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Review

Giving Improved and New Properties to Fibrous Materials by Surface Modification

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Abstract: This review summarizes the results of research published in recent decades on the main directions in the functionalization of fibrous materials using surface modification. Methods for thepreliminary activation of the surface of fibrous materials are described, allowing increasing the adhesion of modifiers. The features of the formation of functionalizing coatings on fibrous materials in comparison with other substrates are analyzed. Some specific methods for evaluating the effectiveness of the surface modification inherent in fibrous materials are considered. Particular attention is paid to giving fibrous materials antimicrobial properties, photoactivity, the ability to protect against ultraviolet radiation, and hydrophobicity.

Keywords: fibrous material; functionalizing coating; antimicrobial activity; photoactivity; UV protection; hydrophobicity

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1. Introduction

One of the most urgent tasks in science is to improve quality of life and life expectancy. As such, new high-tech products are needed from various fields of the economy, including fibrous materials with improved and fundamentally new properties, produced by the textile industry. For example, in a reviews devoted to textile materials, what people want to see modern clothing's listed: it should be waterproof; fire-resistant; self-cleaning; protect against insects, infections, UV radiation, and chemical and biological agents; be warm in winter and cool in summer; and at the same time be bright and not bulky [1]. Thus, many serious requirements are imposed on the properties of fibrous materials for the manufacture of clothing, and which often contradict each other. The existing fibers and products made from them do not have the necessary characteristics. It is possible to give improved consumer characteristics and special properties to polymer fibrous materials through directed chemical or physical treatments of them; that is, by modification. There is information in the literature reporting that the adhesive ability, chemical resistance, surface energy, hydrophobicity, hydrophilicity, biostability, and many other properties of polymer materials can be determined by a surface layer with a thickness of ~10 nm to several micrometers [2]. Thus, a simple and economical approach to giving fibrous materials improved and previously non-inherent consumer properties is to regulate their surface characteristics; that is, surface modification of fibrous materials.

The global consumption of fibers and threads has shown a steady upward trend. At the same time, there has been an increase in the consumption share of chemical fibers and threads in the market of fibrous materials, due to a reduction in the consumption share of natural fibers. Currently, the share of chemical fibers and threads of world consumption exceeds 70%–75%. According to forecasts, 98% of fabrics will be partially or completely synthetic in the next 5–7 years. Thus, special attention should be paid to the modification of chemical fibers. This is especially difficult, since these fibers have a fine-pored structure and a smooth surface.

It is important to note that surface modification aimed at acquiring special properties should not negatively affect the properties originally inherent in fibrous materials: for example, after applying a modifier, fabrics should retain their softness, drapability, air and vapor permeability [3,4], and strength characteristics [5]. As modern research shows, these effects can be achieved using nanoscale modifiers.

In recent decades, it has been possible to obtain and thoroughly investigate a large number of functional nanoparticles and nanostructured materials that can be used as modifiers. Due to this, as well as due to the development and improvement of technologies for the formation of nano- and micro-dimensional modifying coatings based on such materials, significant progress has been observed in the field of the surface modification of fibrous materials. Many researchers are working on giving the most practically significant properties (individually and in combination) to natural and synthetic textile materials: antimicrobial activity, self-cleaning in light, superhydrophobicity, protection from UV radiation, etc. These goals can be achieved by using a variety of modifying substances and methods of application to fibrous materials.

In this review, an attempt is made to systematize the scientific works on the main areas of surface modification of fibrous materials published over the past 20 years. When analyzing these works, various methods for the preliminary activation of the surface of fibrous materials are considered, which allow increasing the adhesion of the applied modifiers, as well as other approaches to increase the fixation strength of functional coatings. Much attention is paid to the peculiarities of the formation of coatings on fibrous materials in comparison with other substrates, as well as some specific methods for evaluating the effectiveness of functional coatings on fabrics. Data on the resistance to operational impacts and the durability of coatings formed using different methods are analyzed.

2. Preliminary Activation of the Surface of Fibrous Materials

Pre-activation is often used to increase the adhesion of coatings to the surface of fibrous materials. This consists in various kinds of treatment soft the fibers, as a result of which their near-surface layer is transformed, new active oxygen-containing groups are formed, and the fiber becomes less smooth. In addition to increasing the surface content of reactive functional groups and increasing the roughness of the fiber surface, an important criterion for the effectiveness of pre-activation is the preservation of a high level of strength of the fibrous material, which can decrease with intense exposure to the fiber [6]. Surface activation is especially used for the pretreatment of synthetic fibers, many of which consist of chemically inert polymer materials.

