



Antimicrobial chitosan finish of cotton and silk fabrics by UV-curing with 2-hydroxy-2-methylphenylpropane-1-one

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ABSTRACT

The purpose of this work was to develop a textile finish based on radical UV-curing of chitosan on cotton and silk to confer antimicrobial properties. Fabrics were impregnated with a solution of 2% w chitosan in aqueous acetic acid (2% v/v) added of 2-hydroxy-2-methylphenylpropane-1-one (2% w/w with respect to chitosan) as photoinitiator and cured at room temperature by exposure to UV lamp for 60 s on both the sides. The antimicrobial activity of finished fabrics was tested according to ASTM standard test performed with *Escherichia coli*. Obtained results showed a strong antimicrobial activity conferred by the treatment, homogeneous on fabric surface, without affecting the hand properties of fabrics due to the low chitosan weight on (about 2%). The treatment durability to domestic laundering was tested after 5 cycles using either anionic or nonionic detergents. The antimicrobial activity resulted completely maintained after washing with a nonionic surfactant, while with anionic detergents the treatment durability was better for samples prepared with a deeper penetration of chitosan inside the fibers. The fabrics were characterized by dyeing tests, SEM and FTIR-ATR analyzes.

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1. Introduction

Fabrics and protective clothes used in hospitals, nursing homes, schools, hotels and crowded public areas or textiles left wet between processing steps for long time can benefit from antimicrobial finishing. Finally the use of antimicrobial finishes to prevent unpleasant odors in intimate apparel, underwear, socks and athletic wear is an important market need.

For these purposes cotton is the most widely used natural fiber (Hebeish et al., 2011), while silk from *Bombyx Mori*, in particular, is not only one of the most luxury fibers used in textile industry but also a biopolymer of great interest, thank to its biocompatibility, if applied in biomedical and biotechnological devices (Leal-Egaña & Scheibel, 2010). In this field, the antimicrobial activity has great importance (Arai et al., 2001).

Today, 2,4,4'-trichloro-2'-hydroxydiphenyl ether, known as Triclosan, quaternary ammonium salts and silver ions are commonly used as antimicrobials. Indeed, popular finishes, already commercialised, bondable at fiber surfaces are based on 3-(trimethoxysilyl) propyl dimethyl octadecyl ammonium chloride or polyhexamethylene biguanide (Alonso et al., 2009; Schindler & Hauser, 2004, chap. 15).

Chitosan, 2-amino-2-deoxy-(1→4)-β-D-glucopyranan, is a biopolymer with unique properties such as biodegradability, non-toxicity, antimicrobial activity. It is widely available as a byproduct of the food industry (Goy, de Britto, & Assis, 2009). Chitosan is already used in textile field for dyeing and finishing (Lim & Hudson, 2004), but the major problems of chitosan as antimicrobial agent are the activity loss under alkaline conditions due to modification of the cationic nature (Sudardshan, Hoover, & Knorr, 1992; Tsai & Su, 1999) and the poor durability on textiles due to lack of strong bonding with fabrics (Shin & Min, 1996). Hence it is applied by wet thermal curing involving relatively high temperature with energy consumption, costs and possible fabric degradation. Moreover the addition of crosslinking agents is required (Alonso et al., 2009; El-Tahlawy, El-Bendary, Elhendawy, & Hudson, 2005). Most of them are toxic reagents, such as glutaraldehyde. Recently genipin was proposed as safer crosslinking agent of chitosan instead of glutaraldehyde for biomedical and pharmaceutical purposes (Muzzarelli, 2009a), but its high cost does not yet allow for the use in textile finishing.

Otherwise chitosan can be made photocrosslinkable by introduction of azide moiety (Muzzarelli, 2009b) while there are no specific studies about photocrosslinking of unmodified chitosan alone. However the effect of exposure to UV light of chitosan films or blends, forming macroradicals, has been widely studied in the field of UV degradation of biopolymers (Saiki et al., 2010; Sionkowska, Wisniewski, Skopinska, Vicini, & Marsano, 2005; Sionkowska et al., 2006). The same radicals can be involved in a

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photopolymerization process of chitosan with acrylates (Alam, Khan, Khan, Ghoshal, & Mondal, 2008; Khan, Ferdous, & Mustafa, 2005).

The advantages of UV-curing technology in the field of coatings, printing, adhesives and electrical insulation are well known: energy savings (low-temperature process), low environmental impact, simple, cheap, and small equipment, high treatment speed. Despite these advantages, until now there have been few applications of UV-curing in the textile industry (Krijnen, Marsman, & Holweg, 1994).

