Barbara Filipowska, *Edward Rybicki, Anetta Walawska, *Edyta Matyjas-Zgondek

New Method for the Antibacterial and Antifungal Modification of Silver Finished Textiles

Textile Research Institute,
ul. Brzezińska 5/15, 92-103 Łódź, Poland
E-mail: filipowska@iw.lodz.pl

***Institute of Textile Architecture, Technical University of Lodz,**
ul. Żeromskiego 116, 90-924 Łódź, Poland

Abstract
In order to give antimicrobial properties to textiles, silver is more and more often used because of its wide spectrum of activity. Silver can be incorporated into textiles with the use of different methods: in the production process of chemical fibres or in the final product through its chemical modification. In this paper, a new method based on the precipitation of metallic silver on a textile surface as a result of the photochemical reaction of deposited compounds is shown. The results obtained for modified textiles using scanning electron microscopy (SEM), UV-Vis spectroscopy and Laser Induced Breakdown Spectroscopy (LIBS) confirmed the presence of metallic silver on the textile surface. The size of metallic silver particles precipitated on the product surface modified was determined using Dynamic Light Scattering (DLS). The results of microbiological tests (diffusion agar test) confirmed the effectiveness of the method elaborated with respect to microbiological resistance against a wide spectrum of bacteria and fungi. The simplicity of the method elaborated and the possibility of its application in textile plants equipped with a standard finishing device are emphasised.

Key words: *antibacterial finishing, antifungal finishing, silver, textiles, silver-finished textiles.*

Introduction

Due to their several precious properties such as breathability, wear comfort, hydrophilic properties and moisture absorption, cotton fibres are widely use in the production of underwear, protective clothing, medical garments and white goods. However, cotton fabrics provide an excellent environment for microorganism proliferation because of high moisture sorption and a large surface area. Microorganism growth on textiles can result in a loss of functional properties, an unpleasant odour, unsightly patches, and even posing a potential risk for users. Moreover, substances added to fibres, such as lubricants, antistatics, natural-based auxiliaries and dirt provide an excellent food source for microorganisms. Some researchers believe that in hospitals contaminated textiles might be an important source of microbes contributing to the transmission of nosocomial-related pathogens [1, 2]. There are many ways to control microbial growth on textiles, such as incorporating antibacterial agents into fibres during the spinning process by coating or padding. The most important antibacterial substances used in textile finishing are quaternary ammonium salts, chloro-ether phenols, poly(hexamethylene biguanidyne), silver and its compounds, organic-silicones, chitosan etc. [3 - 5]. Unfortunately, many of these agents have possible harmful or toxic effects [3, 6, 7]. Silver, on the other hand, is a relatively non-toxic disinfectant that can significantly reduce many strains of bacteria and fungi. The mechanism of the action of silver is linked with its binding to a cell wall and cell membrane, deactivating enzymes and the inhibition of the respiratory system [8 - 10]. Recently there has been a significant increase in using silver or silver compounds in medicine due to the appearance of numerous antibiotic resistant strains of bacteria [8]. Silver is usually applied to textiles in a colloidal state and in a dispersion of nano-metallic form or non-soluble silver salt [11 - 13]. In the case of synthetic fibres, silver can be introduced into a spinning solution in the form of a zeolite complex or nano-particles [14 - 16]. Silver zeolite is made by complexing alkaline metal with crystal aluminosilicate, which is partially replaced with silver ions using the ion exchange method [9, 17]. Nanoparticles are clusters of atoms in the size range 1 - 100 nm. Silver nanoparticles show good antibacterial properties due to a large surface area [9, 18]. Various methods of synthesising nano-silver particles are known, such as chemical reduction, photochemical reduction, biochemical reduction, and electrochemical reduction [19]. Among these methods, chemical reduction using sodium borohydrate, hydrazine hydrate or L-ascorbic acid as a reducing agent is the most common [19 - 22].

Nowadays, the incorporation of silver nanoparticles into textiles has received great interest due to their strong antibacterial effect. Chuh-Yean at al [23] developed a method of preparing cotton microfibers containing silver nanoparticles by ultraviolet irradiation. Wang at al. [24] reported a method of incorporating silver into an ultra-thin film by the electrochemical method. Ravindra at al. [25] developed a method of nano-silver particle synthesis using a "green process" and their incorporation into cotton fibres. El-Rafie at al. [26] prepared nano-silver particle colloids by the fungi process and applied them to cotton textiles. Potiyaraj at al. [27] also reported the in-situ synthesis of silver chloride nanoparticles on silk fabric.

