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REVIEW

Biofunctionalization of textile materials by antimicrobial treatments: a critical overview

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Abstract

The current paper approaches a novel and interdisciplinary issue concerning the bioactive treatments -antimicrobial, antibacterial and fungicide- in the field of textile industry. Implementation of these modern processes will result in the functionalizing of the textile materials, to the benefit of both clothing comfort and medical therapy. In this paper, issues such as knowledge of the newest antimicrobial products, state of the art of antibacterial and antifungal processes and technologies, possibilities of the improvement of bioactive performances and properties, as well as new ways of evaluation and testing of antimicrobial treated textile products, have been highlighted.

Key words: antibacterial, antifungal, bioactive, textiles, testing, properties, performances

Introduction

Functional textiles and clothing provide the expected traditional properties, e.g. look, social identification, attraction, protection against cold, easy-care, as well as some new properties and functions of thermoconducting, deodorant, avoidance of unpleasant odors, antibacterial and antifungal protection. These new functionalities can be obtained by new bioactive treatments applied on textile materials during both the manufacturing stage of raw material and applying of some antimicrobial products during their finishing treatments [1, 2, 3, 4].

The textile material activity and the bioactive effect resistance depend on both binding type between the bactericidal product and fiber polymer, and chemical nature of the used antibacterial agent.

Due to rapid progress, new antimicrobial agents and improved polymer substances, such as copper and silver compounds, chitosan, triclosan, quaternary ammonium compounds, polymeric phosphonium salts, polymeric biguanides, N-halamin compounds and antibiotic drugs were investigated [5, 6, 7].

Providing antimicrobial properties to textile materials has mainly two directions/applications: protection of materials against pathogenic microorganisms and therapeutic and prophylactic actions.

Durability and performance of the new properties induced to the textile materials are determined to evaluate the efficiency of the antimicrobial treatment and the capacity to increase the added value of the product (increased quality, customer perception, performance and comfort properties, specific protection, etc.).

Technology of antimicrobial treatment

In order to obtain textile materials with antimicrobial performances, the following procedures are used: *(1)* impregnation of the fibrous material with a solution, suspension or emulsion of the bactericidal (fungicidal) product; *(2)* padding of an antimicrobial product, from its soluble state into an insoluble one on the fibrous material; *(3)* binding of an antimicrobial product on the fibre through chemical bonds (ionic, coordinative, covalent); *(4)* immersion of a bactericidal product either in the spinning solution or melt, during preparation of the chemical fibers.

The most adequate procedure is considered the binding of the antimicrobial product on the fiber through chemical bonds, which is achievable under industrial conditions allowing the manufacturing of some biologically active textile materials and fabrics with durable properties under repeated washing cycles.

The antimicrobial agent must show a selective action against different microorganisms and can be applied on textile supports by means of surface treatments or by their embedding either into the solution or in polymer melts.

Chemical treatments on the surface represent an efficient solution favorable to the bioactive effect, since the active surfaces are placed on a large contact surface. Durability of the treatment to washing and friction depending on the bonds type established between the fiber and the active substance used at the treatment should be considered [6, 8]. The treatment efficiency can be improved, using application of the above mentioned products by means of grafting techniques or reactive resins. Among the techniques belonging to the new generation of treatments, radiochemical grafting allows obtaining of materials with new properties, starting from the standard materials and functional monomers destroying several microorganisms (biocides). Grafting is a technique of modification and functionalization of polymers, focused on improving their native characteristics or adding specific properties to them.

Bulk treatments are performed by embedding the active products into the polymers, before the spinning process. Success of the treatment depends on the existing chemical compatibility of the active compound and both the polymer structure and the applied spinning procedure. In order to obtain a good distribution of the above-mentioned compounds into the core fibers, it is important to apply substances which can be dissolved or dispersed into the polymer [6, 7]. Since not all the active molecules are compatible with the spinning process conditions, a selection of them according to the parameters of fibers manufacturing is required. In order to be active, a substance embedded in the textile fiber core should slowly migrate to the surface and should not modify the initial characteristics of them, e.g. the fibre section shape.

In order to obtain some antimicrobial wool textile materials, methods based on binding of a bactericidal product to functional groups of the fibrous material through chemical links are used. Chemical binding of biologically active products on wool fibers is performed either by synthesis of grafted copolymers (bactericidal products bind to the wool keratin) or by applying antimicrobial dyes (acridines, aminoacridines, quinones, methylene blue, etc.). The fabrics obtained by wool grafting demonstrated good ionic change properties [9, 10].

