PHARMACEUTICAL SCIENCES (p-ISSN: 2348-5213: e-ISSN: 2348-5221)

www.ijcrcps.com

(A Peer Reviewed, Referred, Indexed and Open Access Journal) DOI: 10.22192/ijcrcps Coden: IJCROO(USA) Volume 9, Issue 6 - 2022

Review Article



DOI: http://dx.doi.org/10.22192/ijcrcps.2022.09.06.010

An Overview on Recent Trends in the Modification of Surface of Natural Fibers

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Abstract

A lot of academics and researchers are looking into new possibilities in the area of natural fiber-based green composites since natural fibres have good qualities including lightweight, accessibility, sustainability, and propensity for being environmentally benign. Natural fibres also have additional benefits that draw interest from the scientific community and businesspeople, such as affordability, diversity, and environmentally friendly nature. Natural fibres are hydrophilic by nature due to their lignocellulosic composition, and one of the main problems that can prevent the commercialization of their final composites is that they adhere poorly to hydrophobic polymer matrices. These natural fibres and their reinforced polymer composites have undergone extensive research on various treatment procedures to obtain the improved qualities. This article reviews the impact of various chemical modifications of fibre surfaces on various mechanical characteristics of composites made of natural fibres. A thorough list of the current treatment approaches, including more uncommon ones like enzymatic, radiation, and ionic liquid treatment, as well as more conventional ones like alkali treatment, silanization, and graft copolymerization, has been discussed. Recent developments in the field of natural fibre surface treatment have been highlighted in order to provide the best possible green composites.

Keywords: Fiber, Cellulose, Modification, Silane, Bleaching

1. Introduction

Numerous studies have been done recently to look into the idea of employing natural fibres instead of synthetic ones in fibre reinforced composites. Natural fiber-reinforced composites are gaining popularity, most likely as a result of its accessibility, affordability, sustainability, and environmental friendliness¹. Researchers and businesspeople are drawn to natural fibres because of their versatility, affordability, and capacity to be renewed. With a compound annual growth rate of 12.3 percent, the market for natural fiber-reinforced composites is expected to reach 5.83 billion by 2019². Natural fibres are hydrophilic by nature because they are lignocellulosic, and their poor adhesion with hydrophobic polymer matrix is one of the major

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issues that can thwart the commercialization of their finished composites¹. Table 1 shows the structural composition of natural fibres^{3, 4}. Each component's properties contribute to the overall properties of the fibre. The least resistant

component of the fibre, hemicellulose, is in responsible of biodegradation, moisture absorption, and thermal decomposition, whereas lignin is thermally constant but causes UV degradation¹².

Name of the fibres	Cellulose (wt%)	Lignin (wt%)	Hemicellulose (wt%)	Pectin (wt%)	Wax (wt%)	Micro- fibrillar/ spiral angle (°)	Moisture content (wt%)
Bast Fiber							
Jute	61-71.5	12-13	13.6-20.4	0.2	0.5	8.0	12.6
Flax	71	2.2	18.6-20.6	2.3	1.7	10.0	10.0
Hemp	70.2- 74.4	3.7-5.7	17.9-22.4	0.9	0.8	6.2	10.8
Ramie	68.6- 76.2	0.6-0.7	13.1-16.7	1.9	0.3	7.5	8.0
Leaf Fiber							
Sisal	67-78	8.0-11.0	10.0-14.2	10.0	2.0	20.0	11.0
Pineapple Leaf Fiber	70-82	5-12	-	-	-	14.0	11.8
Cotton Seed Fiber	82.7	0.7-1.6	5.7	-	0.6	-	33-34

Table 1. Components of Natural Fibers

Cellulose is the primary framework component of the natural fibre structure. Natural lignocellulosic fibres have several benefits over conventional reinforcing materials, such as glass fibres, talc, and mica, including adequate specific strength and other mechanical properties, low cost, low density, non-abrasivity, good thermal properties, increased energy recovery, and biodegradability⁵.

Cellulose is the biopolymer that is most prevalent on Earth. Over 10¹² tonnes of cellulose are produced annually by plants⁶. Compared to 50% in cotton and 90% in cotton, plants contain just around 33% of the cellulose that is present in wood. Figure 1 shows the chemical structure of different components of natural fibers^{15, 16}. The majority of the cellulose is used as a raw material in the manufacture of paper. This is equivalent to roughly 108 tonnes of pulp produced per year⁷. Each year, only 4 million tonnes of material are used for additional chemical processing. This clearly demonstrates that relatively little cellulose is utilised in the creation of common materials and chemicals⁸.

