

**ПОЛИМЕРНЫЕ БИОКОМПОЗИТЫ НА ОСНОВЕ АГРООТХОДОВ: ЧАСТЬ I. ИСТОЧНИК,
КЛАССИФИКАЦИЯ, ХИМИЧЕСКИЙ СОСТАВ
И МЕТОДЫ ОБРАБОТКИ ЛИГНОЦЕЛЛЮЛОЗНЫХ ПРИРОДНЫХ ВОЛОКОН**

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В настоящее время истощение природных ресурсов и проблемы окружающей среды вызвали огромный интерес в поиске устойчивой альтернативы для создания новых материалов, которые являются экологически чистыми. В последние годы многие исследователи сосредоточились на исследованиях, связанных с сельскохозяйственными отходами, для решения экологических проблем, связанных с удалением агроотходов. Благодаря своей ежегодной возобновляемости и способности к биологическому разложению, агроотходы за последние два десятилетия стали экологически чистой альтернативой синтетическим волокнам. В связи с этим, в данном обзоре дано общее представление о новых лигноцеллюлозных полимерных композиционных материалах, в которых в качестве наполнителей и армирующих добавок используются различные природные волокна. А также приведены источники, классификация и химический состав лигноцеллюлозных натуральных волокон. Натуральные волокна включают функциональную группу, названную гидроксильной группой, которая делает волокна гидрофильными. Гидрофильные натуральные волокна и гидрофобная полимерная матрица приводят к несовместимости и слабой межфазной связи между ними. Для устранения проблемы несовместимости растительных волокон с неполярными полимерными матрицами в статье рассмотрены различные методы обработки поверхности натурального волокна, включая физическую, химическую и биологическую обработку. С этой точки зрения, по нашему мнению, данные, представленные в этой статье, могут быть использованы в качестве базы данных для дальнейшего изучения агроотходов в качестве наполнителей или армирующих добавок в полимерных композитах с целью ускорения развития и стимулирования коммерческого производства этих новых материалов.

Ключевые слова: биоккомпозит, экологически чистый, агроотход, натуральное волокно, модификация поверхности

**POLYMER BIOCOPPOSITES BASED ON AGRO WASTE: PART I. SOURCE, CLASSIFICATION,
CHEMICAL COMPOSITION AND TREATMENT METHODS
OF LIGNOCELLULOSIC NATURAL FIBERS**

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At present, the depletion of natural resources and environmental problems have generated great interest in finding a sustainable alternative to create new materials that are environmentally friendly. In recent years, many researchers have focused on investigations related to agricultural waste products to solve environmental problems associated with the disposal of agro waste. Thanks to their annual renewability and biodegradability, over the past two decades, agricultural waste has become an environmentally friendly alternative to synthetic fibers. In this regard, this review gives a general idea of new lignocellulosic polymer composite materials in which various natural fibers are used as fillers and reinforcing additives. The sources, classification and chemical composition of lignocellulosic natural fibers have been also given. Natural fibers include a hydroxyl functional group that makes the fibers hydrophilic. Hydrophilic natural fibers and hydrophobic polymer matrix lead to incompatibility and weak interfacial bonds between them. To resolve the problem of incompatibility of plant fibers with non-polar polymer matrices, various methods of surface treatment of natural fibers, including physical, chemical and biological processing, have been considered. From this perspective, in our opinion, the data presented in this article can be used as a database for further study of agricultural waste as fillers or reinforcing additives in polymer composites in order to accelerate the development and stimulate the commercial production of these new materials.

Key words: biocomposite, agro waste, natural fiber, surface modification

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INTRODUCTION

In the past few years, the use and application of agricultural waste for the development of polymer composite materials has been growing due to the demand for environmentally friendly materials, depletion of natural resources and awareness of environmental problems. Agro waste is the most abundant renewable resource residue on earth [1]. Their accumulation in large quantities every year leads not only to environmental pollution, but also represents an economic problem. About 10-50 billion tons of dry lignocellulosic wastes per year are produced worldwide [2]. Reuse and utilization of these residues can minimize environmental problems associated with their accumulation [3, 4]. Agricultural wastes are potential candidates, either as replacement or as complement to synthetic fiber in various applications due to its comparable properties. The potential applicability of this waste is unlimited and rapidly expanding due to their variety of unique characteristics which offered many properties that meet different requirements [5]. In addition, composites made from lignocellulosic materials are given considerable attention because of their properties: easy access, low density, low cost, good heat and sound insulation, environmental friendliness, the possibility of

processing, renewability and satisfactory mechanical properties, use in large volumes, low power consumption [6].