A special field in the technology of antimicrobial treatment represents the production of new bioactive biocompatible polymers by cellulose chemical modifications, leading to insoluble bioactive polymers. The main reactions of cellulose chemical modifications consist of oxidation, etherification, esterification, halogenation, synthesis of graft copolymers and drugs (antibiotics) grafting. A wide range of polymers with pharmaceutical, medical and biological applications has been obtained this way [8, 11, 12].

A biotextile product with multiple or specific destinations, or a fibrous system exposed to therapeutic substances is considered a biologically active fibrous material. This is usually a complex composition, having a biologically active agent embedded into the structure and bound by various types of chemical or physical bonds or a simple deposition, finishing preparation, grafting [8, 11, 12].

The fibrous system exposed to drugs is activated in contact with wet environments (air, water, skin, blood, etc.), and the biologically active compounds are released. This process takes place at the surface of the organism. Thus, the textile material become drugs deposit. Antimicrobial biotextiles have the widest biomedical application by efficient reduction of bacteria, fungi or viruses contamination [13].

Considering aspects such as chemical action, impact on humans and environment, durability, costs and interaction mode with various microorgansims, antimicrobial products are very different and are used in order to reduce or eliminate processes such as deterioration/degradation, decolouring, odor and user's health [8, 12].

Substances inhibiting or killing microorganisms are: a) *organic compounds containing zinc, tin, silver ions*, b) *chlorine derivatives of phenols* showing a well-known bactericide action: pentachlor-phenol (Preventol PN); dichlor-bis-phenolmethane (Preventol GD, Germocide); hexachlor-bis-phenol-methane (Triclosan, Irgasan DP 300);dichlor-dihydroxy-diphenil-sulphide, and c) *ammonium salts* [2, 3].

The most used chemical substances for bioactive treatments of textile materials are shown in table 1.

Table 1. Antimicrobial compounds used in biofunctional treatments of textiles.

Type of antimicrobials	**Representative examples**	**Observations**
Antimicrobials for controlled release	Copper naphthenate, copper-8-quinolinate, organo mercury compounds	strictly regulated because of their toxicity and potential for environmental damage
	Tributyl tin oxide, dichlorophene, 3-iodopropynylbutyl carbamate	show very broad spectrum of activity against bacteria and fungi, but suffer from application and durability problems
	Benzimidazol derivatives, salicylanilides and alkylolamide salts of undecylenic acid	can improve durability to laundering particularly effective against fungi
	Formaldehyde, formalin	released in small amounts from easy-care and durable press finishes, small antimicrobial side effect
	Triclosan (2,4,4'-trichloro-2'-hydroxydiphenyl ether)	effective against most bacteria, poor antifungal properties
	Quaternary ammonium salts	effective antibacterial agents, but are limited as textile finishes
	Organo-silver compounds and silver zeolites, silver ions	used for fibre modification, an alternative to the antimicrobial finishes with high permanence [3, 14]

Bound antimicrobials	Octadecylaminodimethyltrimethoxysilylpropylammonium chloride	can be applied by exhaust or continuous methods to form a siloxane polymer coating on the fibre surface
	PHMB (polyhexamethylene biguanide)	can also be either pad or exhaust applied and has the proper molecular structure to bind tightly to fibre surfaces, still be an effective antimicrobial.
	Chloramine produced in the fibre when methylol-5,5-dimethyldyantoin reacted with cotton and then treated with hypochlorite	can function as renewable antimicrobial agent by continued treatment through bleaching and washing after reacting with bacteria
	Chitosan	may be applied by micro-encapsulation or reactive binding of cellulose. The advantages of antimicrobial finishing include high absorption, moisture control, the promoting of wounds and biodegradable properties [3, 10]

The most frequently used antimicrobial technology is based on unique silver ions [1,15], system Docair [9], antibacterial treatment of PES biofunctionalised knittings [16], new techniques of antimicrobial protection with drugs. Considering the latter technique, experiments on gentamycin-sulphate imobilization of polyacrylonytril fibre showed antibacterial properties both on Gram negative (*Escherichia coli*, *Pseudomonas aeruginosa)* and Gram positive bacteria (*Staphylococus aureus)***,** as well as on some fungi [4, 12]. Other drugs, such as streptomycin and kanamycin have been immobilized on cellulosic supports [2, 5, 17].

Evaluation of antimicrobial activity of treated textile materials

Two types of antimicrobial testing methods are mostly used.

The first method is based on agar zone inhibition, and consists of the immersion of treated material in an agar culture medium containing inoculated microorganisms (bacteria or fungi). It is standardized by standard EN ISO 20645/2004, which set up a method for determining the effect of applied antimicrobial treatments on woven and knitted textiles and relatively new ISO/DIS 20645. The ISO 11721 is a burial test. The antibacterial effect can be defined as an inhibition of bacterial growth under favourable conditions [18, 19].