If a monomer and initiator mixture is adsorbed onto the fibers and subsequently UV-cured, the polymeric chains can form inside the textile structure, which can also establish graft bonds, making the treatment solid and water-resistant (Princi et al., 2006). Moreover, the interpenetration of components and homogeneous distribution of monomers, even at a low concentration, contribute to obtain textile materials with modified surface properties without high add-on of polymer (Ferrero, Periolatto, Sangermano, & Bianchetto Songia, 2008). Consequently the fabrics may retain their original properties of hand and breathability.

In our previous study chitosan was applied on cotton and synthetic fabrics by radical UV-curing and high values of antimicrobial activity were found (Ferrero & Periolatto, 2011). Moreover the importance of impregnation time and dilution on the process yield was evidenced. The high viscosity of the chitosan solution hinders its removal by solvent evaporation during the drying phase, but the substrate impregnation is made difficult. Hence a dilution is important to lower the solution viscosity and to improve the spreading homogeneity: fabrics prepared with acetic acid dilution of chitosan showed more homogeneous properties on the surface than undiluted formulations, while water dilution caused lower yields. In the case of cotton and synthetic fabrics an impregnation time of 12 h at 25 °C was found necessary to obtain high yields.

In the present work the same antimicrobial finish was applied also to silk fabrics and, in order to make the process more suitable for industrial applications, tests on silk were carried out on samples prepared with shorter impregnation time either at ambient temperature or at 50 °C. Eventually ultrasounds were also experimented.

Moreover the treatment durability after repeated washings on cotton as well as on silk was examined in depth with anionic and nonionic detergents.

2. Experimental

2.1. Materials

Fabrics treated were plain-weave pure cotton (144 g/m²) previously washed but not subjected to any finishing process and pure silk stockings knitted with degummed and purged *Bombyx mori* silk yarn, 20–22 decitex, four-ply.

Low viscosity chitosan, 75–85% deacetylation degree (Fluka) was used. This was dissolved in aqueous solution of 2% v/v glacial acetic acid under magnetic stirring at ambient temperature for 24 h. Mostly a chitosan concentration of 2% w was used.

2-Hydroxy-2-methylphenylpropane-1-one (Darocur 1173, Ciba Specialty Chemicals) was used as photoinitiator.

2.2. UV-curing

The photoinitiator was added to chitosan solution in the amount (2% w/w with respect to chitosan) that was determined suitable to produce a solid and dry chitosan film by UV irradiation.

Further steps were dilution, with water or 2% v/v acetic acid solution, spreading of the mixture on the fabrics with various

impregnation times till to 12 h and at different temperatures, drying for 10 min at 80–100 °C and finally UV-curing.

The surface coated fabrics were exposed to UV radiation using a medium pressure mercury lamp with a light intensity on the fabric of about 20 mW/cm², in a small box equipped with a quartz window under nitrogen atmosphere (oxygen content under 20 ppm). The required radiation dose was obtained adjusting the distance of textiles from the lamp at about 20 cm and the exposition time at 60 s. To assure the complete curing on the fabrics, they were radiated on both the sides.

On silk, sonication during impregnation was also carried out in order to improve the diffusion of chitosan inside the fiber structure. The fabric wetted with chitosan solution was put in a sealed tube and sonicated with Elmasonic S60H (ELMA GmbH & Co., Singen, Germany). This sonicated and thermo-controlled bath can generate ultrasounds at 37 kHz, with effective ultrasound power of 150 W and heating power of 400 W.

2.3. Process yield

The weight gain of fabrics, that is the add-on of polymer, was calculated as:

$$\text{Weight gain (\%)} = \frac{w - w_0}{w_0} \times 100 \quad (1)$$

where w is the weight of grafted fabric and w_0 the weight of the original fabric.

All the treated samples were prepared with a chitosan add-on of about 2% w: in this way fabrics maintained their hand characteristics.

The yield of the process was evaluated calculating the difference, in percentage, between the amount of chitosan present in the solution used for the fabric impregnation and the chitosan grafted to the fibers after UV-curing. Chitosan grafted to fabric was calculated as difference between the fabric weight before and after the treatment.