SOURCE, CLASSIFICATION AND CHEMICAL COMPOSITION OF LIGNOCELLULOSIC NATURAL FIBERS

Agricultural waste is the crop residue left in the plantation field after harvesting. This lignocellulosic material has a composition, structure and properties that make them suitable for use in various traditional and modern applications. They can be obtained from several sources, both from forest and agricultural resources [7].

Plant fibers, also called lignocellulosic fibers, are mainly composed of hemicellulose and cellulose embedded in the lignin matrix, and the quantity, morphology and how these components are found in the fibers depend on many factors [8]. In addition to these structured basic components, some other non-structural components, such as waxes, pectin, inorganic substances, nitrogenous salts, starch, protein, etc., are also present [9]. However, the exact composition of agricultural residues varies from plant to plant [10] and to a large extent depends on the geographical region, climate and the area where it grows. Natural fibers are classified according to their origin or botanical type

[11]. Table shows the classification and chemical composition of various natural plant-based fibers. As can be seen from the Table 1 cellulose is the main structural component of agricultural residues [33], and it varies from 22.8% to 92%, while hemicellulose and lignin are found in 0.15-46% and 0-45%, respectively. However, the composition is not exhaustive; the actual percentage of components may increase or decrease.

Cellulose is hydrophilic and has a degree of polymerization of approximately 10000, which depends on the type of fiber. Hydrogen bonds provided by hydroxyl in the structure, as well as Van der Waals forces make cellulose molecules crystalline packed. In addition to these highly ordered (crystalline) regions, cellulose has less ordered amorphous regions [34], they are interconnected by lignin and hemicellulose fragments. These regions do not have clearly defined boundaries and represent a transition from the ordered arrangement of cellulose chains to amorphous [35], which is shown in Fig. 1.

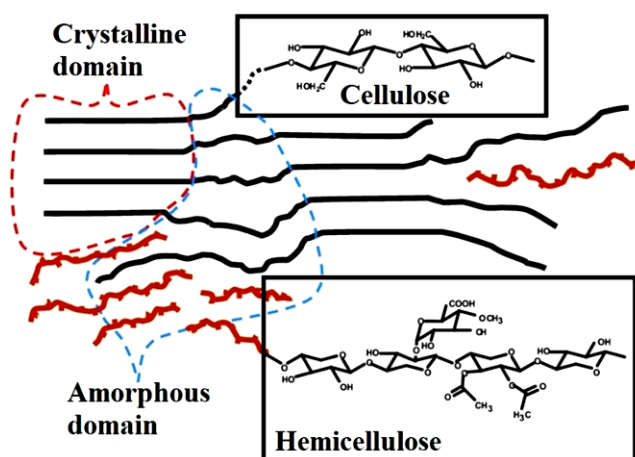


Fig. 1. Schematic representation of crystalline and amorphous regions in plant fibers [36]

Рис. 1. Схематическое изображение кристаллических и аморфных областей в растительных волокнах [36]

Table

Classification and chemical composition of natural fibers
Таблица. Классификация и химический состав натуральных волокон