Natural fibres have a high moisture absorption capacity due to their hydrophilicity, absorbency and sluggish attachment to hydrophobic matrices. Furthermore, because most natural fibres degrade temperatures $(200^{\circ}C)$, at low they are incompatible with thermosets that cure at high temperatures. This also limits natural fibre composites to relatively low temperature applications. Other difficulties include: high variability of mechanical properties^{9, 10}, lower elongation¹¹, lower ultimate strength¹¹, bubbles in the product¹¹, poor resistance to weathering of natural fiber.

Extensive research on various treatment methodologies has been conducted on these natural fibres and their reinforced polymer composites in order to improve their properties. To achieve the excellent results, various chemical, physical, and biochemical methods have been proposed. Cellulosic fibres at the micro- and nanoscale are preferred to replace synthetic fibres as reinforcement in sustainable green products.

2. Methods of Modification of Natural Fibers

2.1. Conventional Methods

For several conventional reasons, methods have been devised to alter the surface of textiles. Cotton's surface lustre can be changed via the mercerization process. For 1-3 minutes at low temperatures, cotton cloth is treated with potent caustic soda solutions under tension, and then washed while still under tension. The fabric will maintain its original proportions if the cotton fabric is kept under tension in the caustic solution. The fibres become more rounded in the cross section, which improves the lustre of the cloth by reflecting light. Unmercerized fibres have a relatively large lumen, a central canal running along the fibre axis. The lumen scatters light at the lumen-cellulose contact because it has a different refractive index from cellulose and is larger than cellulose. In mercerizing, the lumen nearly completely vanishes, and the convolutions in the fibres lessen. The fiber's cross section transforms from about kidney-shaped to round. Along with a decrease in the amount of crystalline material, mercerized cotton also experiences a change in its crystal structure. Along with a decrease in the amount of crystalline material, mercerized cotton also experiences a change in its crystal structure. Applying desizing fiber structure and composition characteristics of fibre spinning and yarn composition Yarn attributes Structure and fabric construction qualities of grey fabric Decorative touches and colour Fabric characteristics for final usage Enzyme to remove the starch added during the twist preparation method, detergent to clean the textile substrate of all foreign substances like grease, wax, and spinning lubricants, and an oxidizer during the bleaching process to get rid of any natural colourants, water-borne stains, and oil-borne soils.

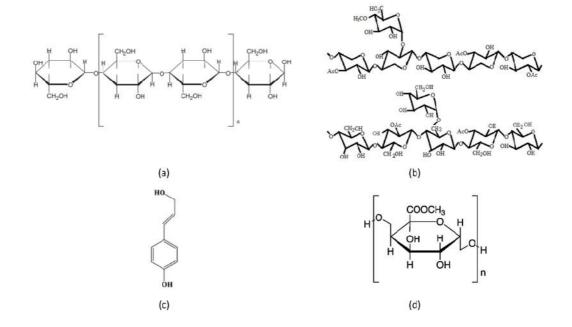


Figure 1. Chemical structure of (a) cellulose; (b) hemicellulose; (c) lignin; (d) pectin

The cost of wastewater treatment has increased significantly in recent years due to consumer demand and environmental legislation. It is necessary to develop ecologically safe, effluentfree procedures with lower costs because legislation to tighten aqueous-based emissions is predicted to increase. The textile sector must now discover alternatives to chemically intensive procedures in order to comply with environmental legislation.

2.2. Physical Methods

Plasma treatment is a physical technique that has been used successfully to modify the surface of various natural fibres. After plasma treatment, the mechanical properties of natural fibres were found to improve significantly¹³. Plasma surface modification of textile goods made from natural fibres has various benefits, including effective adjustment of textile surface qualities and decreased use of environmentally toxic chemicals. Surface modification of a variety of natural textiles, including linen, wool, cotton, silk, and synthetic fibres, has been achieved using low temperature plasma. Following are some benefits of plasma cleaning over traditional finishing techniques¹⁴:

- There is no need for additional drying after the dry modification process.
- The dry production method is safer and more reliable.
- Very low chemical requirement
- The original properties of fiber remains undistorted
- Plasma treatment makes it simple to change materials that are hard to modify
- It generates no waste, and processing time is quite quick.