2.4. Antimicrobial tests and durability to laundering

The antimicrobial activity was evaluated on chitosan treated samples, about 1 g, according to ASTM E 2149-01 “Standard test method for determining the antimicrobial activity of immobilized antimicrobial agents under dynamic contact conditions”. This method is designed to evaluate the resistance of non-leaching antimicrobial treated specimens to the growth of microbes under dynamic contact conditions. The bacterium was *Escherichia coli* ATCC 11229. The incubated test culture in a nutrient broth was diluted to give a concentration of $1.5\text{--}3.0 \times 10^5$ CFU/ml (working dilution). Each fabric was transferred to flask containing 50 ml of the working dilution. All flasks were shaken for 1 h at 190 rpm. After a series of dilutions, 1 ml of the solution was plated in nutrient agar. The inoculated plates were incubated at 37 °C for 24 h and surviving cells were counted. The antimicrobial activity was expressed in % reduction of the organisms after contact with the test specimen compared to the number of bacterial cells surviving after contact with the control.

On samples finished with chitosan, treatment durability to domestic laundering after 5 cycles was tested according to UNI-EN ISO 105-C01 using either standard ECE detergent, distributed by EMPA Testmaterials (CH), mainly anionic, or nonionic Tween 20 surfactant by Sigma-Aldrich.

2.5. Characterization of the treated fabrics

Chitosan confers to fabrics dyeability towards acid dyes due to the interaction between protonated amino groups and sulfonate

Table 1

Yield, antimicrobial activity before and after washing with two detergents evaluated on chitosan treated samples (formulation diluted with 2% acetic acid).

Fabric	Impregnation time and temperature	Weight on (%)	Yield (%)	Microorganism reduction (%) before washing	Microorganism reduction (%) after washing	
					With ECE	With Tween 20
Cotton	5 min, 25 °C	2.0	75	99.6	38.9	94.4
Cotton	12 h, 25 °C	2.0	–	98.9	52.6	94.7
Cotton	12 h, 25 °C	3.0	–	97.2	97.2	–
Silk	1 h, 25 °C	2.2	99	100	63.0	99.5
Silk	3 h, 25 °C	2.2	100	100	65.7	–
Silk	6 h, 25 °C	2.9	100	100	51.1	–
Silk	1 h, 50 °C	2.1	99	100	73.0	100
Silk	2 h, 50 °C	2.2	99	93.7	55.2	–
Silk	3 h, 50 °C	1.8	87	95.1	60.8	–
Silk	1 h, 25 °C sonicated	2.7	100	100	47.6	–

groups of the dye ions. So the chitosan presence on finished fabrics and the treatment homogeneity were tested by dyeing with Telon Turquoise M5–G85%, C.I. Acid Blue 185 (DyStar). The dyeing process was carried out on both untreated and treated fabrics prepared with the different impregnation methods and even after 5 domestic washing cycles, to prove if a release of chitosan from the fabrics was evidenced.

Color measurements were performed on dyed samples according to ISO 7724-2:1984, measuring the spectral reflectance with a spectrophotometer (Spectraflash SF600XV) from Datacolor (Switzerland), under CIE standard illuminant D₆₅ and 10° observer.

The surface morphology of the fabrics was examined by SEM with a Leica (Cambridge, UK) Electron Optics 435 VP scanning electron microscope with an acceleration voltage of 15 kV, a current probe of 400 pA, and a working distance of 20 mm. The samples were mounted on aluminum specimen stubs with double-sided adhesive tape and sputter-coated with gold in rarefied argon using an Emitech K550 Sputter Coater with a current of 20 mA for 180 s.

FTIR analyzes were performed on a Nicolet FTIR 5700 spectrophotometer, equipped with a Smart Orbit ATR single bounce accessory mounting a diamond crystal. Each spectrum was collected directly on single yarn by cumulating 128 scans, at 4 cm^{−1} resolution and gain 8, in the wavelength range 4000–600 cm^{−1}.

3. Results and discussion

3.1. Process yield

The results of process yield on silk fabrics reported in Table 1 are very high, reaching values of 100% or very close with every impregnation procedure. It could be due to the greater affinity of chitosan to fibroin. In fact it was reported in literature that UV irradiation of chitosan can yield many type of macroradicals (Saiki et al., 2010) which are responsible of UV crosslinking of chitosan in the presence of a photoinitiator or grafting with acrylates. However silk in comparison with cellulose under UV irradiation is more able to generate macroradicals due to the presence of the photosensitive aromatic amino acids tyrosine, tryptophan, and phenylalanine. Hence besides UV crosslinking of chitosan with itself onto the fiber, strong graft bonds between chitosan and silk can be generated.

3.2. Antimicrobial activity and durability to laundering

The results of the determination of antimicrobial activity on chitosan UV-cured fabrics are also reported in Table 1. Total microorganisms reduction or close values were found on all treated fabrics before washing, regardless of adopted process conditions. These results are of particular interest because they have to be compared with 0% organism reduction measured on the untreated samples.