The authors [7] divide the solvent (or "wet") activation methods for fibrous materials into those based on hydrolysis, oxidation, halogenation, complexation, and the formation of layers that promote adhesion. However, we do not consider the formation of coatings on fibers that promote the adhesion of functionalizing drugs as an activation method, since these usually have a significant effect on the properties of fibers. The method of modification of aliphatic and aromatic polyamides based on complexation causes significant changes in the bulk properties and structure of fibers [8–10]. Therefore, it cannot be attributed to the methods of surface activation. Thus, in this section we will briefly consider surface activation using the methods of alkaline and enzymatic hydrolysis, oxidation, and halogenation.

Fiber activation methods based on weak alkaline hydrolysis of the surface can be considered traditional. However, they are still being developed and improved. These methods are particularly suitable for polymer materials containing ester bonds [11,12].

Thus, it was found in [13,14] that it is possible to select a reagent, its concentration, and the duration of hydrolysis in such a way that a significant number of active groups are formed on the surface of the polyester fiber, which cause the fixation of functional preparations. In particular, the treatment of polyester fiber with a urea solution of a concentration of 0.05–0.1 mol/L at a boiling point for 15–20 min led to the formation of hydroxyl groups, the number of which was six-times higher than the initial one [14]. At the same time, the strength of the fiber remained at the initial level.

Another method of hydrolysis is based on the use of enzymes. Enzyme proteins act as biocatalysts. Of particular interest for fiber modification are the hydrolases that provide controlled degradation of the fiber surface, with the formation of functional end groups and an increase in roughness, similar to alkaline hydrolysis [15–18]. Optimization of the results of enzymatic treatment can be carried out by regulating parameters such as the treatment temperature, pH value, concentration, and duration of exposure [19,20].

Oxidative methods for the surface activation of fibrous materials are based on the action of nitric, chromic acids, or potassium permanganate [21,22]. Such treatments usually lead to the formation of new functional groups on the fiber surface, such as hydroxyl, carbonyl, and carboxyl groups. Another method of oxidation of polymer fibers is treatment with phosphoric acid [23]. When using aqueous solutions of sulfuric acid, sulfonation can also occur simultaneously with oxidation [24]. This activation method was used in [25], the authors of which treated the fiber with concentrated H_2SO_4 to increase the adhesion of polypropylene fiber, before forming a coating based on modified graphene oxide on its surface.

Halogenation is based on the substitution of a hydrogen atom by halogens, through radical reactions or addition and substitution reactions. Chlorination is used to activate aromatic polyamide fibers in a wet state [26,27]. In previous works [26,27], this process was implemented using active chlorine-containing reaction agents, such as solutions of sodium dichloroisocyanurate or sodium hypochlorite. As a result, the wettability of the fiber was increased and the formation of active chlorine-containing groups was observed.

In addition to "wet" activation methods, so-called "dry" methods are often used, which do not require water consumption. These are considered more environmentally friendly. Thus, oxidative activation of fibrous materials can also be carried out by the "dry" method using ozone treatment [28]. Ozone is a powerful oxidizer, whose interaction with fiber leads to the formation of carboxyl, hydroxyl, and amino groups, in the case of polyamide fibers [29,30]. The morphology of the fiber surface changes and the roughness increases [31,32]. Ozone treatment can lead to a loss of fiber strength [31–33].

A popular method of "dry" pre-activation of fibrous materials is low-temperature plasma treatment [34–36]. When plasma interacts with polymer substrates, several reactions and processes can occur, most of which lead to the formation of free radicals and unsaturated organic products, cross linking, destruction of macromolecules, and the formation of gaseous products. Under the action of plasma, chemical and physical changes of the fibrous material occur in a thin surface layer, without affecting the entire volume of the polymer material. The authors in [37] estimated the thickness of a plasma-discharge modified layer at about 10 nm. The changes caused were regulated by several factors, such as the type of gas used, pressure, frequency, power, and processing time, as well as the nature of the fibrous material [34–36]. First of all, plasma pretreatment is a tool for improving the adhesive properties of fibers [37,38]. For these purposes, plasma at both low [39,40] and atmospheric pressure can be used, although atmospheric pressure plasma is more often used [37,41,42]. For example, the authors of [43,44] used plasma pretreatment of cellulose fabrics to increase the absorption of chitosan and silver nanoparticles, and in [37,45–47] plasma activation was used to increase the degree of fixation of TiO_2 on fibrous materials. It should be noted that the effect of plasma on the surface of a fibrous material associated with the formation of new functional groups may decrease over time [48,49].

In recent years, the attention of researchers has also been attracted by the effect on fibrous materials of a plasma discharge ignited in electrolyte solutions. It was shown in [50,51] that the treatment of polyester thread with diaphragm discharge plasma in an electrolyte solution ensured the formation of hydroxyl groups on the surface of the polymer material, the number of which was 3.2 times higher than the initial number. In this case, carbonyl and carboxyl groups were generated (in larger quantities than under the conditions of the chemical activation method) at an acceptable level of thread strength loss.