Type	Fiber	Cellulose, % wt.	Hemicellulose, % wt.	Lignin, % wt.	References
Grass	Bagasse	44.1	31.8	22.3	[12]
	Bamboo	73.83	12.49	10.15	[13]
	Canary	37.2-41.7	19-22.9	-	[14]
	Corn	41.7	46	7.4	[15]
Bast	Jute	61-71.5	13.6-20.4	12-13	[16]
	Flax	74.93	10.37	2.62	[17]
	Hemp	75	15	3	[18]
	Kenaf	31-57	21.5-23	15-19	[19]
	Kudzu	33	11.3	14	[20]
	Nettle	79-83.6	6.5-12.5	3.5-4.4	[21]
	Ramie	61.85-73.21	5.27-7.58	4.6-9.06	[22]
Leaf	Abaca	60.4	20.8	12.4	[23, 24]
	Banana	63-64	10-24	5	[25]
	Henequen	70-77.6	4-20	8-13.1	[24, 26]
	Pineapple	70-82	18	5-12	[27]
	Sisal	26	38.2	26	[28]
Seed	Cotton	82.7-92	5.7-6	0	[24]
	Капок	64	13	23	[29]
Straw	Wheat	33-38	26-32	17-19	[27]
	Rice	28-36	23-28	12-14	[27]
Fruit	Coir	36-43	0.15-0.25	41-45	[16]
	Капок	35	22	21.5	[30]
	Oil palm	47.91	19.06	24.45	[31]
Husk /shell	Rice husk	45	19	19.5	[24]
	Hazelnut shell	25.9	29.9	42.5	[24]
Wood	Hard wood	31-64	25-40	14-34	[32]
	Soft wood	30-60	20-30	21-37	[32]

Hemicellulose is the second major structural component of fibers. Hemicellulose consists of various sugar units, is very hydrophilic, non-crystalline and

acts as a matrix for cellulose microfibrils. Hemicellulose has a degree of polymerization of about 50-300. Hemicellulose can dissolve in alkali and hydrolyze in acids [37].

Lignin is a complex hydrocarbon polymer having both aliphatic and aromatic components, amorphous and hydrophobic in nature [38]. Lignin acts as filler between cellulose, hemicellulose and pectin structures. Lignin provides rigidity to cellulose and hemicellulose structures.

SURFACE MODIFICATION OF NATURAL FIBERS

Despite the many advantages of using natural fibers as a reinforcing agent, their characteristic incompatibility with non-polar polymer matrices remains a serious problem [39]. Cellulose-based natural fibers are hydrophilic, while commonly used polymer matrices are hydrophobic. Hydrophilic natural fibers are incompatible with a hydrophobic polymer matrix and tend to form aggregates that affect the quality of the fiber-matrix interface. Therefore, poor interfacial connection between the fiber and the matrix is caused by surface incompatibility, which leads to poor surface properties and material defects that contribute to premature mechanical failure of the composites [40]. Since they are hydrophilic and thus have poor moisture resistance. To eliminate this problem, plant fibers undergo several types of surface modification [41], including physical [42], chemical [43] or biological [44] treatments; but their goal is the same: to break the bonds of atoms of some surface groups of fibers to ensure their functionalization [45]. Thus, modification of natural fibers is attempted to produce hydrophobic fibers in order to improve interfacial adhesion between the fiber and the matrix polymer.

PHYSICAL METHODS FOR SURFACE MODIFICATION OF NATURAL FIBERS

Physical treatment methods change the structural and surface properties of the fibers, which leads to improved mechanical adhesion to the polymer, in other words, to greater compatibility with the matrix in the composite [46]. To modify the surface of the filler, methods such as plasma (thermal) treatment, dielectric barrier methods, or corona treatment (non-thermal) are used.

Plasma treatment is one of the most common physical surface modification processes currently used in industry; it is very effective, especially when applied to the modification of natural fibers [47]. Plasma treatment is used to remove surface contaminants (cleaning effect) that cause changes in surface properties, such as wettability, dyeability, flammability, etc. This treatment can also increase surface roughness, providing better mechanical bonding to the polymer and improving adhesion at the fiber interface. At the same time, plasma can form free radicals capable of reacting with

oxygen or other gases, which leads to the formation of surfaces with different hydrophilic/hydrophobic nature [48].

Atmospheric plasma treatment is a very attractive method because it allows you to process the sample in place, rather than being limited to a vacuum chamber [49, 50]; provides continuous and uniform processing, reliable, reproducible and not as expensive as processing at low pressure, avoiding the use of expensive vacuum systems. There are four main types of atmospheric plasma treatment methods: corona treatment, dielectric barrier discharge technology, glow discharge, atmospheric pressure plasma jet.

Corona treatment is one of the most interesting methods for activating surface oxidation of materials [51, 52], while the fiber polarity and the number of -OH and -COOH groups increase, and, therefore, adhesion between the fiber and the polar matrix is improved. Corona treatment is based on high-frequency discharges applied to the electrodes and a grounded metal shaft. This treatment can also be used as a preparation step for grafting the surface of a cellulosic fiber to hydrophobic non-polar polymers.