The method of applying metallic silver to a textile product described in the study is a simple method allowing the uniform modification of a flat textile product and providing it with antimicrobial properties. The results of tests carried out previously indicate that even a small quantity of metallic silver applied to cotton fabric provides it with antibacterial and antifungal properties in the presence of different kinds of fungi [28 - 30].

The method presented is based on the photochemical transformation of precipitated silver chloride on a fabric surface into metallic silver under the influence of high-energy UV radiation. In order to introduce silver chloride into textile products, they are first padded with a silver nitrate solution, squeezed, dried and then padded with a sodium chloride solution, squeezed again and dried. Appropriately selected process parameters (padding solution concentration, squeezing degree) allow to apply different quantities of metallic silver to a textile product [31].

The silver particles deposited onto cotton fibres were characterised by different instrumental methods. The antibacterial and antifungal properties of silver particles deposited on cotton fabric were estimated using *Staphylococcus aureus* and *Escherichia coli* bacteria and *Candida albicans* and *Aspergillus niger* fungi.

Experimental part

Materials and equipment

Tests were carried out on cotton canvas, with a surface weight of 140 g/m^2, warp density: 250 threads/10 cm and weft density: 225 threads/10 cm. A part of the textile fabric was dyed black with *DYSTAR Procion H EXL* reactive dyestuffs (1.1% *Procion Brill. Scarlet H EXL*, 1.3% *Procion Yellow H EXL*, 4.5% *Procion Navy H EXL*, with a batch method (according to manufacturer's instructions) in an *Ugolini REDKOME* laboratory dyeing machine.

The samples were padded with solutions of chemical agents in a double-roller padding machine - *Fulard DV 450* equipped with two squeezing rollers with adjustable pressing. A specially constructed laboratory radiator - UV HF4 – CENARO [32], with a 2850 W lamp was used for sample irradiation. A device for cyclical reverse sample transport with adjustable speed allowed to select the irradiation time.

Photochemical modification

The process of textile fabric modification proceeded according to the reaction (1):

$$AgNO_3 + NaCl \rightarrow AgCl + NaNO_3 \quad (1)$$
$$\downarrow UV$$
$$Ag^o$$

The textile fabric samples were padded in baths containing silver nitrate (2 ÷ 20 g/dm^3), squeezed (squeezing degree 80%) and then dried at 60 °C. The amount of silver nitrate deposited on the surface was determined according to the equation:

$$p = T \times a / 1000 \quad (2)$$

where:
p - silver nitrate deposited on the fabric, in % to the weight of fabric,
a - concentration of silver nitrate in the padding solution in g/dm^3,
T - solution's pick-up in %.

Next the samples were padded in baths containing sodium chloride, squeezed (80%) and dried again. The concentration of sodium chloride in the padding bath was selected in excess of the amount resulting from stoichiometric calculations in order to ensure the total conversion of silver nitrate to silver chloride.

After silver chloride was precipitated as a result of the reaction of exchanging silver nitrate and sodium chloride ions, the samples were subjected to high-energy radiation for 24 seconds.

The amount of metallic silver incorporated into the textile fabric was calculated in relation to the amount of silver nitrate deposited, based on the stoichiometric equation, assuming 100% yield of the reaction.

Antimicrobial activity of the modified textiles

The effectiveness of the bio-stabilising finish was assessed by determining the antibacterial and antifungal resistance using a diffusion test on agar against the following microorganisms: *Staphylococcus aureus* ATTC 6538 and *Escherichia coli* ATCC 11229 according to PN-EN 20645:2007, and *Candida albicans* ATCC 10259 and *Aspergillus niger* ATCC 6275 according to SN 195921 :1994. The microbiological tests were carried out at the Microbiological Laboratory of the Biopolymer and Chemical Fibre Institute, Lodz, Poland. In ***Table 1***, the criteria of the antimicrobial efficacy according to Standard PN-EN ISO 20645 are specified.

Evaluation of the greying of the modified textile fabric

Colour measurement was made on a *Datacolor Int. Spectraflash 500* spectrophotometer with *dataMaster* software. A remission calculation was made at *D65* illuminant and for a 10^o observer (observer's field of vision involves an angle of 10º).

The colour difference - DE was calculated according to the formula of Commission Internationale de l'Eclairage - CIE Lab [33] in relation to an unmodified sample:

$$DE = \sqrt{(DL)^2 + (Da)^2 + (Db)^2} \quad (3)$$

where:
DE – colour difference expressed in CIELab units,
DL – difference in lightness,
Da – difference in the chromaticity coordinate, green/red axis,
Db – difference in the chromaticity coordinate, blue/yellow axis.