Each of the considered methods of preliminary activation of fibrous materials has certain advantages and disadvantages. The choice of method is determined by the type of fibrous material, as well as the requirements for the properties, structure, and morphology of the formed coatings.

3. Antimicrobial Properties

When considering fibrous materials with antimicrobial properties, we will not take into account medical materials (bandages, napkins, surgical threads with various impregnations), since these must have special, strictly regulated properties. Fibrous materials used in engineering and everyday life are given antimicrobial properties for various purposes [52]. Thus, when using fabrics in conditions that provide intensive biological load (for example, in the tropics), antimicrobial treatment is necessary to avoid biodegradation of fibers and destruction of the dye [53,54]. It is also known that fibrous materials, especially those contaminated during the wear process, are a good breeding ground for bacteria. As a result of their vital activity, an unpleasant smell can appear. Fabrics should have antimicrobial properties to avoid this occurrence [55]. The most important and significant task is the antimicrobial treatment of fibrous materials that reduce potential risks to human health, for example, giving antimicrobial properties to the clothing and underwear of patients in medical institutions, while protective clothing for hospital staff can significantly increase its protective functions [56,57].

Antimicrobial finishing can be divided into biocidal and biostatic [58,59]. Preparations for biocidal finishing kill microorganisms, while biostatic ones suppress their growth and development. As shown in [59], researchers use a variety of different methods to assess the biocidity and biostaticity of fabrics. This makes it difficult to evaluate the results and compare them.

In some cases, the wrong choice of method for evaluating the effectiveness of antimicrobial finishing can lead to incorrect results. For example, the AATCC 147 method, known as the "parallel streak method", can only be used to assess the antimicrobial effect of drugs diffusing into the environment, and not drugs fixed on a fibrous substrate. This method refers to qualitative factors, as do the methods AATCC TM30 (American Association of Textile Chemists and Colorists Test Method), ISO 20645, ISO 11721 (International Organization for Standardization), SN 195920, and SN 195921 (Swiss standard) [60]. Qualitative methods are fast and simple, but they do not allow comparing the effectiveness of different types of antimicrobial drugs for fibrous materials of different chemical compositions [61].

Quantitative methods include AATCC TM100, ISO 20743, SN 195924, JIS L 1902 (Japanese industry standards), and ASTM E 2149 (or its modification). These methods are more complex and take longer to implement, but facilitate the comparison of the effectiveness of different drugs on the same fiber basis [62,63]. The choice of a quantitative method also depends on the mechanism of action of the antimicrobial drug. For example, the ISO 20645 method can only be used for drugs diffusing into the environment [63]. The most informative methods can be considered AATSS100 and JIS L 1902, which are suitable for the analysis of drugs acting by any mechanism [60]. They are designed to determine both the ability of fabrics and textiles to inhibit the growth of microorganisms (evaluation of biostatic properties) and the ability to kill them during a 24 h contact period (evaluation of biocidal properties).

The antimicrobial drug can be injected into the inner areas of synthetic fibers during their molding or applied to the fabric at the stage of its finishing. In the first case, many requirements are imposed on the drugs, significantly narrowing their range [52,64–66]. In the second case, the choice of antimicrobial agents for fibrous materials is much wider. These include metals and their salts; triclosan, belonging to the group of halogenated phenols; quaternary ammonium compounds; a number of antibiotics; and some other compounds [60,67,68]. The use of heavy metals in the textile industry is limited for environmental reasons [67]. The possibility of developing resistance limits the use of antibiotics and quaternary ammonium compounds, and triclosan is known for its danger to the environment and can cause endocrine disorders in humans [60]. Biopolymers [69,70] and the nanocomposites based on them [71–73] can serve as a good alternative to these antimicrobial drugs for fibrous materials. In some publications, nanoglines [74,75], nanotubes, and the nanocomposites based on them [76,77] are mentioned as antimicrobial agents for fibrous materials. Currently, the majority of studies are devoted to the use of metal nanoparticles and their oxides, which are not addictive and have antimicrobial activity, as antimicrobial agents for fibrous materials, as well as nanocomposites based on them [52,59,78–80]. The antimicrobial activity of metal-containing nanoparticles is determined by their type, shape, size, and morphology [59,78–80].

The authors of [53,60], devoted to the use of metal nanoparticles, noted that the effectiveness of the antimicrobial action of fibrous materials depends on the activity of the antimicrobial agent and its concentration on the fiber. At the same time, it was shown in [4,47,81,82] that applying an excessive amount of the drug to a fibrous material can lead to the deterioration of the hygienic properties of the fabric, a decrease in air and vapor permeability, as well as an increase in stiffness. In addition, an excessive amount of drug is easily washed off from the fibrous material during washing and crumbles during operation with abrasion effects. Thus, too much antimicrobial drug should not be applied to a fabric.