Glow discharge is very suitable for more even surface treatment. Glow discharge is characterized as a uniform, homogeneous and stable discharge, usually generated in helium (sometimes argon and nitrogen can be used, but helium is the preferred gas). Alkaline pre-treated fibers exhibit several morphological changes during glow-discharge plasma treatment [53]. The surface roughness, as well as the grinding ability, increased with increasing plasma power and processing time.

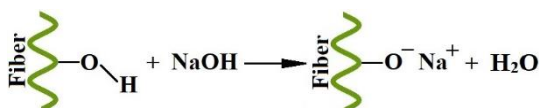
Ultrasonic modification is also effective in removing various substances and contaminants from surfaces even without the use of surfactants in the wash bath. This effect can be attributed to acoustic cavitation, known as the formation and destruction of bubbles, responsible for most of the physical and chemical effects of ultrasound observed in solid/liquid or liquid/liquid systems [54]. A study of the effect of ultrasonic treatment on natural fibers showed that ultrasonic treatment can change the crystal morphology and subsequent mechanical and chemical properties of the fiber [55].

Ultraviolet radiation is a potential energy source that can stimulate photochemical reactions in the molecular structures of natural fibers, changing its mechanical properties, and at the same time acts as a pure method for modifying the surface of natural fibers [56]. Ultraviolet radiation turned out to be a clean and cost-effective method, as well as a very promising method for industrial applications because of its cost effectiveness and simplicity.

CHEMICAL METHODS FOR SURFACE MODIFICATION OF NATURAL FIBERS

Chemical modification methods have been applied to natural fiber to improve matrix adhesion to fiber. Some chemical modifications may reduce the moisture absorption of natural fibers. Most chemical modifications of natural fiber include silanization, alkalization (mercerization), treatment with maleated coupling agent, acetylation, benzylation, treatment with permanganate and peroxide, etherification, grafted copolymerization, treatment with isocyanates, etc. The only result of all chemical treatments of fibers is the removal of contaminants and "OH" coatings, the removal of non-cellulosic materials, inorganic substances and wax, which leads to a higher surface roughness of the fiber. These chemical modifications consist of chemical reactions between the reactive components of natural fibers (for example, hydroxyl groups) and a chemical reagent that forms a covalent bond between them [57].

Alkaline treatment, also known as mercerization, is one of the oldest and most used chemical treatments for natural fibers. This method consists in treating the fibers with sodium hydroxide (NaOH) to remove certain amounts of lignin and hemicellulose from the surface and completely remove pectin, wax, oils and other organic compounds [58]. It is assumed that after this removal, more cellulose molecules will be exposed on the surface, which will improve the adhesion of the fibers to the polymer matrix due to the larger number of possible reaction sites. Thus, the treatment affects the chemical composition of the fibers and the molecular orientation of the cellulose crystallites. Surface roughness also increases, improving mechanical properties and adhesion [59, 60]. Scheme 1 illustrates in general terms the alkaline treatment reaction in which the addition of sodium hydroxide promotes the ionization of the hydroxyl groups of natural fibers into alkoxides.

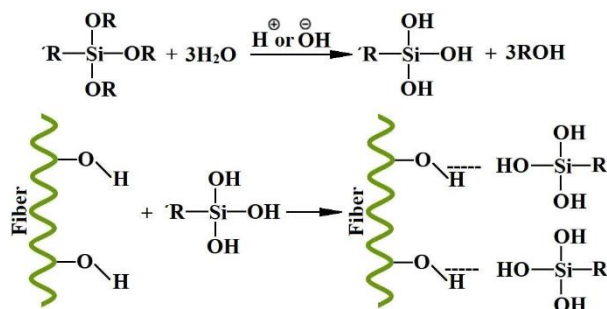


Scheme 1. The mechanism of alkaline treatment of natural fibers
Схема 1. Механизм щелочной обработки натуральных волокон

Studies have shown that fiber-based composites treated with lower alkali concentrations had higher tensile strength and impact strength [61]. Treatment with a low concentration of NaOH solution causes hydrophobicity in natural fibers. Further research showed that a higher concentration of NaOH

leads to fiber degradation. In addition, alkaline treatment can be combined with other surface modification methods to obtain a synergistic effect [62].