Table 1. *Criteria of the antimicrobial effect according to PN-EN ISO 20645.*

Growth inhibition zone, mm	Growth under sample	Description	Assessment
>1	none	inhibition zone exceeding 1 mm, no growth under sample	good effect
0-1	none	inhibition zone up to 1 mm, no growth under sample	
0	none	non-inhibition zone, no growth under sample	
0	slight	non-inhibition zone, growth under sample nearly totally suppressed	limit of efficacy
0	moderate	non-inhibition zone, growth under sample reduced to half	insufficient effect
0	heavy	non-inhibition zone, growth under sample non reduced or only slightly reduced	

*Table 2. Results of the antimicrobial properties of cotton textile fabric (plain weave, 140 g/m²) containing 1% (w/w) of silver; * - according to Polish Standard PN-EN ISO 105 – C06: 1999 (temp. 40 °C; ratio 1:10, 4 g/l of ECE detergent).*

Cotton fabric		Microorganism type							
		Staphylococcus aureus		*Escherichia coli*		*Candida albicans*		*Aspergillus niger*	
		growth inhibition zone, mm	growth under sample	growth inhibition zone, mm	growth under sample	growth inhibition zone, mm	growth under sample	growth inhibition zone, mm	growth under sample
non-dyed before modification		0	heavy	0	heavy	0	heavy	0	heavy
non-dyed after modification	non washed	1.2	none	0.5	none	0	none	0	none
	after 15 washes*	0	none	0	none	0	none	0	slight
after dyeing before modification		0	heavy	0	heavy	0	heavy	0	heavy
after dyeing and after modification		0.7	none	0.5	none	0	none	0	slight

Table 3. Colour difference between non-modified cotton fabric samples and those modified with silver (in CIELab units), according to CIE.

Cotton textile fabric type:	Colour difference DE	Difference in lightness DL	Difference in chromaticity coordinate green/red axis Da	Difference in chromaticity coordinate blue/yellow axis Db	Difference in chroma DC	Difference in hue Dh
non-dyed, without silver	0.0	0.0	0.0	0.0	0.0	0.0
non-dyed, containing 0.1% (*w/w*) of silver	26.236	- 21.997	8.404	11.568	13.825	-3.650
non-dyed, containing 0.2% (*w/w*) of silver	34.356	- 32.416	9.338	6.510	10.301	- 4.845
non-dyed, containing 0.5% (*w/w*) of silver	40.147	- 38.904	9.722	1.924	7.795	- 6.120
non-dyed, containing 1.0% (*w/w*) of silver	47.747	- 46.362	11.061	- 2.821	7.807	- 8.327
dyed, without silver (reactive black)	0.0	0.0	0.0	0.0	0.0	0.0
dyed (reactive black), containing 2.8%(*w/w*) of silver	6.064	5.264	- 0.532	- 2.962	2.967	- 0.509

Changes in the textile fabric surface as a result of modification

The microstructure of the textile fabric surface's appearance before and after modification was evaluated with a JEOL JSM 35 C scanning electron microscope in high vacuum conditions.

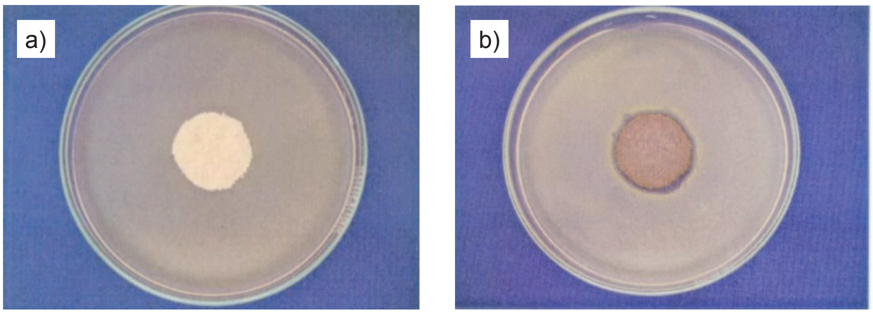

Figure 1. Change in microbiological resistance - after finishing consisting in precipitating metallic silver (1% w/w) – due to the action of Staphylococcus aureus: a) white cotton fabric before processing – lack of activity (insufficient effect); b) cotton fabric after processing – good activity (good effect).