The effectiveness and durability of the antimicrobial properties of a fibrous material is influenced by the method of applying the antimicrobial agent, since the adhesion of the agent to the fiber and the structure and morphology of the formed coating depend on it.

One of the most common methods for applying an antimicrobial coating to a fibrous material is the deposition of biologically active metal-containing nanoparticles obtained by sol-gel synthesis [47,83–85]. The authors recommend performing a preliminary chemical or plasma treatment to obtain an ordered ultrathin coating on fibers [47]. In [85], a binder was used to increase the fixation strength of the antimicrobial coating.

Similar methods of increasing the fixation strength of metal-containing nanoparticles are used when applied using colloids. Thus, plasma pretreatment was used in [44,86,87], and binders and stabilizers were used in [88–94]. Biopolymers that enhance the antimicrobial effect of nanoparticles are often used as binders and stabilizers. However, the use of binders, as shown in [95], sometimes reduces the comfort of the fabric and its resistance to abrasion, and [96] reported that an antimicrobial coating applied from a colloid without the use of a stabilizer was easily washed off during washing.

The authors of [97–99] proposed an ultrasonic method for the simultaneous synthesis of antimicrobial nanocomposites and their application to a fibrous material. The method provides the fabric with high antimicrobial efficiency and resistance of the resulting coating to washing.

Significant antimicrobial activity and a high degree of fixation on fibrous materials are provided by methods based on the reduction of silver ions during fiber processing [79,100–105].

A separate group includes methods for applying antimicrobial metal-containing nanoparticles to fibrous material, by loading them into organic carriers: nano- and microcapsules, cyclodextrins, liposomes, and dendrimers.

Innovative encapsulation technologies were proposed by the authors in [106,107]. A high antimicrobial activity of fibrous materials with microcapsules immobilized on their surface was shown in [108–111]. It was established that the achieved effect was resistant to washing. The presence of microcapsules provides prolonged antimicrobial properties to the fibrous material.

Cyclodextrins (cyclic oligosaccharides) [112], liposomes (spherical vesicles) [113], and dendrimers (regularly branched three-dimensional artificial molecules) [114] can act as nanocontainers to contain antimicrobial drugs and gradually release them.

As shown in [115–118], metal nanoparticles with antimicrobial properties are stabilized when incorporated into cyclodextrin molecules. Their bioavailability remains high, which manifests itself in an intense antimicrobial effect.

The author of [119] investigated the process of loading nanoparticles into liposomes. It was shown in [120,121] that liposomes have great potential for use as stabilizers for metal nanoparticles with antimicrobial properties, since they significantly increase the resistance of the antimicrobial effect to washing.

The authors of [122,123] presented a methodology for modifying textile materials with dendrimers containing metal nanoparticles. It was shown in [123] that encapsulation of copper nanoparticles in a dendrimer increased the antimicrobial activity.

The work in [52] presented a fundamentally new method for stabilizing nanostructures on the surface of a fibrous material, to impart antimicrobial properties to it. This method used the embedding of silver nanoparticles in a cross-linked layer of polysiloxane, which was applied to the surface of the fabric. This method ensures the resistance of the antimicrobial effect to washing and drying friction.

Recently, a method has been developed to impart antimicrobial properties to polypropylene thread by embedding iron oxide nanoparticles in a coating based on polytetrafluoroethylene [124,125]. The coating is formed on the surface of the thread at the stage of its molding from the melt [126]. The coating with embedded iron oxide nanoparticles is resistant to dry friction, washing, and the action of chemically aggressive liquids [124,125].

4. Photoactivity

Photochemically active materials under the influence of sunlight ensure the destruction of adsorbed organic pollutants, and such materials have the ability to self-clean. A simple method of imparting photochemical activity to materials is the formation of coatings based on semiconductor photocatalysts on their surface. The action of photocatalysts is based on the formation of reactive oxygen species, which provide decomposition of a wide range of organic compounds into carbon dioxide and water. The following requirements apply to photocatalysts [127,128]: (1) low toxicity; (2) ability to function at ambient temperature and pressure; (3) ability to completely decompose organic compounds; (4) low cost; (5) sensitivity to a wide range of contaminants; and (6) photocatalytic activity both indoors and outdoors. Most researchers considered nanoscale crystalline titanium dioxide (TiO_2) and zinc oxide (ZnO) as photocatalysts suitable for the formation of coatings on fibrous material [129–131].