The plasma gas (or gases) utilised, along with other treatment variables, affect the characteristics of the substrates. Plasma treatment allows for the creation of both smoother and coarser surfaces. By adding different functional groups, it is possible to alter not only the surface shape but also the chemical make-up of the treated substrate surface. In subsequent textile processing techniques, the processed fibre and fabric surface's chemical transformation is significant. There are many advantages to using plasma technique as a pre-treatment to traditional finishing, dyeing, and other production processes, as well as being an environmentally friendly way to do so, despite the complexity of the surface property variations produced by plasma treatment.

Plasma polymerization is a technique that can be used to graft a polymer onto the surface of a natural fibre. This process involves activating the monomer that needs to be grafted first by gaining energy from the plasma through an inelastic collision. These small, energised molecules join again to produce a larger molecule. Finally, polymers are grafted onto the natural fiber's surface. And the end product is known as plasma polymer¹⁷. In many instances, polymers produced using plasma polymerization differs from those produced through traditional polymerization in terms of their chemical composition, physical and chemical characteristics.

One of the most beneficial and efficient techniques for surface modification of natural fiber, is plasma implantation, a treatment method involves inserting functional that groups containing oxygen on the treated substrate surface. Although oxygen plasma is typically employed. the polymer surface becomes hydrophilic when plasmas of other chemicals such CO₂, CO, NO₂ and NO are used. Plasma activates gas molecules like oxygen and nitrogen. Various functional groups such as -COOH, -OH, -CO, -NH₂ and amido are introduced on to the surface of natural fiber, when these activated oxygen and nitrogen species are interacted with fiber surface and finally this implantation reaction leads to significant change in the surface properties of the polymer from hydrophobic to hydrophilic. In the plasma, radical species may make a significant contribution more to the implantation reaction than ionic species. Aside from oxygen radicals, chlorine components such as CCl4 can also increase hydrophilicity¹⁷.

During the implantation process, hydrogen will be extracted from polymer chains to form radicals at the centre of the polymer chains, which will then combine with normal species.

Plasma-aided graft-copolymerisation can occur via radical formation on the polymer surface followed by contact with monomer or via direct grafting of the polymer with monomers under' monomer' plasma conditions¹⁸. In the first situation, plasma treatment results in the formation of free radicals on the polymer surface. These radicals have the ability to start grafting either directly or via oxidising with an oxidising gas to produce peroxide or hydroperoxides. When the monomer species are present, these activated peroxides will likewise start $grafting^{20}$. The second situation includes the employment of gaseous monomers in the working gas combination to expose plasma and monomers simultaneously²¹. Due to the wide variety of chemical compounds that can be utilised as monomers, the variable thickness of the monomer layers, and the minimal amount of destruction, both of these approaches have demonstrated advantages numerous over conventional grafting²².

2.3. Chemical Methods of Surface Modification

Utilizing a hydroxyl group, which can alter the composition of the material by adding new components that can interact with the matrix, chemical treatment is used to modify and activate the fibre structure²³.Natural fibres can be treated with various chemicals, such as alkali, silane, water repellents, peroxides, permanganates, etc. to improve their physical, mechanical, and chemical properties²⁴. By altering their crystalline structure and removing weak elements like hemicelluloses and lignin from the fibre structure, some of these chemical treatments have been found to significantly improve the mechanical properties of natural fibres²⁵.

