

Natural Fiber-Reinforced Biocomposites

Subjects: **Biology**

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In the last ten years, environmental consciousness has increased worldwide, leading to the development of eco-friendly materials to replace synthetic ones. Natural fibers are extracted from renewable resources at low cost. Their combination with synthetic polymers as reinforcement materials has been an important step forward in that direction. The sustainability and excellent physical and biological (e.g., biocompatibility, antimicrobial activity) properties of these biocomposites have extended their application to the biomedical field.

natural fibers

biocomposites

surface modification

specialized biomolecules

immobilization methods

1. Introduction

The use of eco-friendly materials has been increasing with time as a result of global environmental awareness. The development of recyclable and environmentally sustainable materials has become an attractive and important field of research. Natural fibers are among these materials and are gradually replacing synthetic fibers made from non-renewable petroleum-based resources [1][2].

Composites are formed of a strong load-carrying material (reinforcement) embedded within a “weaker” material (matrix). Because of the beneficial properties, abundance and low cost of natural fibers, these are considered a new generation of reinforcements for polymer matrices. By themselves, natural fibers are very unpredictable (with properties varying from batch-to-batch) and do not possess the mechanical resilience desirable for most applications; as such, combinations with polymer matrices have been proposed [3][4]. A biocomposite is considered a material that is composed of at least one natural resource. The natural fiber added value endows the biocomposites with a wide range of physical, mechanical and biological properties [5]. Manufacture of biocomposites can be accomplished by different processing techniques, including compression molding, injection molding, resin transfer molding, sheet molding, hand lay-up, filament winding, extrusion and pultrusion. These processes allow the natural fibers, which are presented in the form of loose fibers, nonwoven mats, aligned yarns and/or woven fabrics, to be placed in the desired direction to acquire specific mechanical properties in the final product [6]. There are other factors that must be considered as well to attain desirable properties, such as the type of natural fiber, the chemical compatibility between the fiber and matrix phases, the corresponding surface energies and the quality of the interface [7]. The interfacial bonding between both materials in a biocomposite are affected by the natural fiber's hydrophilicity and polymer matrix hydrophobicity. Chemical and physical methods are required to treat the surface of the fiber to optimize this interaction [3].

The natural fibers' abundance, availability and low-cost have made biocomposites very attractive for several industrial applications. However, in biomedicine, specific requirements must be met prior to their use. The most important is to be accepted by the human body without causing any adverse response, namely inflammation, allergies and/or early rejection associated with toxicity. Biocompatibility is, therefore, essential for the successful development of a biomedical device [8][9]. Even though biocomposites on their own have been reported in medical textiles [10], the addition of specialized biomolecules with particular properties, such as antimicrobial, anti-inflammatory, analgesic, sedative, anti-oxidative, UV-protection or chemical stability, to name a few, have demonstrated improved performance on specific biomedical applications. Biomolecules such as peptides, antibiotics, nanoparticles (NPs) or plant extracts functionalized onto biocomposites contribute significantly to their biocompatibility towards host cells, while improving other dormant material properties [11][12][13][14][15]. These combinations have been desirable for prospective applications in sutures, coatings for cell culture and drug delivery matrices, as well as for 3D scaffolds for ligaments, bone, cartilage, skin and vasculature engineering [10]. Still, even though they have demonstrated tremendous potential, research in this field is only now taking the first steps with the use of biocomposites for biomedicine, requiring further study and understanding. The present work explores this subject further by introducing some of the most recent (last ten years) biomolecule–biocomposite combinations and their final product properties. Fiber extraction, separation and chemical and physical processing prior to interfacial bonding with polymer matrices were also discussed. Finally, a detailed and critical analysis of the biomolecule's inherent characteristics and the most recurrent methods employed for their immobilization onto natural fibers, fabrics and biocomposites was provided.