Organic dyes, other organic compounds, and gases are used to evaluate the photocatalytic activity of fibrous materials [129,132]. Dyes are usually used as model pollutants in the analysis of the photoactivity of household fabrics. The degradation of dyes in solution or on the fabric surface can be easily monitored spectroscopically [133]. Spectrophotometric determination of the photoactivity of self-cleaning materials in the UV/visible range based on photoinduced discoloration of methylene blue is widely used (ISO 10678) [134,135]. However, according to the authors in [136], this dye is unsuitable for assessing the activity of a photocatalyst in the visible region, because it is characterized by significant absorption in this range. Other dyes used include Direct Green 6, Direct Dark Green BN, diazodyes Congo redand Acid Blue 113, monoazodyes Reactive Orange 72, Reactive Orange V-2G, Acid Orange 7, and many others [129]. In addition to synthetic dyes, natural colored compounds such as coffee, tea, wine, and other substances are applied to the surface of coated fabrics as pollutants [129–131].

Toxic compounds such as formaldehyde [137,138], p-nitrophenol [139], and organophosphate methyl parathion [140] are used to assess the photoactivity of military and technical fabrics [129], including fabrics for air filters [141–143]. The decomposition of toluene was studied in [144]. In some cases, substances such as 2-chloroethyl ethyl sulfide are used, which mimics mustard gas, due to the similarity in their structure, chemical composition, and physical properties [145].

To form a photochemically active coating on fibrous materials, the photocatalyst is applied to a textile substrate in the form of a pre-prepared suspension, or synthesized in situ. To give the fibrous materials photoactivity resistance to operational effects, it is necessary to ensure a strong fixation of the coating on the fiber (the achieved effect should be resistant to friction, washing, and chemical cleaning) [4,47,131,146]. This condition also makes it possible to meet the environmental requirements for processes based on the use of nanoparticles, since TiO_2 and ZnO nanoparticles fixed on a fiber will not pollute the environment [147]. In addition, the coating applied to the fabric should not adversely affect the softness or elasticity of the fibrous material [4,47,131], nor change the color of the fabric [146].

Photocatalyst-based coatings are formed on the surface of a fibrous material mainly by immersion, spraying, or layer-by-layer methods [131]. Many researchers use binders to increase the strength of photocatalyst fixation on a fibrous material. Thus, in [148], silicon dioxide played the role of binder, which according to the authors simultaneously protected the cotton fiber from the destructive effects of day light. SiO_2 can also be used as a binder when TiO_2 is applied to a polyester filter fabric [149], and SiO_2 or Al_2O_3 wereapplied to a fabric made of a mixture of cotton and polyester [150]. The authors in [151] suggested using succinic acid as a binder. Other non-toxic saturated polycarboxylic acids can also be used [85]. In [152,153], citric acid was used to increase the degree of fixation of TiO_2.

When analyzing the process of interaction of TiO_2 with fibers, most researchers rely on the fact that the carboxyl group is the best for fixation [154,155]. The authors of [88] increased the content of carboxyl groups on the surface of the fibrous material by using acrylate as a binder. A common method of introducing reactive functional groups into a fiber is the preliminary activation of the textile material using various types of plasma [37,45,46,89,131,156]. The disadvantage of this method is the need to form a coating immediately after activation, due to the fact that a number of the active functional groups formed as a result of plasma treatment are short-lived. In [157,158], it was proposed to treat a textile material with enzymes, to increase the fixation of titanium dioxide-based coatings on a fiber, while in [159] the activation of a polyester fabric using alkaline hydrolysis was considered. It is not possible to compare the data of different authors on the effect of various types of pretreatments on the photochemical activity of fibrous materials of various compositions. However, in [4,47], based on a comparison of the hydrolytic activation and treatment of polyester tissue with diaphragm discharge plasma, it was shown that these methods of pretreatment are close in their efficiency.

A simple and effective method is the formation of a photocatalytic coating on a fabric combined with low-temperature sol-gel synthesis of TiO_2 [93,160–163]. During implementation, the fibrous material is immersed in the reaction mixture, and the synthesized nanoparticles are deposited on its surface. Then the fibrous material is processed at temperatures below 100 °C, to further crystallize the photocatalyst and remove the solvent. This method is suitable for all types of fibrous materials, is characterized by high productivity, and ensures the uniformity of the formed nanoparticles at the molecular level.

Combining the stages of the formation of TiO_2 nanoparticles in the form of anatase or ZnO and the formation of coatings based on them on cotton and silk fabrics is also practiced using the ultrasonic acoustic method [98,164,165].

The use of various methods to increase the degree of fixation of photocatalysts on fabrics makes it possible to obtain coatings resistant to a sufficiently large number of washes, although after washing the rate of photodegradation of impurities decreases slightly [4,47]. A similar phenomenon was observed after the dry cleaning of samples with perchloroethylene [166].