2.3.1. Alkali Treatment

Effective mercerization or alkali treatment is the most popular and affordable chemical method for surface modification of fiber. By breaking the internal hydrogen bonds, the treatment increases surface roughness and improves the mechanical properties of the fibre by altering surface topography, crystallinity, unit cell structure, moisture absorption, and orientation of fibrils²⁷. Lignin, wax, and oils that cover the fibre cell wall's outer surface will be partially removed during the process, along with hemicellulose, which will cause the cellulose to break down and reveal short-length crystallites²⁸. The increase in tensile strength is clearly connected with the amount of alkali used in the treatment. demonstrating that using a high concentration of alkali more than 10% weakened or damaged the fibre and decreased its tensile strength²⁶. In comparison to untreated fibres, a mild alkali addition of 6 to 9 percent will boost the fibres' tensile strength by about 30%. The relationship between sodium hydroxide solution and the mechanical characteristics of both PF and PFreinforced composites was examined in investigations by several researchers²⁹⁻³¹. Their findings demonstrated that the alkali treatment, which generates a slightly rough texture by removing natural and manufactured impurities, has a direct impact on the thermal and mechanical properties of plant fiber. Research has shown that composites with alkali-treated fibre bundles have better mechanical properties than composites without the treatment³². However, carrying out the alkali treatment of PF at high temperatures is the only way to further boost its efficacy, as the heat energy will catalyse the breakdown of more hydrogen bonds inside the fibrils³³. The following reaction occurs in the alkali treatment.

Fiber-OH + NaOH Fiber-O-Na+ + H_2O

2.3.2. Coupling agents

The design of coupling agents to boost the mechanical durability and strength of the fibre matrix and to fortify the interfacial bonding required for associated adhesion. The coupling agents work well with the fibre matrix to lessen water absorption, stop leaching, and improve the wettability of fibres by the polymer chains³⁴. The surface modification procedures carried out on natural fiber using coupling agents like acetylation, silanes, and graft copolymerization aim to improve the chemical bonding of the oxide groups on the surface of fiber with the polymer molecules that connect the hydrophilic end of fibres and hydrophobic group of polymers for excellent composite mechanical properties³⁵. According to the fibre, matrix, and type of surface treatment used, the chemical treatment brought on by coupling agents significantly improves the characteristic values of composites³⁶.

2.3.3. Silane

Silane coupling agents may reduce the number of hydroxyl groups in the fiber matrix interface. A hydrolyzable alkoxy group causes the production of silanols when there is moisture present⁴³. The silanols react with the fiber's hydroxyl group to produce strong, chemisorbed connections to the cell wall. In order to change the natural fiber-polymer matrix interface and boost the interfacial strength, it has been discovered that silane coupling agents are useful. In the figure 2, silane interactions with cellulosic fibre are depicted.

2.3.4. Acetylation/Acylation

For the purpose of plasticizing natural fiber, the two types of acylation or esterification are acetylation (using acetate) and valerylation (using valerate). When a fibre is acetylated, an acetyl group combines with its hydrophilic hydroxyl groups to produce esterification, which lessens the fiber's hydrophilicity by absorbing moisture from the fibre³⁷. As a result of the acetylation process, the fiber's dimensional stability and ability to disperse into polymeric matrices were both

improved, which increased the fiber's hydrophobicity. Acetyl groups were used in place of the fiber's hydroxyl groups. In natural fiber composites, alkaline treatment was typically performed before to acetylation and has been reported to decrease impact strength and stiffness. increase interfacial bonding, as well as the thermal stability, dimensional stability, and fungal resistant. The amount of fibre in the composite determines the potential amount of coupling agents as well as its mechanical and other physical properties³⁸.

2.3.5. Graft Copolymerization

Graft copolymerization is a well-known crosslinking method that has been in use since 1943 to chemically increase the compatibility of fiber surface or wood with a suitable solution by forming free radicals on the cellulose molecules by reacting through chosen ions with hydrophobic matrices³⁹. A variety of vinyl monomers, methyl methacrylate, acrylonitrile, and are grafted using this technique⁴⁰. By using this technique, functional groups that can chemically react with cellulose or other natural fibre components are grafted into related polymers or polymers that resemble a certain matrix⁴¹. As a result, the grafted systems can serve as a conduit to reduce polarity mismatches between the hydrophilic end of fibres and hydrophobic matrices. Isocyanates, methyl groups, triazines, maleic anhydride, benzoylation, and organosilanes are among the functional groups now in use. However, due to price, effectiveness, and commercial availability, maleic anhydride is the best functional group for compatibility through graft copolymerization 42 .