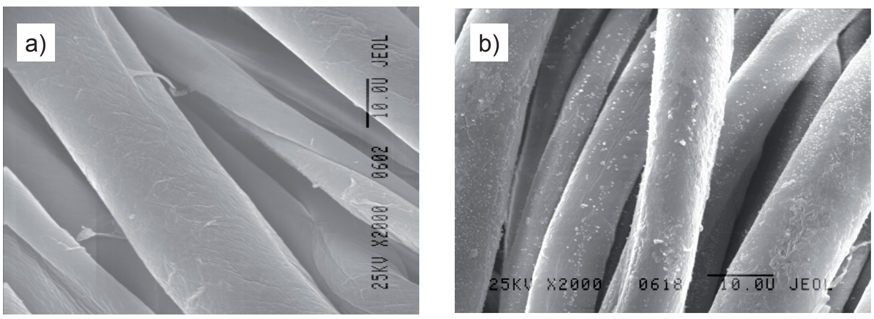

Figure 2. Cotton fabric surface modification after finishing consisting in metallic silver precipitation: a) cotton fabric before processing; b) cotton fabric after processing (1% w/w of silver).

Determination of the size of metallic silver particles precipitated on the modified surface of textiles

Dynamic Light Scattering (DLS), also known as Photon Correlation Spectroscopy (PCS), was used to determine the particle size distribution profile in dispersion. DLS is one of the most popular methods used for determining the size of particles. When a monochromatic light beam (such as a laser) passes through a dispersion, the particles scatter some of the light in all directions. The intensity of light scattered by a single particle depends on its molecular weight, overall size and shape. The Particle Sizing System NICOMP 380, a product of Nicomp, Santa Barbara, California, was used to obtain the particle size distribution of samples with particles ranging from 5 nm to 10 microns. Through the use of an appropriate Nicomp analysis algorithm, the 380 type is able to analyse complex multi-modal distributions with the highest resolution and reproducibility available [34].

Moreover the size and distribution profile of silver particle dispersion in the ferric sodium tartrate (EWNN) solution of silver finished cotton fibres were measured using the Particle Sizing NICOMP 380 System. For this purpose 0.1 g of the finished fabric was placed in 50 ml of the ferric sodium tartrate solution for ten hours with stirring to dissolve the cellulosic component.

Moreover, the size and distribution profile of silver particles extracted to water from the fabric were measured. The extraction was performed using a 50:1 liquor ratio, where 2.0 g of each finished fabric was shaken at 38 °C for two hours.

Test results and discussion

Both non-dyed and dyed and modified (by precipitating metallic silver on their surface) cotton textile fabrics show good antimicrobial properties against bacteria and fungi (***Table 2, Figure 1***)

Contrary to unmodified textile fabrics, in all cases no microoorganism growth was observed within the sample. The modified textile fabric shows a better antibacterial action than the antifungal one, confirmed by the existence of a small bacteria growth inhibition zone. After 15 washes the modified fabric practically retains its antibacterial properties, whereas the antifungal ones deteriorate to a slight extent in the case of *Aspergillus niger*. Textile fabric modified after dyeing demonstrates an antimicrobial action that is a bit weaker than the fabric modified without dyeing. Probably, in the case of preliminary dyed fabric, a part of silver ions from silver nitrate reacts with sulphonic groups of the dyestuff, which can result in a smaller amount of silver precipitated on the dyed fabric as compared to a non-dyed one.

The fabric dyeing had no influence on the variation in antimicrobial properties, both within the growth and inhibition zone of the microorganism growth. A lack of an inhibition zone or its small size (up to 1.2 mm) can be evidence of permanent silver particle fixing in the fabric structure.

Greying of the modified textile fabrics

Sample greying occurs as a result of modification – the more metallic silver that has been introduced into the fabric, the greater the greying is (***Table 3***). This ostensible defect becomes a merit in the case of a chosen assortment, where fabrics dyed with achromatic colours are applied, e.g. textile footwear elements. When the process of textile fabric modification is performed with the silver precipitation method, the dyeing process can be limited or even eliminated.

The scanning electron microscope photographs (***Figure 2***) indicate the appearance of small particles, most probably silver particles, on the fabric surface, which is confirmed by some previously published results of LIBS [35].

In the LIBS spectrum of the modified fabric, some lines corresponding to the presence of silver appeared (***Figure 3***), and the greater the quantity of silver deposited on the surface, the more intense they become.