It was shown in [167–170] that the self-cleaning effect increased with an increase in the amount of photocatalyst applied to the fibrous material. However, the authors of [4,47] found that an excessive amount of TiO_2 in the coating led to its shedding from the fibrous material during operation, as well as to an increase in the stiffness of the fabric. In order to reduce the amount of photocatalyst used, without reducing the level of photodegradation of contaminants, it is necessary to enhance the photochemical activity of the catalyst. Various methods for this are proposed in [171,172]. A commonly used method of increasing the photocatalytic activity of catalysts is doping them with a number of metals, oxides, and other compounds. Thus, the authors [173] found that the photoactivity of the coating increased when modified with TiO_2 aminosilane. Evidence of an increase in the photoactivity of TiO_2 during gold doping is presented in [174]. The effect on the activity of the photocatalyst of iron oxide was considered in the works [169,170,175]. Most researchers note that the maximum photochemical activity was shown by TiO_2 and ZnO doped with silver [4,46,47,86,100,138,176].

5. The Ability to Protect against UV Radiation

UV protection is one of the important functions of fibrous materials. Both organic and inorganic compounds are used as agents applied to textiles to block UV radiation. Organic UV absorbers are relatively cheap and usually transparent, which makes it possible to use them for colored fabrics [177,178]. However, they are washed out of fibrous materials during operation; in addition, their molecules undergo photodestruction over time [177]. Unlike organic UV absorbers, inorganic UV absorbers such as zinc oxide (ZnO), titanium dioxide (TiO_2), and cerium dioxide (CeO_2) have excellent light resistance. The mechanism of UV protection when using radiation blockers is based on the absorption of UV radiation, as well as the refraction and/or scattering of UV rays [179]. When using radiation blockers, the UV protection mechanism is based on the absorption of UV radiation, as well as the refraction and/or scattering of UV rays. On the contrary, TiO_2 has excellent chemical stability but is characterized by a narrower UV absorption range than ZnO [180]. In the textile industry in recent years, both TiO_2 and ZnO in the form of nanoparticles have been increasingly used to give textile materials the ability to protect against UV radiation [180].

The UV-blocking function of a fibrous material is usually estimated using the value of the ultraviolet protection factor (UPF) [88,155,181–183]. UPF is determined by measuring the direct and diffuse transmission of a fabric in the wavelength range of 290–400 nm, including the UVB (290–315 nm) and UVA (315–380 nm) regions. UPF is the ratio of the amount of transmission of UV radiation measured for a fabric without protection to the corresponding value determined for a fabric with UV protection. Initially, UPF was set as the standard AS/NZS 4399 of Australia/New Zealand [184], then other standards appeared, for example EN 13758-1 [185], AATCC Test Method 183 [186], and ASTM D6544-12 [187]. UPF shows how much the material reduces UV exposure and allows classifying clothing into categories of protection: good (UPF 15–24), very good (UPF 25–39), and excellent (UPF 40–49). Textile materials that protect against UV radiation must have an UPF of at least 15 [181–183].

To protect human skin from UV radiation, TiO_2 and ZnO are mainly applied to cellulose fibrous materials, as they are usually used for sewing light summer clothes [183,188–192]. However, research is also being conducted on other textile materials [193,194]. In [188,189] it was found that the protective functions of fibrous materials increase with an increase in the amount of the blocker fixed on them. It was also shown that due to the high specific surface area, TiO_2 and ZnO in the form of nanoparticles have a much stronger blocking effect than the microparticles of these oxides [194].

In order to maintain a long-term UV-blocking effect, it is necessary that the TiO_2- and ZnO-based coatings applied to the fibrous material are resistant to operational influences, especially to washing [195,196]. As shown in [197], ordered continuous coatings have higher adhesion to the fiber and, as a result, higher resistance to washing. Methods of forming coatings based on TiO_2 and ZnO nanoparticles on fibrous materials are described in the previous part of this review, devoted to giving textiles photoactive properties. To increase the resistance to washing of coatings that give fibrous materials protection against UV radiation, techniques of mixing TiO_2 and ZnO nanoparticles with binders, as well as the formation of covalent bonds between the coating containing these nanoparticles and the fibrous material [152,198,199], are also used [95,150]. In [200,201], it was proven that high protection against UV radiation can also be achieved by combining the processes of forming protective coatings from TiO_2 or ZnO nanoparticles with the dyeing of fibrous material, and by forming bionanocomposites containing these nanoparticles and biopolymers on fibers [84,89,92,94].

It should be noted that many researchers have solved the problem of forming multifunctional coatings on fibrous materials based on the use of TiO_2 and ZnO nanoparticles that give textiles various properties at the same time; for example, antimicrobial properties and photoactivity, or photoactivity and the ability to protect against UV radiation, etc.

