



Multifunctional durable properties of textile materials modified by biocidal agents in the sol-gel process



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ABSTRACT

The studies were conducted on a cotton fabric modified by the mixture of tetraethoxysilane (TEOS) and triclosan (TC)–(2,4,4′-trichloro-2′-hydroxydiphenyl ether) or TEOS and quaternary ammonium salt (QAS) such as the hexadecyltrimethylammonium bromide in order to obtain biocidal (i.e. antifungal) and hydrophobic properties. The modified unwashed and washed fabrics were tested for the action of a mixture of five mould species which most often cause the decomposition of cellulose (*Chaetomium* sp., *Aureobasidium* sp., *Paecilomyces* sp., *Aspergillus* sp., *Penicillium* sp.). The hydrophobicity was examined by measuring the contact angle. The fabrics modified by TC-TEOS are characterized by a higher antifungal effectiveness, however, the durability of the modification can be maintained only when they are dry-cleaned. Silanes form a silica layer on the surface of textiles which results in increased hydrophobicity. These multifunctional textiles can find applications as materials for human health, e.g. insoles, wound dressings, protective clothing, filters.

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1. Introduction

Extensive use of materials based on natural fibers, particularly in the construction, transport (polymer composites containing natural fibers) and in the textile industry (clothing, wound dressing materials, filtration, textiles for the footwear industry) is aimed at endowing them with multifunctional properties. Due to such different fields of application, biocidal properties of textile materials constitute a part of their multifunctionality. Their failure could pose a significant problem when they are exposed to the action of harmful external factors. High humidity and temperature, insufficient air circulation result in enhanced growth of microorganisms, especially mould fungi [1].

These external factors affect, among others, fibrous materials used in filters [2]. However, the above problem concerns not only filters. For textiles used as medical materials of importance is also to impart to them antibacterial properties. Several techniques have been reported to immobilize antibacterial groups onto the textile surfaces, including tethering antimicrobial polymers introduced onto surface via chemical coupling reactions, surface-initiated polymerization of monomers having antibacterial moieties, synthesis of surface-tethered antimicrobial polymer brush via post-polymerization modifications [3–5] and immobilization of antibacterial compounds in silica matrix obtained by

hydrolysis and condensation of alkoxy silane-based sol-gel precursors [6–8].

The latter method is among the most popular ones because it enables the formation of a durable silica coating at room temperature and using standard devices applied to fabric finishing. Such a coating can act as a carrier for embedded functional additives (such as organic or biological compounds), improves mechanical properties of textiles, binds well to fibers and is stable against light, heat and chemical attacks [8]. Functionalization of textiles by inorganic sol-gel coatings has been reported in numerous papers by Böttcher and Textor [8–13], in which valuable information has been provided on conditions of conducting the sol-gel process and producing coatings with desirable properties. The sol-gel process is usually equated with obtaining silica sol by hydrolysis of tetraethoxysilane followed by condensation of silanols formed as a result of TEOS hydrolysis. However, during the aforementioned process one can additionally introduce an organofunctional trialkoxysilane that during the condensation incorporates into nanosol structure. There is also a possibility of incorporating into previously formed nanosol various compounds that are dissolved or dispersed in the nanosol and impart desirable properties to the final coating.

To obtain textiles with bactericide properties a modification with various derivatives is employed, e.g. with quaternary ammonium compounds [14–17], quaternary phosphonium salts [18,19], silver and gold nanoparticles [10,20–23], triclosan [24–26], *N*-halamines [27–32], chitosan and its derivatives [33–35].

A major problem is the attachment of these modifiers to fibers and this is why the possibility of applying organosilicon compounds or sol-

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gel technique solves this problem and at the same time imparts to a material additional qualities resulting from specific properties of silicon compounds and thereby such a modification of materials makes it possible to create multifunctionality.

In many studies conducted by researchers from different countries a very good antibacterial activity was obtained in the case of cotton fibers coated with silver nanoparticles and hexadecyltrimethoxysilane [36]. Moreover, the aforementioned modification has also resulted in superhydrophobic cotton textiles. Durable multifunctional properties are imparted to textile materials by various finishing agents, such as silicon compounds and triclosan. The cotton/wool and viscose/wool blended fabrics were padded twice in a single bath containing, among others, silicon softeners and triclosan as an encapsulated organic antimicrobial agent in order to obtain antimicrobial properties [24]. Antibacterial efficacy was obtained for *Staphylococcus aureus* and *Escherichia coli*. The microencapsulated antimicrobial agents such as triclosan and quaternary ammonium salt in the form of dodecyltrimethylammonium chloride were applied to wool fabrics [25]. In both cases a very good antimicrobial efficacy was achieved for unwashed fabrics and those washed 5 and 10 times. No bacterial growth near the edge or under the fabrics occurred and inhibition zone was observed for *S. aureus* and *Klebsiella pneumoniae*.

