Potential Application Techniques for Antimicrobial Textile Finishes

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Abstract

The usage of antimicrobial textile products aims to prevent microbial contamination and improving environmental hygiene. Antimicrobial agents are applied to textiles fabric that inhibit and kill the microbes when they come in contact with finished surface. Antimicrobial textiles are being utilizing continuously hygienically, where textiles surface is anticipated to contact with microbes. Owing to vastly diverse applications of antimicrobial textile products and intended usages, significant progress in developing novel application methods of antimicrobial agents has been accomplished in recent years. The efficacy of antimicrobial property also majorly depends application method beside nature and concentration of antimicrobial agent. Various antimicrobial agents require different application method on to textiles where they can maximum antimicrobial action. This article critically summarizes the potential applications techniques for finishing textiles with antimicrobial agents.

Keywords: Antimicrobial finishes; Application techniques; Testing; Performances

Introduction

Human beings always have learned to make textile products for their use by processing raw materials, dyeing, and finishing processes. Now in recent decades, both natural and synthetic fibers specially functionalized for smart applications are of the great importance for textile researchers to develop functional textiles. Such functional textiles can perform particular function for which these textiles are design, for example, anti-microbial, superhydrophobicity, UV protection, flame retardency, anti-smug, etc. Among these properties, capability to inhibit and kill the microbes is quite important for technical clothes. Textile products finished with antimicrobial functionalities are capable to resist and kill infectious microbes such as viruses, bacteria, fungi, spores etc. The synthesis and development of application methods of novel antimicrobial agents on to textile fabric has gained researchers interest owing to increased trend of continuous consumption of antimicrobial finished textiles. The potential antimicrobial agents have critically discussed in my previous article [1]. This study summarizes the efficient application methods for antimicrobial textile finishes.

Pre and post application testing

Various testing methods are employed before and after application of antimicrobial finishing to evaluate the antimicrobial functionalities. Such testing methods are as follows

a) Safety testing of antimicrobial agent
b) Efficiency testing of antimicrobial agent
c) Durability testing after application
d) Resistance risks

Human skin must directly have exposed with antimicrobial textiles during usage, so it should be assured that finished textile will not irritate or harm the human skin. There must evidence that human skin is safe during use of antimicrobial textiles as antimicrobial agents do attack to bacterial cells and they will not harm the human skins cells. Several biological skin safety testing methods are available, such as DIN EN ISO 10993-5 (test for in vitro cytotoxicity), DIN EN ISO 10993-10 (skin irritation). Various microorganisms are populated in human skin which protect it and are collectively called as resident skin flora. The examples are propionic bacteria, yeasts, micrococci, coryneform bacteria and staphylococci. Pathogens infect and harm the humans when a disturbance of skin flora occur. The testing of antimicrobial agent for influence of resident skin flora is so necessary because finished agents cannot differentiate between beneficial microbes and infectious pathogens and thus they may damage resident skin flora too. An artificial skin model cultured with Staphylococcus epidermidis and Micrococcus luteus is used for testing resident skin flora in laboratory method.
The finished textile products should exhibit validated antimicrobial effect regarding registration, marketing and customer satisfaction. Various standardized methods are available to test effectiveness of antimicrobial textiles including qualitative and quantitative evaluation with different experiment setups. The most important methods are mentioned below.

**Antifungal assessment**
- a) Quantitative evaluation (DIN 14119)
- b) Semi quantitative evaluation (AATCC 30)

**Antibacterial assessment**
- c) Shake flask test (ASTM E2149)
- d) Agar plate diffusion test (DIN EN ISO 20645)
- e) Parallel streak method (AATCC 147)
- f) Quantitative Evaluation (AATCC 100)
- g) Quantitative Evaluation (DIN EN ISO 20743)

Durability testing is another important evaluation regarding antimicrobial textiles products, as the antimicrobial efficacy should be continual for a long period preferably lifetime of finished product. However, the nature, concentration, consumption, stability and method of application of antimicrobial agent do greatly affect the durability of antimicrobial finished textiles [2,3]. Degradation, consumption reduce the concentration of agent and ultimately affect the effectiveness. The durability is evaluated in laboratory from many washing cycles and storage times while antimicrobial efficiency is to test before and after durability tests. A slight decrease in antimicrobial activity reflects high durability of finished textile while a low durability is revealed from significant decrease in antimicrobial activity.

Commonly, 50 washing cycles are employed to check remaining antimicrobial activity for durability testing. Various standard washing test methods are DIN EN ISO 6330 and DIN EN ISO 15797. The most test conditions for storage stability are 75% humidity; 40 °C; 3-9 months (accelerated aging) and 60% humidity; 25 °C; 12-36 months (real time aging). Pathogens may resist against the effect of antimicrobial agent by developing diverse resistance mechanism to destroy or neutralize the effect of agents. Thus, resistance risks should be precisely addressing to get maximum antibacterial performance of finished textiles product.