The second method is based on bacteria number testing and consists in determination of bacteriostatic/fungistatic activity of the treated material which has been sterilized and inoculated with microorganisms, by numbering the bacteria/fungi colonies.

The Technical Manual of the *American Association of Textile Chemists and Colorists* (AATCC) presents a number of test methods that are useful for evaluation of antimicrobial finishes on textiles (Table 2).

Table 2. Antimicrobial test methods used with textiles (Schindler et al., 2008) [3].

Test methods	Observations/description
Antimicrobial activity of textile materials: parallel streak method; test method 147 (agar plate test)	Rapid qualitative method for determining antibacterial activity of treated textile materials against Gram-positive and Gram-negative bacteria. Treated material is placed in nutrient agar inoculated with test bacteria. After incubation, antibacterial activity is determined by inhibition zones on and around the textile.
Antibacterial finishes on textile	Quantitative method for determining the degree of antimicrobial activity of treated textiles. The amount of bacterial growth in inoculated and incubated textiles is determined through serial

materials, assessment of: test method 100	dilutions and subsequent inoculations of sterile agar.
Antifungal activity, assessment on textile materials: mildew and rot resistance of textiles; test method 30	Four methods: method 1 involves testing fabric properties after burial in soil that contains fungi; method 2: cellulose fabric is exposed to *Chaetomium globosum* in an agar plate and the subsequent growth visually determined; method 3: exposes textiles to *Aspergillus niger* in an agar plate and visually determines any fungal growth; method 4: uses a humidity jar to expose textiles to mixture of fungi spores. Any growth on the textile is visually determined.
Antimicrobial activity assessment of carpets;test method 174	Methods are given for the qualitative and quantitative determination of antibacterial activity and qualitative evaluation of antifungal properties of carpet samples using procedures and materials similar to those in the above test methods.

A two steps test has been developed at the *Institute of Research for Hygiene and Biotechnology in Textiles* - Hohenstein Institute from Germany, in order to quantitatively verify the unpleasant odors caused by different microorganisms [19].

Properties and performances in textiles biofunctionalization

Studies on wool materials were performed with a new class of bis-quaternary ammonium surfactants: NN'-bis (N-dodecyl-N,N-dimethyl glycine) cystamine dihydrochloride (DARK) and (N-dodecyl-N,N-dimethyl glycine) 1,4-diaminobutane dihydrochloride (DABB) which belong to the new generation of surfactants known as *gemini* quaternary ammonium compounds or *bis-Quats*, as possible antibacterial agents [20, 21]. Strains of *Bacillus pumilus*, *Staphylococcus aureus*, *Escherichia coli* and *Pseudomonas aeruginosa* were used. Antimicrobial activity was in strong correlation with the concentration of the new compounds used for the treatment (0-5% wt). Moreover, no damage to the wool fibers was observed, suggesting that the property of tensile strength was not influenced [20].

The experimental results performed on knittings made of polyamide/elastan treated with gentamycin-sulphate and the essential oil from *Picea abies* showed that the textile product can be successfully used for medical applications, as compressing material. The gentamycin-sulphate/essential oil fixed in the knitt structure by inclusion has performed a bioactive textile material.

The applied inclusion procedure is a physical-chemical modification by which antimicrobial molecules are embedded in the polymeric matrix of the polysaccharide gel based on chitosan, which is subsequently applied as additive to the investigated knit [22, 23]. The results showed that the antimicrobial treated knitted fabric expressed a wide range of bactericidal/bacteriostatic, and fungicidal activity on various strains of microorganisms, e.g. *Staphylococcus aureus*, *Escherichia coli, Klebsiella and Candida albicans*. The test samples treated with gentamycin-sulphate showed a wider antimicrobial activity compared to fabrics treated with the essential oil from *P. abies*. In case of *Candida albicans*, a total inactivity was noticed for both treatments. The influence of the antimicrobial treatments on the tensile strength and tensile strength and their corresponding elongations, stiffness and elasticity of the chosen test was examined and an increasing of strengths by 14% has been reached. Regarding the bending elasticity, it was shown that the values of the stiffness as well as the bending modulus are increased 3 times lengthwise and 7 times widthwise compared to the untreated fabrics, while tensile elasticity remains almost unchanged. Through the variety of structural parameters of the knitting good, by selection of an appropriate technique of producing it, of the adequate parameters for the antimicrobial treatment, a high performance textile material may be obtained, producing pressure on the textile material/skin interface, which could be useful in compression therapy [22, 24].