6. Hydrophobic Properties

Water-repellent properties (hydrophobicity) are important for a number of household and special purpose fabrics. It is known that giving a high hydrophobicity to a fabric, as well as to any solid [202–206], can be achieved through the combined action of two factors. The first factor is a decrease in surface energy, by changing the chemical composition of the surface. This goal is achieved either by applying a substance with a lower surface energy (hydrophobizer) to the fiber, or by forming a low-energy coating directly on the fiber using admicellar polymerization or fluorination. The second factor is the texturing of the surface, to give the material a multimodal roughness.

The generally accepted criterion for assessing the non-wettability of materials is the water contact angle, which exceeds 90 degrees for hydrophobic materials and 150 degrees for superhydrophobic materials [202]. To characterize the wetting, the value of the drop sliding angle is also often used. However, fibrous materials are complex capillary-porous systems formed by cylindrical fibers or filaments of different chemical composition, different thickness, weave, and density. Therefore, first of all, the drop sliding angle is determined by the features of the surface structure of the fibrous material. Therefore, to assess the degree of hydrophobization of textiles, the use of this indicator is impractical.

It should also be noted that for fibrous materials, the water contact angle is a metastable indicator, since over time water begins to penetrate into the pores and capillaries [207]. Therefore, the most important characteristic of the degree of hydrophobicity of fibrous materials is water absorption, which is estimated by the amount of water retained by the fabric sample after it is completely immersed in liquid for one hour [3,207–209]. To achieve the low water absorption of a fabric, it is necessary that water does not penetrate the hydrophobic coating. This can be achieved in the case of the formation of a continuous coating on the surface of fibers with a minimum number of defects.

The requirements imposed on the consumer properties of finished products significantly complicate the solution of the problem of giving water-repellent properties to fibrous materials [3,207,210]. In particular, it is necessary that the fabric retains the ability to "breathe" after hydrophobization, which is characterized by high values of air and vapor permeability. Therefore, the coating formed by the hydrophobizer should be applied only to the surface of the threads, without occupying the space between them. The fabric should not acquire too high stiffness after hydrophobization. This dictates additional requirements for the rigidity of the coating based on the hydrophobizer, which characterizes its plastic properties [211]. Another prerequisite is also the stability of the achieved effect to intensive operational influences, such as friction, washing, and chemical cleaning; that is, the adhesion of the coating to the fibrous material should be high [3,202,207,212–214]. Thus, in order to obtain a high-quality hydrophobic fibrous material, it is necessary to form a well-fixed, friction-resistant, continuous, and defect-free coating with low surface energy on the surface of each fiber.

Coatings based on fluorinated polymers are characterized by the lowest surface energy. In [215,216], the formation of coatings with high hydrophobicity based on polytetrafluoroethylene is described, while in [217,218], coatings based on fluoroalkylsilanes were developed. The high chemical inertia, insolubility, and a number of other properties of fluoropolymers greatly complicate the formation of coatings based on them. The use of substances with a shorter chain length (fluorinated oligomers) as materials for hydrophobic coatings is more technologically advantageous [219]. When applied from solutions, some of these oligomers are capable of forming coatings on the surface of filaments with properties similar to those of polytetrafluoroethylene [220,221]. Fluorinated oligomers, obtained either by thermal degradation of fluoropolymers [207,222–227] or by synthesis from fluoromonomers [3,207,211,221,228–232], are the basis of such coatings. The use of organosilicon compounds as hydrophobizers also significantly reduces the surface energy of fibrous materials [233–236]. Hydrophobic fluorinated coatings can also be formed on fibrous materials, without the use of hydrophobizers. One of the ways to create such coatings is admicellar polymerization, which is a surface analogue of emulsified polymerization and allows the formation of long-lived hydrophobic coatings on the surface of the fibers [237,238]. Another way to impart water-repellent properties to fibrous materials without the use of a hydrophobizer is fluorination based on the interaction of elementary fluorine with molecules of a fiber-forming polymer [239]. As a result, a highly fluorinated surface layer, with properties similar to those of perfluorinated polymers, is formed [240–246].

The application of hydrophobizers is usually carried out using chemical and physical methods. The authors of [214] proposed dividing these into wet chemical and dry physical methods. Wet chemical methods include dip-coating methods, wet chemical etching; chemical bath deposition, electric-field assisted etching/deposition, and spray-coating, etc. Dry physical methods include chemical vapor deposition and plasma etching processing, etc. The researchers determined a strategy for the formation of the multimodal roughness of a coating, choosing the method of applying a hydrophobizer.