2. Treatments of Natural Fibers for Successful Biocomposite Production

As mentioned earlier, it is important to modify the natural fiber surface to achieve a good interface bonding with the polymer matrix. Because of their low water and moisture absorption, and wettability [16], natural fibers require further chemical and surface treatments to optimize their performance as reinforcement agents.

2.1. Chemical Treatments in Plant-Based Fibers

Plant-based natural fibers are composed of cellulose, hemicellulose, lignin and wax [17]. [Table 1](#) shows the percentage of chemical compounds in some of the most common natural fibers. Cellulose is the strongest and stiffest component of the fibers, endowing the fiber surface with several hydroxyl (-OH) groups and making them hydrophilic in nature. In addition, waxy substances cap the fiber reactive functional groups acting as an interference to interlock with the matrix that results in poor interfacial interaction with the hydrophobic polymer matrix. To turn the fibers less hydrophilic and, consequently, increase their mechanical and physical properties, modifications are necessary. Generally, the fiber structure composition is altered by chemical treatments using functional groups to react with the surface available hydroxyl groups. This can be accomplished through [3][16][17][18][19][20]:

Table 1. Chemical composition of some of the most common natural fibers (adapted from [1][2][21][16][22][23][24][25]).

Fiber	Cellulose (wt %)	Hemicellulose (wt %)	Lignin (wt %)	Wax (wt %)
Bast fibers				
Jute	61.0–71.5	13.6–20.4	12.0–13.0	0.5
Flax	71.0	18.6–20.6	2.2	1.7
Hemp	70.2–74.4	17.9–22.4	3.7–5.7	0.8
Kenaf	45.0–57.0	21.5	15.0–19.0	--
Ramie	68.6–76.2	13.1–16.7	0.6–0.7	0.3
Seed fibers				
Cotton	82.7–91.0	5.7	--	0.6
Milkweed	55.0	24.0	18.0	1.0–2.0
Coir	32.0–43.0	0.2–0.3	40.0–45.0	--
Kapok	13.0–35.0	23.0–32.0	13.0–21.0	--
Leaf fibers				
Sisal	67.0–78.0	10.0–14.2	8.0–11.0	2
Pineapple	70.0–82.0	--	5.0–12.0	--
Agave	68.4	4.9	4.9	0.3
Banana	63.0–64.0	6.0–0	5.0	--
Abaca	56.0–63.0	20.0–25.0	7.0–12.4	3
Grass fibers				
Bagasse	55.2	16.8	25.3	--
Bamboo	26.0–43.0	30.0	21.0–31.0	--
Straw fibers				
Rice	41.0–57.0	33.0	8.0–19.0	8.0–38.0
Corn	38.0–40.0	28.0	7.0–21.0	--
Wheat	38.0–45.0	15.0–31.0	12.0–20.0	--
Wood fibers				

Fiber	Cellulose (wt %)	Hemicellulose (wt %)	Lignin (wt %)	Wax (wt %)	h sodium matrix;
Softwood	40.0–45.0	7.0–14.0	26.0–36.0	--	
Hardwood	38.0–50.0	19.0–26.0	20.0–30.0	--	ne bridge

between them. Silanol (Si-OH) groups react with -OH groups of the fibers and the matrix functional groups;

Acetylation by introducing an acetyl group on the fiber surface. Here, the -OH groups react with the acetyl groups decreasing their hydrophilic nature;

Peroxide treatment by generating free radicals that react with the -OH groups of both fiber and polymer. This treatment requires an alkaline pre-treatment;

Benzoylation treatment using benzoyl chloride to treat the fibers and decrease their hydrophilic nature by replacing of -OH groups with benzoyl groups. In this method, an alkaline pre-treatment is required;

Potassium permanganate treatment by forming highly reactive permanganate ions that react with the -OH groups, generating cellulose-manganate to initiate graft copolymerization;

Stearic acid treatment by inducing the interaction between reactive carboxyl groups of stearic acid with the fiber -OH groups, and thus improving water resistance properties;

Isocyanate treatment by acting as a coupling agent between the fiber and the matrix. Isocyanate functional groups react with the cellulose and lignin -OH groups, forming a chemical linkage by means of strong covalent bonds;

Maleated coupling treatment by means of maleic anhydride, which is used to modify the fiber surface and the polymeric matrix, ensuring high compatibility between them. Maleic anhydride is grafted onto the polymer, becoming available to react with the cellulose -OH groups by means of hydrogen or covalent bonds.