The vast majority of papers concern the modification of fabrics towards achieving bactericidal properties. However, it should be noted that materials with bactericidal activity can be obtained easier than biocidal systems showing the activity against a wider range of microorganisms including fungi. For this reason in the present study we have made an attempt at verifying the antifungal activity of triclosan and quaternary ammonium salt immobilized in silica matrix (the latter was prepared from tetraethoxysilane by the sol-gel process). The task of the siloxane layer formed during the process consists not only in the biocidal agent immobilization, but also in the stable attachment to fibres through the formation of covalent bonds. Its task also includes imparting hydrophobic properties. Hydrophobicity of fibres protects products against the penetration of moisture into the fibre structure thereby preventing from the formation of favorable conditions for the growth of bacteria and fungi. An additional asset of hydrophobic fabrics is their increased resistance to dirt which opens up a broad field for applications of such multifunctional fabrics. The hydrophobic properties of modified fabrics were determined by measuring the contact angle of water on their surfaces.

2. Experimental

2.1. Materials

The fabric was made of 100% cotton fibres and characterized by the surface mass of 158 g/m². Before the modification process, the fabric was bleached in a hydrogen peroxide bath.

Mould strains of *Aspergillus niger* van Tieghem, *Chaetomium globosum* Kunze, *Aureobasidium pullulans* (de Bary) Arnaud, *Paecilomyces variotii* Bainier and *Penicillium ochrochloron* Biourge were purchased from Pure Culture Collection of the Institute of Fermentation Technology and Microbiology, Technical University of Łódź, Poland.

Tetraethoxysilane (TEOS) was supplied by Unisil (Tarnów, Poland). Triclosan (2,4,4'-trichloro-2'-hydroxydiphenyl ether), hexadecyltrimethylammonium bromide, isopropanol, and hydrochloric acid were purchased from Sigma Aldrich.

2.2. Modification of cotton fabric

The modification was performed on a fabric made of 100% cotton fibres. To impart biocidal properties to the fabric, it was modified with hexadecyltrimethylammonium bromide (QAS) or triclosan (TC), formulae of which are shown in Fig. 1.

The above compounds were immobilized in a silica matrix prepared by the sol-gel process as a result of hydrolysis and condensation of tetraethoxysilane. The fabric subjected to the modification with QAS was firstly modified with silica gel and subsequently with a solution of QAS, whereas that subjected to the modification with TC was covered with triclosan-containing silica gel. Further studies were carried out on modified fabrics, both unwashed ones and those washed once and five times with water at 40 °C for 1 h.

2.2.1. The modification with hexadecyltrimethylammonium bromide-TEOS mixture

Tetraethoxysilane (10 vol%), distilled water (0.9 vol%), dibutyltin diacetate (0.9 vol%) and isopropanol (88.2%) were placed in a flask equipped with a magnetic stirrer and a reflux condenser. A 15-min hydrolysis process was conducted at room temperature and after that time the solution was transferred into trays in which the cotton fabrics were impregnated for 2 min. Then the excess of the modifier solution was removed by squeezing followed by drying the samples at 80 °C for 2 h. Afterwards, a solution of hexadecyltrimethylammonium bromide (5 vol%) in isopropanol (90 vol%) with the addition of water (5 vol%) was prepared in a flask equipped with a reflux condenser and stirred for 15 min at room temperature. The obtained solution was transferred into trays in which the fabrics modified at the previous stage were subjected to impregnation. After a 15-min process of modification, the excess of the modifier solution was removed by squeezing followed by drying the samples at 80 °C for 1 h and fixing at 130 °C for 3 min.

2.2.2. The modification with triclosan-TEOS mixture

Tetraethoxysilane (18 vol%), isopropanol (78 vol%) and 0.01 M hydrochloric acid (4 vol%) were placed in a flask equipped with a magnetic stirrer and a reflux condenser. The reactants were stirred for 24 h at room temperature. Afterwards, triclosan (10 wt% relative to TEOS) was added while stirring was continued for 30 min and the biocidal agent underwent dissolving in the TEOS solution. Then the mixture was transferred into trays and fabrics were impregnated in it. After a 15-min process of modification, the excess of the modifier solution was removed by squeezing followed by drying at 80 °C for 1 h and fixing at 130 °C for 3 min.

2.3. Synthesis of triethoxysilyl derivative of triclosan

The synthesis was carried out according to the reaction equation given below (Scheme 1).

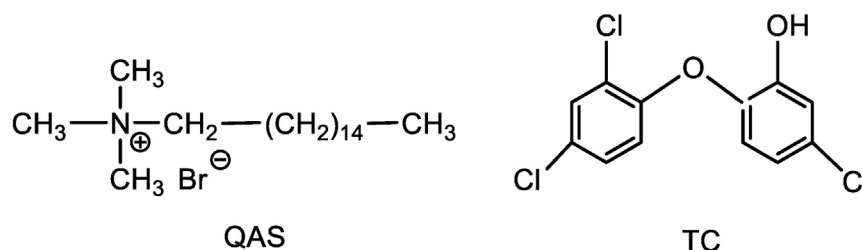


Fig. 1. Formulae of hexadecyltrimethylammonium bromide (QAS) and triclosan (TC) used as biocidal agents.

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