Silanes are very promising and versatile coupling agents that can be used in the treatment of natural fibers to improve the chemical interaction between the fiber and the polymer matrix [63]. The silane molecule must have bifunctional groups that can accordingly react with two phases, thereby forming a bridge between them [64]. As can be seen from Scheme 2, the first stage of the reaction mechanism is the hydrolysis of a silane coupling agent. The alkoxy groups of the used coupling agent are first hydrolyzed to form reactive silanol groups. After that, the hydrolyzed silane solution is mixed with natural fibers. The reactive silanol groups of the silane molecule allow the silane to bind to the OH groups of the fiber surface via hydrogen bonds. On the other hand, alkyl groups connect the silicon atom with polymer functional groups, forming a siloxane bridge between the fiber and the polymer. This phenomenon increases the compatibility with organic compounds and the hydrophobic nature of the surface, which leads to an increase in the strength of the interface in the polymer matrix [65].

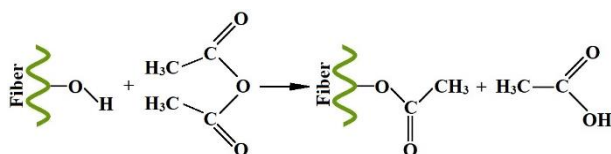


Scheme 2. The mechanism of silanization of natural fibers
Схема 2. Механизм силанизации природных волокон

In addition, the silane treatment efficiency may be higher when the samples are alkaline treated before the silane treatment. In this case, the formation of reactive centers in the molecule increases, which leads to a more successful reaction with silanes. Both treatments can be combined, improving the properties of the fibers and enhancing the fiber-matrix connection in composites [66].

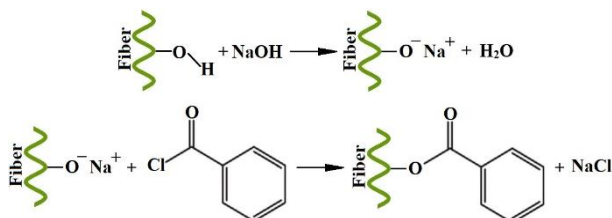
The acetylation method is very effective for modifying the surface of natural fibers in order to increase their hydrophobicity. As usual, the treatment consists in the use of acetic anhydride and acetic acid to plasticize cellulose fibers (esterification method). Etherification occurs as a result of the reaction of acetyl groups (CH₃CO-) with hydroxyl groups (-OH) on

fibers [67], as shown in Scheme 3. This chemical modification promotes the replacement of polar hydroxyl groups found on the cell wall of the fiber with less polar acetyl groups [68]. As a result, the hydrophilic nature of the fiber decreases, which leads to better compatibility with a non-polar matrix. Acetylation treatment is effective in reducing moisture absorption of natural fibers, as well as in improving the mechanical properties of composites, such as interfacial shear and tensile strength [69]. In some cases, acetylation may be preceded by alkali pretreatment for best results [70].



Scheme 3. Acetylation mechanism of natural fibers
Схема 3. Механизм ацетилирования натуральных волокон

As with acetylation, the main goal of the benzylation method is also to reduce the hydrophobicity of the fibers. Firstly, as shown in Scheme 4, alkali pretreatment is usually performed to activate the hydroxyl groups of the fiber. Secondly, benzylation is carried out by the reaction of benzoyl chloride with hydroxyl groups of cellulose [71].

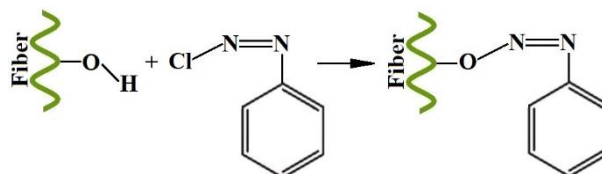


Scheme 4. Alkali treatment reaction followed by a reaction between fiber and benzoyl chloride
Схема 4. Реакция щелочной обработки с последующей реакцией между волокном и бензоилхлоридом

This reaction leads to the introduction of functional benzoyl groups ($\text{C}_6\text{H}_5\text{CO}-$) into the fibers, improving possible adhesion to the matrix [72]. The benzylation process improves fiber/polymer interactions, but also increases the thermal stability of the fibers and the strength of the resulting composite [73].