Many other chemical treatments can be used to treat fibers in order to reduce the number of hydroxyl groups and improve the fiber adhesion to the matrix, including acrylation, acrylonitrile grafting, triazine, zirconate, titanate, sodium chlorite, fungal and enzyme treatment. Chemical treatments comprehend a class of the most important approaches to improve natural fiber adhesion to a polymeric matrix, modifying their microstructure, improving tensile strength, wettability, surface morphology and increasing the number of available chemical groups [17].

2.2. Chemical Treatments in Animal Fibers

Animal-based fibers are mainly composed of structural proteins; hence, specific chemical modifications must be employed to these fibers, including coupling reactions (cyanuric chloride-activated, carbodiimide and glutaraldehyde coupling), amino acid modification (arginine masking, sulfation of tyrosine and azo-modified tyrosine) and grafting reactions (tyrosinase-catalyzed and poly(methacrylate) grafting). The primary structure of silk fibroin (SF), the protein from silkworm, contains a repetitive sequence of glycine-alanine-glycine-alanine-glycine-serine amino acids, which self-assemble into an anti-parallel β -sheet structure. The crosslinking between β -sheets

along the protein is done by means of strong hydrogen bonds and Van der Waals interactions that endows silk with excellent mechanical properties [26][27]. SF is widely used in biomedical applications. However, it is essential to modify the SF surface chemistry to better control the interaction between silk and the living systems. SF possesses many reactive functional groups that facilitate crosslinking with other polymers, thus increasing its use as a reinforcing fiber [28]. Due to the presence of several reactive amino acids in SF, chemical modifications via coupling and grafting reactions and amino acid modifications can be applied. Wool and chicken feathers are mainly composed of keratin, a structural protein similar to SF. The chemical structure of keratin is predominantly an α -helix in chicken feathers [29] and a super coiled polypeptide chain with an α -helix and β -sheet in wool [30]. These structures are tightly packed via cross linkages, hydrogen bonds, Van der Waals and electrostatic interactions.

Chemical modifications play an important role in fiber functionalization, improving existing physicochemical properties or incorporating new ones. The fiber protein amino acid residue side chains may be conveniently conjugated with a variety of chemical groups [31]. These modification methods can be classified into coupling reactions, amino acid modification and grafting reactions. Coupling reactions are mainly used to immobilize peptides, molecules and polymers in fiber proteins. Copper-catalyzed azide-alkyne cycloaddition reactions, cyanuric chloride, carbodiimide and glutaraldehyde are very effective coupling agents [32][33]. The amino acid modifications are made through arginine masking, which is used to regulate the surface charge, sulfation/oxidation of tyrosine, which causes the hydrolysis of the fiber protein [32], and azo-modified tyrosine that can be used to install small molecules into fiber protein, resulting in hydrophobic and hydrophilic derivatives [34]. The grafting reactions include tyrosinase-catalyzed grafting and poly(methacrylate) grafting. Still, the chemical treatments may also be applied to these protein fibers when used as composite reinforcements due to their several reactive functional groups [35][36].