**Application Methods**

A few application method for antimicrobial finishing of textile fabric have been reported and are under development to be implement on large scale depend upon particular fiber type and antimicrobial agent. As far as natural fiber is concerned, the most commonly used method to finish surface with antimicrobial agent either binding antimicrobial agent covalently or other type of electrostatic interactions with fiber. Such antimicrobial finish may remain on surface of fiber or undergo leaching mechanism depending upon the interaction between agent and fiber. An extra advantage may utilize for imparting antimicrobial functionality to synthetic fiber where the active agent can be blend in precursor polymer prior to its extrusion for fiber formation. As the active agent become physically embedded in polymeric microstructure of fiber which leaches slowly with usage and thus provide best durability. Trevira is the best example containing silver-based antimicrobial agent embedded in polyester. Such advantage may also avail from semi-synthetic fiber such as regenerated cellulose fibers doped with silver-based antimicrobials prior to spinning process [4-9].

Surface finishing of fabric with antimicrobial agent is commonly employ by using pad-dry-cure, conventional exhaustion, padding, spraying or foam finishing for both synthetic as well as natural fibers. Other methods are polymer grafting of active agent during polymerization, crosslinking of active agent on to fiber surface via a suitable cross-linker, chemical modification of active agent for binding with fiber covalently, nano-scale core-shell particles, nano-scale colloidal solutions or hydrosols [10]. Among them potentially, antimicrobials are microencapsulated via emulsion polymerization or entrapping them in suitable sol-gel nanoparticles and applied to both natural and synthetic fibers [11-25].

Whatever the method is chosen for antimicrobial finishing, several requirements should meet by applied antimicrobial finish to accomplish optimal performance during its service time. A good antimicrobial finish should exhibit following major requirements.

a) Should not be toxic, irritating or allergic to human body
b) Should not affect or kill the resident skin flora
c) Should meet standards regarding compatibility test, e.g., sensitization, irritation, cytotoxicity
d) Should be compliance with government regulations
e) Should be efficient for a wide range of microorganisms
f) Should not affect adversely with quality of fabric, e.g., comfort, handling or strength
g) Should be compatible with other textile processes during textile treatments
h) Should be durable to laundering even after several wash cycles, drying or hot pressing
i) Should be durable to storage for long time
j) Should be eco-friendly and cost effective

**Sol-gel technology**

Sol-gel method is one of the most effective and simplest methods for developing various nanocomposites among all other fabricating techniques of nano-coatings. Antimicrobial agents applied to fabric via sol-gel method offer maximum antimicrobial power and durability of applied agent. Inorganic matrices (e.g, silica) act as supporting hosts for antimicrobial agent. The entrapment of biocidal agent with inorganic matrix depends upon extent of control on various stages of sol-gel processes. Advantageously,
these antimicrobial functionalized sol-gels can perfectly deposit on surface of fabric via electrospinning, spray coating, spin coating, dip coating, and pad dry curing methods. Most widely used application method of sol-gel is pad dry curing.

Sol-gel method is a technique for fabrication of variety of inorganic or hybrid inorganic-organic networks resulting in porous, glass or ceramics like material from sol-gel reactions of metal alkoxide precursors. Colloidal suspensions of solid particles in solvent called sol while gel exhibit solid phase framework surrounds a continuous liquid phase. Advantageously, room temperature is requiring for sol-gel reactions as gel cures at moderate temperature. Such simplicity and versatility in sol-gel method make it suitable for film formation with functional properties.

Hydrolysis, condensation, and polycondensation are fundamental reactions, which occur during sol-gel process. Figure 1 & 2 depicts principle steps of sol-gel process. Silicon alkoxides are most widely used alkoxides, e.g., tetramethoxy silane (TEOS) and teramethoxy silane (TMOS) but other metal alkoxide also follow similar reaction mechanism (i.e., zirconates, titanates, aluminates) [26-28].

Figure 1: Three stages of sol gel process.

Incorporation of water in silane solution under basic, neutral or acidic conditions results in hydrolysis process (Figure 3). Corresponding alcohol and Hydroxylated (silanol group) products are produced as the results of hydrolysis of silicon alkoxide precursors (Step 1). Hydroxyl group and unhydroxylated alkoxide group undergoes condensation reaction that results in appearance

Figure 2: Three reaction schemes involving in sol-gel process.
of colloidal mixture (sol) with elimination of solvent. Formation of three-dimensional crosslinking between sols as a result of polycondensation reaction increased the viscosity of solution gradually. The resultant product is called gel that is relatively porous and rigid produce from interconnecting sols. Various parameters of sol-gel process like nature and concentration of precursors, temperature and water to silane ratio, sol aging time and nature of catalyst do affect the properties of final gel formation. Both base and acid catalyzed gels forms structures with varying degree of crosslinks. Linear structure is formed from acid catalyzed reaction while crosslinked structure is formed from base catalyzed reaction (Figure 3).