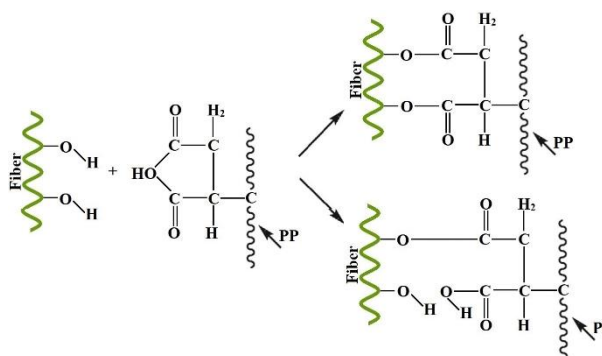
The outer side of the natural fibers is modified, and the interfacial connection is improved by diazo-treatment [74]. Benzenediazonium chloride in the fiber forms a rough coating and interaction on the surface of the fiber, which enhances the morphological properties. In addition, benzenediazonium chloride treatment partially fills the voids spaces of the fiber and aligns uneven fiber, which provides a smoother surface texture.

This result also indicates that the chemical reaction forms a stable interface with the cell walls of the fiber, which explains the observed increase in tensile properties of the composites [75]. Scheme 5 shows an example of a possible mechanism for the reaction of benzenediazonium chloride with natural fibers.



Scheme 5. Reaction of benzenediazonium chloride with natural fibers
Схема 5. Реакция хлорида бензодиазония с натуральными волокнами

The maleated coupling agent treatment method is widely used to strengthen natural fiber composites [76]. The fundamental difference from other chemical treatments is that maleic anhydride is used not only to modify the surface of the fibers, but also for the polymer matrix, in order to achieve a better interfacial connection between them and improve mechanical properties [77]. One of the most commonly used polymers is polypropylene, which in the presence of maleic anhydride forms maleic anhydride grafted polypropylene (MA-g-PP) [67]. With this treatment, MA-g-PP can react with hydroxyl groups in the amorphous region of the cellulose structure, leading to the formation of hydrogen or covalent bonds between them [78]. Maleic anhydride can form a carbon-carbon covalent bond with the polymer matrix, as well as combine with the hydroxyl groups of the fiber, forming an effective bridging interface. Scheme 6 shows an example of a possible mechanism for the reaction of MA-g-PP with fibers.



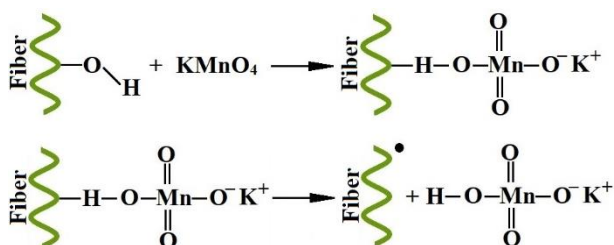
Scheme 6. Reaction of MA-g-PP with natural fibers
Схема 6. Реакция МАПП с натуральными волокнами

Many other chemical compounds can also be used to reduce the number of hydroxyl groups, as well as to improve interfacial adhesion between the fiber

and the polymer. Permanganate is a compound containing the permanganate group MnO_4^- . Treatment with permanganate leads to the formation of a cellulose radical through the formation of MnO^{3+} ions. These highly active Mn^{3+} ions are responsible for initiating graft copolymerization, as shown in Scheme 7.

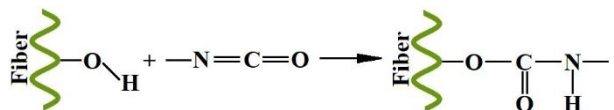
Most permanganate treatments are carried out using a solution of potassium permanganate in acetone in various concentrations with a holding time of 1 to 3 min after preliminary alkaline treatment [79].

Peroxide functional groups can initiate free radical reactions between the matrix and cellulose fiber. Benzoyl peroxide $((\text{C}_6\text{H}_5\text{CO})_2\text{O}_2)$ and dicumyl peroxide $(\text{C}_6\text{H}_5\text{C}(\text{CH}_3)_2\text{O})_2$ are chemicals of the class of organic peroxides that are used in surface modifications of natural fibers [80].