On cotton a slightly microorganism reduction decrease with impregnation time or weight on increase can be justified by a probably decrease of the content of free amino groups of chitosan due to a more extended crosslinkage and grafting. Moreover the same considerations can account for antimicrobial activity reduction on silk at 50 °C at impregnation time longer than 1 h.

In the same Table, the results of antimicrobial activity evaluated on chitosan treated samples after 5 washing cycles are compared. The samples subjected to washing with ECE detergent yielded a reduction of antimicrobial activity, but some differences arise in dependence on the fiber nature and impregnation method.

On cotton with 2% chitosan weight on, 12 h of impregnation at ambient temperature was necessary in order that treated fabrics maintained about one half of its own antimicrobial activity after washing with standard ECE detergent, while at shorter impregnation time the antimicrobial activity after washing was more reduced. It suggested that the antimicrobial activity loss was due to inadequate penetration of chitosan inside fiber structure, caused even by the high viscosity of the solution. A prolonged contact time between chitosan solution and fabrics improved this penetration and chitosan could graft to the fibers even inside their structure in a strongly bounded way and in higher concentration. This last aspect was confirmed by the good result shown by the cotton fabric with 3% chitosan weight on.

In general on silk with 1 h and 3 h impregnation time, durability of the antimicrobial activity after washing with ECE detergent was better than on cotton confirming a stronger affinity between silk and chitosan. Among all, the sample impregnated for 1 h at 50 °C gave best durability results, with a microorganism reduction of 73% after washing, a good value confirming the positive effect due to temperature increase. On the opposite, sonication during the impregnation was harmful on durability after washing.

However the substitution of ECE detergent with the nonionic Tween 20 in some washing tests showed that the antimicrobial activity was only slightly reduced on cotton and completely maintained on silk even at the lower impregnation time at 50 °C as well as at ambient temperature.

The different behavior of the two types of detergents towards the treated fabrics can be explained if it is taken into account that chitosan not strongly bonded to the fibers can be removed by washing with anionic surfactants like LAS (Lim & Hudson, 2004), whereas a nonionic surfactant is unable to establish strong bonds with chitosan which practically cannot be removed by repeated washings.

3.3. Characterization of the treated fabrics

For what concerns dyeing tests on chitosan treated samples, the process efficiency was soon revealed comparing untreated and treated fabrics. The former assumed in fact just a pale cyan coloration while a darker coloration was well evident on all chitosan

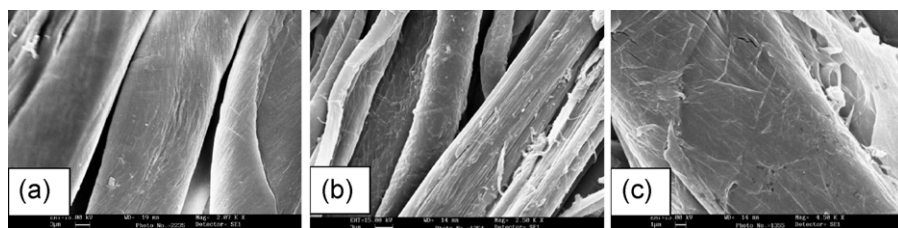


Fig. 1. SEM analysis on: (a) untreated cotton (magnification ratio 2500 \times), (b) cotton treated with 3% chitosan weight-on (magnification ratio 2500 \times) and (c) cotton treated with 3% chitosan weight-on (magnification ratio 4500 \times).