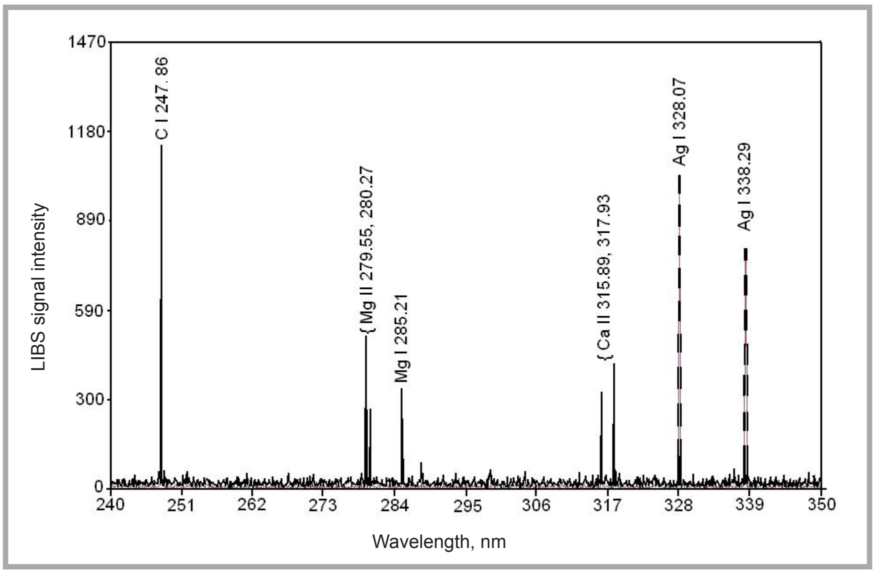

Figure 3. *Spectra of cotton fabric with silver (1% w/w), obtained with LIBS. Silver lines 328 and 338 are present only in the case of samples containing silver [35].*

Table 4. *Distribution of silver particles obtained by the DLS method in EWNN solution and extracted to water from the fabric.*

Ag particles	Intensity weight		Volume weight		Number weight	
	Mean diameter, nm	Percent, %	Mean diameter, nm	Percent, %	Mean diameter, nm	Percent, %
EWNN solution	82.0	9.9	78.8	33.3	77.7	83.3
	824.6	90.1	836.6	66.8	824.6	16.7
Extracted from fibres	42.5	6.9	38.5	82.6	37.4	95.4
	166.9	93.1	177.5	17.4	168.0	4.8

Size of silver particles

The distribution of the silver particle size for all the silver finished fabric is shown in ***Table 4***. The size of silver particles synthesised directly on and into the fibres as obtained by the DLS technique was in the range of 77.7 nm to 824.6 nm (number-weight). The particles extracted to water from the silver finished fabrics are different in size and distribution profile to those observed in the EWNN solution of cotton fabrics (***Table 4***).

Conclusions

The method of modification presented is an effective way of incorporating metallic silver into cotton textiles. Precipitated silver particles are deposited not only on the surface but also in the cotton fibre structure. The mean diameter of the precipitated silver particles varied from about 40 nm to about 800 nm.

The presence of silver renders antibacterial and antifungal properties to textile fabrics. At the same time, the products modified demonstrate slightly diversified activity against different kinds of microorganisms. In the case of fungi, the action of silver is expressed by the lack of growth of *Candida albicans* and *Aspergillus niger* under the sample, while in the case of bacteria *Staphylococcus aureus* and *Escherichia coli* an additional growth inhibition zone is observed.

The preliminary dyeing process of textile fabric before silver deposition can cause a slight reduction in its antimicrobial activity.

Under the influence of modification, the colour of textile fabric is changed towards grey, characteristic for silver colour. In the case of assortments, where fabrics dyed with achromatic colours are applied, e.g. textile footwear elements, this fact enables the limitation or elimination of the dyeing process.

Due to the wide spectrum of the operation of silver, the method can be used for modifying different textile products: cotton and polyester-cotton blends (at present under investigation), where antibacterial and antifungal protection is required: dressing products, textile footwear elements, underwear, protective clothing, among others.

The process can be continuous and be applied in textile industry plants using existing machinery.

The method of textile fabric modification described provides good antibacterial and antifungal properties, as well as resistance to washing, at a concentration of silver of about 1% (w/w). Currently, tests are being performed to determine the amount of silver necessary to obtain antimicrobial properties. The results will be presented in the next publication. In turn, after the determination of optimal parameters for textile finishing by means of silver, a comparison of the economics of this method with the commercial one will be possible.

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