Scheme 7. Mechanism of permanganate treatment
Схема 7. Механизм обработки перманганатом

Isocyanate is very sensitive to reaction with hydroxyl groups. In this treatment method, the isocyanate functional group $-\text{N}=\text{C}=\text{O}$ reacts with the hydroxyl groups of cellulose and lignin [81] and forms strong covalent bonds (Scheme 8), which, in turn, adhere to the matrix.



Scheme 8. Mechanism of isocyanate treatment
Схема 8. Механизм обработки изоцианатом

Stearic acid $(\text{CH}_3(\text{CH}_2)_{16}\text{COOH})$ is also used to reduce the hydrophilicity of lignocellulosic fibers and, therefore, increase their water resistance [82]. Surface treatment of natural fibers with stearic acid leads to a change in surface topography. Stearation was tested for various types of lignocellulosic biomass, including jute [83-85], sisal [86], kenaf [87], and sugarcane [88].

BIOLOGICAL METHODS FOR SURFACE MODIFICATION OF NATURAL FIBERS

Currently, chemical or physical modifications are one of the most common types of surface treatment. However, they have several disadvantages, such as: the

use of large amounts of solvents and hazardous chemicals, waste generation, pollution, high energy consumption and the high cost of some chemicals and equipment. Microorganisms, such as fungi, bacteria and enzymes, can be used to overcome this problem; they can change the surface of natural fibers with less energy consumption. There are three types of biological treatment methods: enzymatic, fungal, and bacterial treatment.

Enzymatic treatment of natural fibers is an environmentally friendly and very specific method. The process consists in the use of enzymes for the selective removal of hydrophilic pectin, lignin, and hemicellulose components from fibers [89], reducing the hydrophilicity of natural cellulose fibers. The main enzymes used in this catalytic process (enzymatic hydrolysis) are hydrolases and oxidoreductases [90]. Several research papers have noted that enzymatic treatment promotes better matrix adhesion and improved mechanical properties of composites [91]. In addition to all these advantages, the cost of using enzymes remains the most important problem and the probable reason for its limited use.

Fungal treatment of natural fibers is also an environmentally friendly and effective alternative to chemical methods. It is used to remove non-cellulosic components (such as wax, lignin or pectin) from the surface of fibers under the action of certain enzymes [92]. The white-rot fungus *Schizophyllum commune* produces extracellular oxidases, enzymes that react with lignin components (lignin peroxidase). These enzymes are responsible for removing lignin from the surface of natural fiber, increasing its roughness [93]. At the same time, fungi are able to form hyphae, which create thin holes on the surface of the fibers, providing roughness to the surface of the fibers, and ultimately increase interfacial adhesion to the matrix [94]. It can be stated that fungal processing is an inexpensive method and green processing, which should lead the composite industry from traditional synthetic materials into green and environmentally friendly products [95].

The pre-cited methods take into account the treatment of natural fibers by removing substances from their surface. Coating the surface with bacterial cellulose is the latest modification technique, which involves adding new material to a surface of natural fibers, rather than removing it. Cellulose can be synthesized by various types of bacterial genera such as *Gluconacetobacter* (*Acetobacter*), *Agrobacterium*, *Aerobacter*, *Azotobacter*, *Rhizobium*, *Salmonella*, *Escherichia* and *Sarcina* [96]. When bacteria of this type are added to the appropriate culture medium in the presence of natural fibers, they produce bacterial cellulose,

which is preferably deposited *in situ* on natural fibers [97]. The purpose of coating natural fibers with bacterial cellulose is to improve interfacial adhesion between the fiber and the polymer, as well as to create hierarchical nanocomposites reinforced with green fiber with improved properties and significantly better strength [98].

In summary, chemical treatment is the most commonly used method of the three. The advantage of this method is, first of all, in the ease with which the process can be controlled in comparison with physical and biological methods. Physical methods are very energy intensive. In the same way, biological methods are more successful on a laboratory scale than on an industrial scale, since there are various parameters that need to be evaluated and controlled in order to operate such a system for surface modification. Thus, the systematization and analysis of the available literature data on use of agro waste as lignocellulosic fillers showed that natural fibers are very promising materials that can be used in the development of polymer composites using various methods of surface modification of the fibers.

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