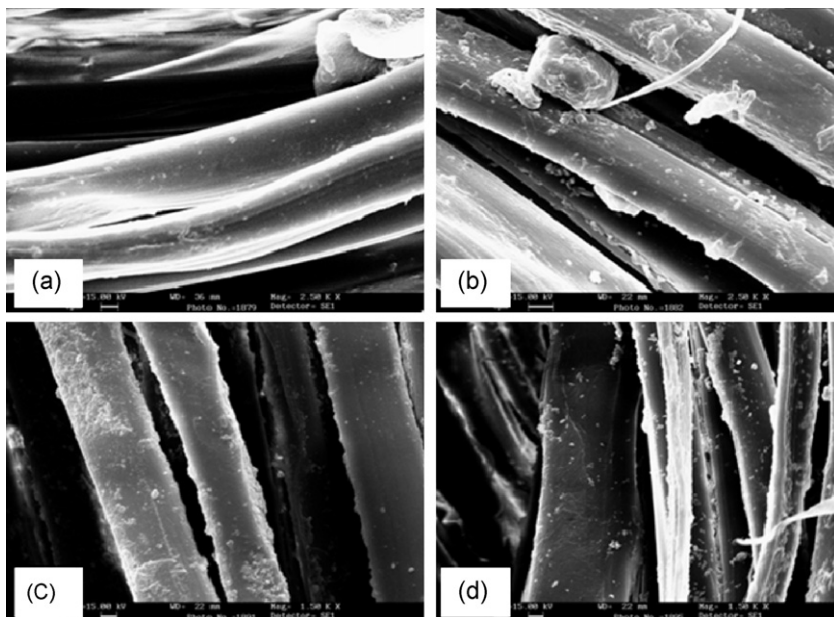


Fig. 2. SEM analysis on: (a) untreated silk, (b) 2% chitosan treated silk, 1 h impregnation at 25 $^{\circ}$ C, (c) 2% chitosan treated silk, 1 h impregnation at 50 $^{\circ}$ C and (d) 2% chitosan treated silk, 1 h impregnation at 25 $^{\circ}$ C with sonication. Magnification ratio 1500 \times .

treated samples. Moreover the uniformity of coloration showed the homogeneity of the treatment.

A less intense coloration of treated fabrics after washing tests with ECE detergent indicated a partial release of chitosan, responsible of the lower antibacterial activity. This effect is in fact more pronounced in samples showing lower antimicrobial activity after washing, with even a change in tone. Moreover silk samples impregnated with sonication revealed an uneven coloration indicating a fiber degradation (Liu, Du, & Kennedy, 2006).

In Table 2 the reflectance percentage corresponding to the peak at 490 nm wavelength related to the dyed silk samples are reported, confirming the evaluation at eyesight: the higher the reflectance value, the brighter the sample.

In Fig. 1a–c the image of untreated cotton fabric obtained by SEM analysis is compared with those of a chitosan treated sample at two magnification ratios. It is shown that the chitosan does not form a

coating on the textile but it covers every single fiber without gluing them. In Fig. 2a–d, images related to SEM analysis on silk fabrics obtained with different impregnation methods are reported. The best distribution on the fibers is observed on the sample prepared with 1 h impregnation time at 50 $^{\circ}$ C. The sample impregnated at ambient temperature presents an uneven distribution of chitosan on the fiber surface, with agglomerates not adherent to silk, easily taken away by washing. Finally, on sonicated sample, damaged silk fibers were detected.

The comparison of FTIR-ATR spectra of untreated and 3% chitosan treated cotton shows the evidence in the spectrum of the treated fabric of typical peaks of chitosan at 3290 cm^{-1} and 2900 cm^{-1} due to NH and CH stretching respectively. Other typical peaks at 3360 cm^{-1} (OH groups), 1648 cm^{-1} (C=O), 1560 and 1075 cm^{-1} (NH bending in amide group) are overlapped by those of cellulose. However on silk fabrics this analysis resulted ineffective due to the presence, on silk, of the same amine and amide groups also present on chitosan.

4. Conclusions

In conclusion we can say that chitosan UV-curing yielded strong antimicrobial properties against *E. coli* even on silk fabrics at low polymer add-on, about 2%, likewise it was found in the previous work on cotton (Ferrero & Periolatto, 2011). In order to have a good treatment fastness, chitosan diluted with aqueous acetic acid solution was spread on fabrics and an impregnation time of 3 h at ambient temperature or 1 h at 50 $^{\circ}$ C was necessary before the

Table 2

Dyeing test on treated samples. Reflectance evaluated on chitosan treated silk fabrics before and after 5 washing cycles with ECE detergent.

Impregnation time and temperature	Weight on (%)	Reflectance (%)	
		Before washing	After washing
Untreated silk	–	45	–
1 h, 25 $^{\circ}$ C	2.2	41	49
1 h, 50 $^{\circ}$ C	2.1	41	52
1 h 25 $^{\circ}$ C sonicated	2.7	42	46

curing to ensure a good penetration inside silk fibers, while in the previous work longer impregnation times at ambient temperature were needed for cotton.

However another novelty aspect was related to the durability after washing of the antimicrobial treatment which resulted strongly dependent on the fiber nature and the ionic character of the detergents. In fact and a nonionic surfactant could assure an antimicrobial activity completely retained after repeated washings on silk as well as on cotton.

The homogeneous distribution of chitosan on silk fabrics, in particular in correspondence to the sample that presented the best durability, was confirmed by dyeing tests with an acid dye. SEM analysis showed that the optimal distribution of the finish on single fiber surface was achieved with an impregnation time of 1 h at 50 °C, while sonication damaged the silk fibers.

In conclusion, chitosan UV curing can be indicated as a valid environmental friendly method to confer a satisfactory washing resistant antimicrobial activity against *E. coli* to cotton and silk fabrics.

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