Furuta et al. investigated the hinokytiol or β-thujapilycine as a model component for bioactive substance inclusion and releasing [25]. Hinokytiol extracted from wood [26] showed activity against aerogenes bacteria, fungi and insects. A high quantity of MCT-β-CD was easily fixed on a Washi Japanese paper tissue, in order to include the antibacterial component. The releasing rate was strongly influenced by the relative humidity of the environment. Wang and Cai included antibacterial agent myconazol-nitrate on a cellulosic fabric grafted with MCT-β-CD [27].

Two other antibacterial agents, neomicyne and tryclosan have been used as inclusion compounds with β-CD, in order to obtain antibacterial thin layers, fibres and fabrics. When these inclusion compounds (CI) with β-CD were processed by melting at biodegradable/bioabsorbable films and fibers from poli-ε-caprolactonă (PCL) and poly-lactic acid (PLA), growth inhibition of *E. coli* has been noticed. Although the concentration of tryclosan in CI is about 10% of the tryclosan thin film, the cotton laminated/coated fabric with PCL films, with reduced melting point, containing tryclosan-β-CD CI, inhibited the growth of *E. coli* as efficiently as pure tryclosan [24]. When PLA fibers have been filated from PLA melt containing a small quantity of CI neomicyne-β-CD, it has been noticed that small agglomerations of fibers inhibited the growth of *E. coli.* Thus, the potential of these inclusion compounds has been showed, in order to perform pemanent antibacterial, biodegradable and bioabsorbable sewages.

The recent use of cyclodextrine in the inclusion procedure showed effects of antimicrobial protection of cynnamic derivatives as guest-molecules, of coffee acid, ferulic acid and ethyl-ferulate, on cellulose supports [17, 28]. The antimicrobial efficiency of fabrics treated with cynnamic derivatives by means of MCT-β-CD has proven satisfactory against four microbial strains: *Escherichia coli*, *Staphylococcus aureus, Pseudomonas aeruginosa* and *Candida albicans*, in particular against *Staphylococcus aureus*. Through inclusion of cynnamic derivatives, sterile surfaces have been created promissing to be useful in medical textiles field [17, 28, 29].

Other important properties need to be investigated in treated materials, such as moisture, dynamic water absorption rate and water transport rate, air permeability and deodorization activity.

Chitosan prevents the growth of several microbes including Gram-negative and Gram-positive bacteria [10,30]. It has been shown that chitosan oligomer is most efficient on *Proteus vulgaris,* showing a reduction growth rate higher than 90%, at a level of 0,01%. It is well-known that *Proteus vulgaris* decomposes rapidly ureea into urine and then into NH_3, causing some unpleasant odors and consequently dermatites. Chitosan treated fabric showed a 85-95% reduction rate of NH_3, resulting in a higher deodorization rate (30-40%), compared to the untreated fabric.The antimicrobial activity of chitosan treated fabric was tested against *K. pneumoniae* and *Ps. aeruginosa* up to 1% concentration. The oligomer was not efficient against these strains, showing only 30% reduction rate [30].

The bioactive treatments also lead to positive modifications in terms of both tensile and tensile strength and elongation of treated fabrics. Consequently, both the tensile and tensile strength values, as well as elongation increased, longitudinally and transversally in the investigated material. In case of elasticity, it has been shown that both durability and flexibility modules values increased. Taking into account the tensile elasticity, the type of treatment showed no influence. The strength module remains almost constant, regardless the applied treatment.

Chitosan treatments lead to improved air permeability. When chitosan concentration was below 0.5%, air permeability of HMW chitosan treated fabrics was optimal, but when chitosan concentration was over 0.5%, that of LMW chitosan treated fabrics was better improved. Deodorization rate increased over the time with higher chitosan concentration. Antibacterial activity was excellent when the HMW chitosan was used, regardless the type of bacteria or concentration of the chitosan. It was shown that it is better to use chitosan with a high molecular weight in order to improve the antibacterial activity and the most important performance of sanitary non-woven fabrics [10, 31, 32]. As a result of some complex biomedical investigation performed, it has been noticed that this oligomer induces certain reactions on skin. This aspect constitutes an advantage, especially for its applications on fabrics that directly contact the skin.

Soluble chitosan-oligomer was prepared for polypropylene non-woven fabrics finishing, treated by padding, in order to develop an antimicrobial activity (PD – 1814, DD deggree of deacetylation – 84%). The oligomer showed high antimicrobial activity against *Proteus vulgaris, Escherichia coli* at 0-0.1% and 0.05% respectively, presenting a reduction rate over 90% [30].