One of the most common strategies is to form a micro/nanoscale structure of nanoscale particles on the surface of a fibrous material, with further application of hierarchical structures of a hydrophobizer to the surface [247–250]. In a number of works, it was proposed to first modify nanoparticles with a hydrophobizer and then fix them on a fibrous material [251–255]. Hydrophobizers are also used, which themselves form hierarchical structures on the surface of the fibrous material [256,257]. The use of such a strategy makes it possible to achieve high hydrophobicity, but it also has some disadvantages. In particular, the coating formed on the basis of nanoparticles obtained mainly by sol-gel synthesis is quite thick; its thickness is several hundred nanometers [173,258]. The hierarchical nanostructures obtained on the basis of nanoparticles have insufficient mechanical strength and resistance to friction and washing [202,214]. This leads to the use of crosslinking agents [259] and is accompanied by an undesirable increase in the rigidity of the hydrophobic fabric.

In addition, the presence of large formations of nanoparticles on the fiber surface does not allow using the natural advantages of fibrous materials that they possess due to their structure. Fabrics and nonwovens are formed by cylindrical fibers and threads, which are characterized by a higher water contact angle than flat films of the same chemical composition [260]. In addition, in previous works [223,224,261,262] it was shown that fabric has a multimodal roughness, due to its complex weave. These factors create favorable conditions for the hydrophobization of fibrous materials and contribute to the achievement of higher water contact angle compared to the treatment of films of similar chemical composition with the same hydrophobizer. However, for their implementation, it is necessary that the coating formed by the hydrophobizer be ultrathin. In this case, the coating will reflect the microrelief of the fibrous substrate, acquiring a similar roughness. In [207,222–232,263,264], such coatings characterized by high water contact angles were formed from solutions of fluorine-containing hydrophobizer in organic solvents and supercritical carbon dioxide; in [265] by plasma spraying, in [266] by combining electric spinning with chemical vapor deposition of the coating, in [239] by chemical etching, and in [267] by electrochemical deposition. As shown in [222–232,268], the degree of hydrophobicity achieved can be further increased by using the methods of preliminary activation of fibrous materials, which increase the roughness. However, the technique of increasing the nanoroughness of a fibrous material by creating hierarchical nanostructures based on nanoparticles, which was used in the deposition of thick layers of a hydrophobizer, is ineffective in the formation of ultrathin coatings [269].

A promising but practically unexplored direction in the creation of fibrous materials with high hydrophobicity is to increase the water-repellent properties of the fabric by increasing the density of its structure, varying the density of the weave, selecting fibers of the desired diameter, etc. [270–272].

7. Conclusions

Fibrous materials with antimicrobial activity, photoactivity, UV protection, and hydrophobicity can provide a higher level of comfort and safety for consumers [273]. It should be noted that the possibilities for imparting special properties to materials (including fiber-based ones) using surface modification have significantly expanded in recent decades, due to the achievements of nanotechnology [210].

It is clear that the processes of imparting special properties to fibrous materials are subject to the theoretical laws common to materials of all types, and the effect achieved in all cases should be long-term. However, there are many additional, special requirements for the qualitative characteristics of textiles with special properties. This is due to the specifics of the structure of fibrous materials, their scope of application, and special consumer characteristics. Even after various treatments, they must retain good hygienic properties, softness, elasticity, drapery, etc. Textile materials are subjected to intense abrasion and frequent washing or chemical cleaning during operation. In this review, we have focused on the compliance of fibrous materials with special properties with the specific requirements imposed on them; we have tried to describe the characteristic methods for textiles for assessing the consumer properties of materials.

In this review, only the antimicrobial, photoactivity, UV protection, and hydrophobicity properties of fibrous materials were considered. Of course, the special properties of fibrous materials needed by the consumer are not limited to this list, and it could be significantly expanded. In addition, the assignment of each of these properties to fibrous materials was considered separately, although currently there is a tendency to obtain multifunctional fibrous materials with a complex of special properties. A detailed analysis of the influence of coating formation methods on the properties of multifunctional fibrous materials would also be of interest. However, consideration of these issues is beyond the scope of this review and is a task for the future.

In addition to experimental research methods, mathematical modeling methods are of great interest for predicting the properties of fibrous materials with various types of coatings. Currently, the modeling of a number of mechanical and special properties of fibrous materials is being developed. In particular, there are known works on modeling the elastic properties of carbon fibers coated with carbon nanotubes [274], as well as the mechanical properties of conventional and reinforced knitted products [275,276]. In addition to mechanical properties, a number of other characteristics are modeled; for example, the thermal conductivity [277], electrical conductivity [278,279], and magnetic properties [280,281]. Studies have been carried out in the field of the interaction of fibrous materials with water [280,281]. The total number of studies on the properties of modified fibrous materials based on mathematical modeling remains small. However, their development and the comparison of calculated and experimental data will allow giving fibrous materials the set of properties necessary for the consumers of the future.

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