![Figure 3: Polymeric distribution of the resultant gel as a function of acid or basic catalysis.](image1)

Use of nanoparticles has gained much popularity in recent decades owing to their decreased size and high surface to volume ratio that makes their surface highly active. One of the efficient methods to apply antimicrobial agent on textile fabric is to entrap them in different metal/metal oxide sol-gel matrices. This method has shown good efficacy and durability even after several washes. Examples are ionic silver or silver-based compounds in silica matrix [11,29-37], Zin based [17,22,23,38-40] and Titanium based [41-43].

![Figure 4: The mechanism of interaction of cotton fibers with TiO2, BTCA [35].](image2)
Microencapsulation

Microencapsulation is a rapidly growing method that have used for diverse applications specially in textile industry [24,25,45-50]. This process involves the formation of microcapsules having two parts core and shell. Figure 5 depicts the schematic view of core-shell microcapsule. Core contains the active agents which are very tiny particles of solid or droplets of liquid coated or surrounded by continuous film of polymeric material forming the shell as the whole. So, core is the interal phase contains active agents while shell is external phase which is the membrane, coating or wall material. Actually, microencapsulation provide the storing, seperating and pasaging of active materials at microscopic level. Microencapsules release the active agents gradually under the condition that have programmed for that particular microcapsules and achieve the particular performance. Amount of core-material and thickness of wall material decides the mechanism of release of active agents. The possible mechanisms that may involve the release of active agents from microcapsule are bursting of shell, biodegradation, dissolution of polymeric wall material, diffusion through wal, change in temperature or pressure, by friction, etc. A number of advantages that make microencapsulation technique prime to others methods of applications for antimicrobial finishing are as follows.

a) Convenient in handling of a large number of antimicrobial agents
b) Improved flowability, dispersibility and solubility is achieve for better processing
c) Improved availability and compatibility is gain for antimicrobial finishing
d) Shelf-life is impoved as some degradation side reactions are avoided
e) Sustained and controlled release of antimicrobial agent
f) Antimicrobial agents become stable for long period of time from external environments (evaporation, moisture, alkanility, acidity, heat etc) prior to use of finished fabric

Figure 5: SEM images of (a) unfinished cotton fabric, (b) unwashed cotton fabric fabricated by pad-dry-cure method, (c) unwashed cotton fabric fabricated by exhaustion method and (d) Zoom-in of image (c) [35].
Chitosan is a well-studied antimicrobial agent [51-57]. The schematic view of preparation of chitosan microencapsulation is depicted in Figure 6. The effective conditions or parameters that affect the yield of microcapsules are:

a) Controlled release behavior, surface morphology and particle size of microcapsules depend upon mass ratio of core ingredients and shell material

b) Speed of agitation prior to crosslinking reaction

c) Yield of microcapsules is proportional to the shell material concentration

d) Surface adhesion and shell strength of microcapsules are influenced directly by crosslinking time

(Figure 7); [58] Controlled release of antimicrobial agents for a prolonged durability even against daily use and after several wash cycles, is achieved by microencapsulation technology. For water in oil or oil in water medium, cocreation is a mature microencapsulation method [59-62]. Encapsulation of natural biocides through this technique is becoming popular in these days performing as multifunctional finishes that not only provides antibacterial functionalities but also serves as a fragrance.

Figure 7: Schematic diagram and SEM images of the chitosan/miconazole nitrate prepared from microencapsulation method [58].

**Conclusion**

Antimicrobial agents inhibit the growth of infectious microbes and kill them whenever microbes become in contact with them. These antimicrobial agents are being extensively applied to textile fabrics in order to use them hygiene purposes regarding human health issues such as, inhibiting microbes from spreading, control of infections, control of odor, healing of wound, etc. Various application methods of antimicrobial agents on to textile fabric surface have developed. The selection of suitable application method depends upon a number of parameters like nature and concentration of antimicrobial agent, type of textile fabric, etc. Microencapsulation and sol-gel methods are convenient and efficient methods that provide excellent efficacy and durability to antimicrobial finished fabrics. Advantagiously, these methods are also suitable for multifunctional finishing of fabrics that possess phase change, antistatic, anti-smog, self-healing properties.

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