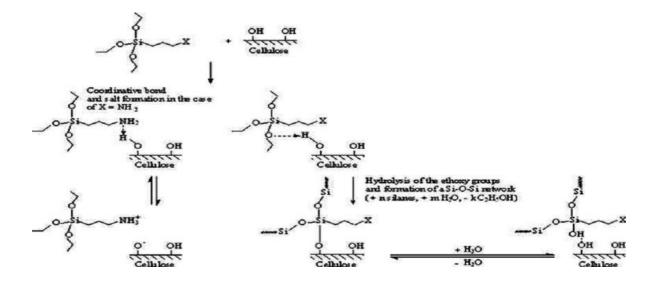


Figure 2. Schematic representation of interaction between silane and cellulose⁴⁴

2.3.6. Bleaching

The look of bast fibres can be improved by bleaching processes such peroxide bleaching, alkali, biobleaching and enzyme treatments. By dissolving and removing lignin and hemicellulose from the matrix enclosing the cellulose microfibrils, bleaching was used to isolate individual microfibrils⁴⁵. Most of the plant fiber used for textile fabrics is bleached with hydrogen peroxide, which is a environmentally safe bleaching process. When employing hydrogen peroxide, the temperature and pH must be kept under control because excessive temperatures and alkalinity will harm the fibres. The effect of bleaching on plant fiber can be observed visually as a change in look and other aesthetic qualities, but it can also result in an intentional reduction of fibre tensile strength⁴⁶ because lignin serves as a cement for the fibres, which lowers the tensile strength of the composite. Reductive and oxidative bleaching are the two main categories used to characterize bleaching.

Reductive bleaching, a brief procedure using sodium dithionite and the conversion of chromophores (coloured fibres) to leucochromophores, is carried out on protein fibres with high cellulose content. In general it is stable, however in hot water and acid solutions it will disintegrate. Reduction bleaching is rarely investigated for modifying the surface of PF for composite applications because to considerable less durability⁴².

This makes oxidative bleaching, which uses hydrogen peroxide, sodium hypochlorite or sodium chlorite, a common treatment approach. The OH groups in lignin were attacked by sodium hypochlorite to produce aldehyde groups (CHO) that can diminish the amount of lignin present, but pre-existing aldehyde groups will cause the cellulose in natural fiber to degrade. In contrast to sodium hypochlorite and hydrogen peroxide, sodium chlorite (NaClO₂) bleaching can remove lignin and pectin with a little quantity of cellulose degradation at an affordable price. Although the chemical cost of utilising H_2O_2 to bleach is higher many other bleaching agents with than comparable capabilities, it is progressively replacing other oxidative bleaching techniques due to its environmental friendliness47.

2.4. Biological Surface Modification of Natural Fibers

Enzyme or biological modification is an efficient process for treating the surface of fibres by removing lignin and hemicellulose, and it uses less energy. The pectinase glue holding the fibre bundles together can be damaged or removed by the fungus or bacteria, releasing the cellulose fibres. The treatment increased the surface hydrophilic contact between the fiber matrix, which in turn enhanced the composites' mechanical characteristics. It was discovered that utilising enzymes to separate hemp fibre bundles into individual bundles increased the crystallinity and thermal characteristics. Additionally, it affects the fibres' structure, chemical structure, final fibre quality, and other characteristics⁴⁸. Enzyme treatment is becoming more and more common due to its benefits for the environment⁴⁹, and the reaction it catalyses is quite specialised and focuses on the specific feature required. The $environment^{50}$ will be impacted by the discharge, although it can be recycled after each usage 51 .

Conclusions

Because of its accessibility, environmental friendliness, and consistent performance of a wide range of fibres, plant fiber is seeing an increase in demand for biocomposites. In order to fully capitalise on the benefits of natural fibres in composite materials and to successfully employ them in a variety of industrial applications, surface modification of natural fibres has been the focus of numerous studies. The literature that is now available, however, discusses a wide range of approaches. including multiple physical. chemical, and biological approaches. Research in this field is highly diversified. The modification of plant fiber composites benefits from the good compatibility and interface bonding provided by physicochemical surface treatments. The reinforcing fibre is the main contributor to the mechanical properties of the composite; hence efforts must be made to identify the best physicochemical for combination surface modification natural fiber.

To choose the best treatment for the problem, it is crucial to thoroughly analyse the volume of existing research in order to understand the benefits and drawbacks of each treatment.

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How to cite this article:

Lokesh Kumar. (2022). An Overview on Recent Trends in the Modification of Surface of Natural Fibers. Int. J. Curr. Res. Chem. Pharm. Sci. 9(6): 78-88. DOI: http://dx.doi.org/10.22192/ijcrcps.2022.09.06.010