The tensile strength of the treated samples was smaller than the control. The fabrics became rougher and less air permeable with increased treatment agent concentration. A liniar relationship of air permeability on the chitosan concentration was noticed. A decrease in the comfort parameter has been observed for the chitosan treated sample compared to the control one. This phenomenon is explained by the fact that chitosan molecules deposite on fiber surface in order to block the pores, more than penetrate the polypropilene fiber and consequently, modify the porous structure of the material. Decreasing of the air permeability means in fact a decrease in the comfort from the hygienic point of view [30].

Recently, cetyl pyridine chloride – as ammonium quaternary salt, has been used directly for the antimicrobial treatment with durable properties of acrylic fabrics [33, 34]. During antimicrobial finishing treatments of acrylan fabrics, some physical properties modification were noticed: ellongation strength and yellowing of the finished fabrics. The yellowing and the tear strength loss of acrylic fabrics show that cetyl pyridine chloride plays a major part in reaction with the polymer, under alkaline conditions [33]. The direct antimicrobial finishing of acrylic fibers may have applicability both in sport wear and biomedical textiles field, by providing some aditional functions of odor control. The antimicrobial functions assigned to cetyl pyridine chloride embedded on fabrics and their durability formed through ionic bonds among the anionic groups on fiber and cationic cetyl pyridine chloride depend on the quantity of the agent embedded during the finishing treatment and which release according to a slow releasing mechanism. The agressive alkaline conditions of the treatment may cause negative impacts both on mechanical properties and chromaticity of the textile material, due to a potential alkaline reaction induced by the acrylic polymers, resulting in C=N conjugated systems [33, 35, 36].

PES and PA fibres have been selected for a similar research, due to their increasing presence in non-implantabile medical/health-care textiles (various bandages, gyps, etc.), hygiene/health care products (uniforms, surgery gown, mask, cap, wear, etc.) and domestic clothes. The investigation aimed to highlight the possibility of using corona treatment in order to activate the fiber surface, which can facilitate the loading of NP silver from a colloid on PES and PA fabrics, thus improving their antibacterial properties. Additionally, the wash resistance of the antibacterial effects on Gram-positive bacteria, such as *Staphylococcus aureus* and Gram-negative bacteria, *Escherichia coli* was investigated [15].

The antimicrobial effect of a special type of fibers produced by Isfahan Poly Acryl Plant on three species of bacteria *Staphylococus aureus, Staphylococus epidermis* and *Staphylococus lugdumensis* isolated from 96 samples of hand and foot skin micro-flora was studied. The sensitivity of bacterial strains to various antibiotics and beta-lactamases was studied, as well. In order to compare the effect of the antimicrobial agent of fibre with that of the known Penicillin G, minimal inhibitory concentrations (MIC) of both antimicrobial agent of fibre and Penicillin G have been tested [5]. The interaction effect of these two antimicrobial agents and their fractional inhibitory concentration (FIC) on selected microorganisms has been studied using the checkboard method.The experimental results showed a significant effect of the antimicrobial fibre (treated with different concentrations of antimicrobial agent: 30%, 60%, 100%) on *Staphylococus aureus* species, after 24 hours. Moreover, despite the high level of MIC for Penicillin G on these bacteria (8-256 μg/mL), the MIC of pure antimicrobial agent of fibre at a level of 10^{-4} μl/mL, caused growth inhibition. The interaction of these two antibacterial agents on the selected strains was evaluated as synergic effects. Obtained data suggest that human skin flora can only be influenced where the skin is in direct and permanent contact with the treated fibre [5, 37].

Conclusion

The recent interest in biofunctional textiles is mainly directed to the use of specific textiles in medical therapy and prevention of deficiencies in wearing comfort. A continuous demand for bioactive antimicrobial finishing has occurred recently, due to the increased health interest of the consumers.

Antimicrobial activity of materials treated with antimicrobial products is tested against different strains of bacteria. Furthermore, high performance properties of the treated textiles are measured, including tensile strength and elongation, roughness, air and liquid permeability, color modification.

The releasing time and rate of the drug embedded into the textile materials as antimicrobial agent, can be controlled. This phenomenon takes place within a long period of time, thus the textile materials would have a minimal toxicity, compared with standard methods of exposure to drugs action.

Application of new antimicrobial agents makes textile materials useful for prolonged therapeutic treatment without the genesis of germs.

Microbial growth control in textiles is of great significance from the point of view of hygiene and increase in fiber duration known to decrease due to biological degradation.

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