2.3. Physical Surface Treatments

In addition to the mentioned chemical treatments, it is also very common to improve the fibers' surface through physical surface treatments. Some of these approaches are used to functionalize the natural fibers' surface and consist of the use of plasma, ultrasounds and UV-light. Plasma treatment is one of the most common surface modification methods. Cold plasma treatment is required to remove the surface impurities which, consequently, induces modifications in the surface properties, such as wettability, flame resistance, printability, etc., and increases surface roughness leading to better mechanical interlocking and interfacial adhesion between the fiber and polymer [18][37]. The hydrophilic/hydrophobic surface character can also be changed with the incorporation of free radicals capable of reacting with oxygen or other gases [19]. Plasma is a partially ionized gas that reacts with the fiber surface. Plasma is generated by applying an electrical field between two electrodes, which transmit energy, accelerating the gas electrons that collide with neutral gas molecules or atoms under atmospheric pressure or in a vacuum. In the case of a plasma vacuum, the gas is introduced at a low pressure in a vacuum chamber causing ionization by means of atom removal or bond rupture, giving rise to free radicals and crosslinking. However, this method requires an expensive closed system and is considered a batch process [38][39]. The treatment with atmospheric plasma is more attractive for industry, as it allows the samples to be treated *in situ* rather than restricted to a vacuum chamber. It is a continuous and uniform treatment, reliable and reproducible [40].

The atmospheric plasma technique can be divided into different types of discharge, such as corona-discharge, dielectric barrier discharge, glow discharge and atmospheric pressure plasma jet.

Corona treatment is a process based on low-frequency discharges applied in two opposing electrodes and grounded metal roll. These discharges induce ionization of the nearby atmosphere generating plasma. The fiber is placed in the gap between the electrodes and is bombarded with high-speed electrons, inducing surface oxidation and increasing the amount of high reactive free radicals [38][41]. It is a low-cost process with low energy consumption and exhibits several advantages compared with others plasma treatments [19]. The dielectric barrier discharge (DBD) technique is similar to the corona treatment. However, here, there is one or more dielectric barriers in the path between the electrodes, acting as an insulator. These accumulate the transported charge and distribute it over the entire electrode area. The gas between the electrodes is not ionized and only serves as a reservoir to absorb the energy dissipated. The main disadvantage of DBD is that it is not completely uniform and has a short duration [42][43]. The atmospheric pressure glow discharge (APGD) is a more stable, uniform and homogeneous surface treatment than DBD. This technique is generated in helium or argon by applying low voltages through parallel conductive electrodes at higher frequencies. The glow of the discharge refers to the characteristic luminescence resultant from excitation collisions followed by de-excitation [37][44]. In the atmospheric pressure plasma jet (APPJ) there are two tubular metal electrodes separated by a gap. Between the electrodes, a quartz cylindrical tube is inserted where helium (or other gases) flows. The plasma is launched into the surrounding air in the form of a plume or bullet, directly into the sample. This process can provide a local and very precise treatment [38]. APPJ is suitable for industrial and research applications, namely treatment of heat-sensitive materials, biological material sterilization and several biomedical devices [45].

Ultrasound treatment, while not as common as plasma treatment, is also effective in surface modifications. This method causes the cavitation effect, which is the formation, by ultrasonic irradiation, of small collapsing bubbles that generate powerful shock waves. The impact of the shock waves on the fiber surface leads to surface peeling, erosion and particle breakdown. Cavitation is responsible for the physical and chemical effects of ultrasound in solid/liquid and liquid/liquid systems and is more effective in heterogeneous systems than homogeneous systems. The effect of ultrasound treatment is related to its frequency; at low frequencies, violent cavitation is produced, and the effects are highly localized. On the other hand, with high frequency, the cavitation is less violent due to the shorter lifetime of the bubbles [20][46][47].

Ultraviolet treatment is based on UV-light, an electromagnetic radiation with a potential energy source capable of promoting photochemical reactions in the molecular structure of the fibers' surface [48]. UV-treatment is a clean and cost-effective process that can be used in industrial applications [19]. In addition to the processes described earlier, there are other physical methods of surface modifications, such as ozone treatment, gamma-ray irradiation treatment, laser treatment and ion beam treatment [18].

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