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Evaluation of multifunctional properties of cotton fabric based on metal/chitosan film

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ABSTRACT

Films of chitosan with both of zirconium and/or titanium at different ratios as model film applied to cotton fabric have been evaluated. The structure of cotton fabric and its morphology were studied using both X-ray diffraction and scanning electron microscope (SEM-EDX), respectively. Chitosan–titanium film showed the highest interaction with cotton fabric that lead to lower crystallinity index of fabric. Whereas, week interaction between cotton fabrics is observed in case of zirconium/titanium–chitosan film. From SEM-EDX, chitosan–titanium film sorption on cotton is the highest where the N content is 9.67%. Moreover, the UV-protecting properties as well as the antibacterial activity of cotton fabric against *Staphylococcus aureus* (*S. aureus*) and *Escherichia coli* (*E. coli*) are investigated. The effect of metallic salt treatments on the UV-protection factor (UPF), UV-protection grade as well as on the percent reduction in bacterial count (% RBC) has been studied. Results showed that UPF rating of prepared samples incorporated with Ti was >50, with Zr 45–50 as well as with the mixed were 45–50 at all used concentration. The extent of improvement in the UPF values is based on the nature of the deposited metal oxide. Ti > Zr > mixed > - none. Furthermore the extent of increasing of oil stain release (OSR) values is based on the type of metal oxides (i.e. 4–5 for Ti, 2–4 for Zr as well as 3–5 for the mixed oxides). On the other hand the antibacterial activities of the prepared cotton fabrics containing the metal oxides using reduction of bacterial count method against G +ve bacteria (96–87%) and G −ve bacteria (93–70.5%) are depending on the nature of the deposited metal oxide, i.e. Zr > mixed > Ti > coated fabric > none. UV-cutting as well as incorporation of metal oxides, enhancing both an excellent UV-protection and prominent antibacterial activities.

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1. Introduction

Chitosan, is a copolymer of *b*[1,4]-linked 2-acetamido-2-deoxy-D-glucopyranose and 2-amino-2-deoxy-D-glucopyranose, is generally obtained by deacetylation of chitin. This polymer possesses hydrogel-like properties through a reaction with glutaraldehyde as a crosslinking agent. In the form of a hydrogel, chitosan is used in a wide range of applications such as wastewater treatment (Crini, 2005), separation membrane (Won, Feng, & Lawless, 2002), food packaging (Arvanitoyannis, 1999), wound healing (Arvanitoyannis, Kulokuris, Nakayama, Yamamoto, & Aiba 1997) and a drug delivery system (Arvanitoyannis, Nakayama, & Aiba 1998; Nunthanid et al., 2004; Puttipipatkhachorn, Nunthanid, Yamamoto, & Peck, 2001). The use of chitosan in the application of drug delivery systems has received special interest. As a biocompatible, non-toxic, and hydrogel-like material, chitosan is a medically suitable candidate as a drug carrier for various drug types. It has been shown (Nunthanid et al., 2004; Puttipipatkhachorn et al., 2001) that the drug release behavior of chitosan is governed mainly by the swelling property, the dissolution characteristic of the polymer films, the pK_a of the drug, and the drug–polymer interaction.

Healing of dermal wounds with macromolecular agents such as natural polymers is preferred to skin substitutes owing to many advantages such as biocompatibility, nonirritant and non-toxic properties, and ease and safety of the application on dermis (Sezer et al., 2007). Biopolymer chitosan has been used as a wound dressing in burn healing for proliferation and activation of inflammatory cells in granulation tissue and consequently for accelerating wound cleaning and re-epithelization properties.

There is considerable interest in organic–inorganic hybrid materials. A variety of organic polymers have been introduced into inorganic networks to afford the hybrid materials with or without covalent bonds between the polymer and inorganic components, respectively (Brinker & Scherer, 1989; Klein, 1988).

The high content of nitrogen atoms in the chitosan allows uptake of several metal ions through various mechanisms such as chelation, electrostatic attraction or ion-exchange, depend on the metal ion and the pH of the solution (Guibal, 2004). The amine

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and two hydroxyl groups on each glucosamine in the repeating unit of chitosan can act as a reactive site for chemical modification. The acido-basic properties of chitosan lead to a cationic behavior in acidic conditions (pK_a near 6.2). The cationic properties of chitosan make the biopolymer efficient at sorbing anionic compounds, including metal anions or anionic dyes through electrostatic.

Furthermore the UV-radiation is one of the major causes of degradation of textile materials due to photo oxidation. On the other hand UVβ-radiation, 280–315 nm, can penetrate into the top layer of the skin causing a range of effects from simple tanning to highly malignant skin cancers, if unprotected. Ultraviolet protection factor (UPF) is determined by the nature of the textile fibres, their chemical structure, fabric construction, presence of UV-absorbers, and coloring and/or finishing agents. The use of UV-protecting fabrics, avoiding sunlight at its maximum as well as reducing the UV-radiation exposure can provide excellent protection against the harmful effects of sunlight (Ibrahim, Allam, El-Hossamy, & El-Zairy, 2007; Saravanan, 2007).

In this study, we will prepare the chitosan film containing metal oxides and applied to cotton fabric by using coating technique for enhancing multifunctional properties such as antimicrobial, UV-protection. Furthermore, the characterizations of the film will also discuses by means scanning electron microscope (ESM-EDX) and X-ray diffraction.

2. Experimental methods

2.1. Materials

Scoured and bleached cotton fabric (125 g/m^2), high molecular weight chitosan with degree of acetylation 85% (Aldrich Co.), acetic acid, titanium dioxide, and zirconium oxide.

2.2. Methods

2.2.1. Preparation of chitosan film containing metal oxides

Chitosan paste was prepared by dissolving chitosan 4% (w/v) in acetic acid (2% v/v), the mixture was strongly stirred by using homogenizer until the paste is formed. Titanium dioxide and zirconium oxide were added to the prepared paste with different ratio (e.g. 1 g titanium dioxide, 1 g zirconium oxide, 0.5 g titanium dioxide + 0.5 g zirconium oxide, 1 g titanium oxide + 0.5 g zirconium oxide as well as 0.5 g of titanium oxide + 1 g zirconium oxide) and the pastes containing metal oxides were stirred strongly by using the homogenizer for 30 min.

2.2.2. Coating of cotton fabric with chitosan film containing metal oxides

Chitosan film containing metal oxides with different ratio were applied to cotton fabric samples by using a coating technique. Coated cotton fabric samples were dried in an oven at 70 °C for 10 min. Dried fabric samples were cured in an oven at 170 °C for 5 min. Cured fabric samples were washed with alkaline water for 30 min at 90 °C and then washed with tap water and finally dried.

2.3. Analysis

2.3.1. X-ray diffraction studies

The X-ray diffraction patterns of chitosan titanium, zirconium and its blend films coated cotton fabrics with different ratios were determined on a Shimadzu Lab-XRD-6000X diffractometer, using Nickel-filtered Cu Kα radiation at 40 kV and 50 mA in the 2θ range of 10–70°. The effect of metals and its blend on the degree of crystallization was determined. Crystallinity was calculated from the diffracted intensity data using the method of Segal, Nelson, and Conrad (1953).

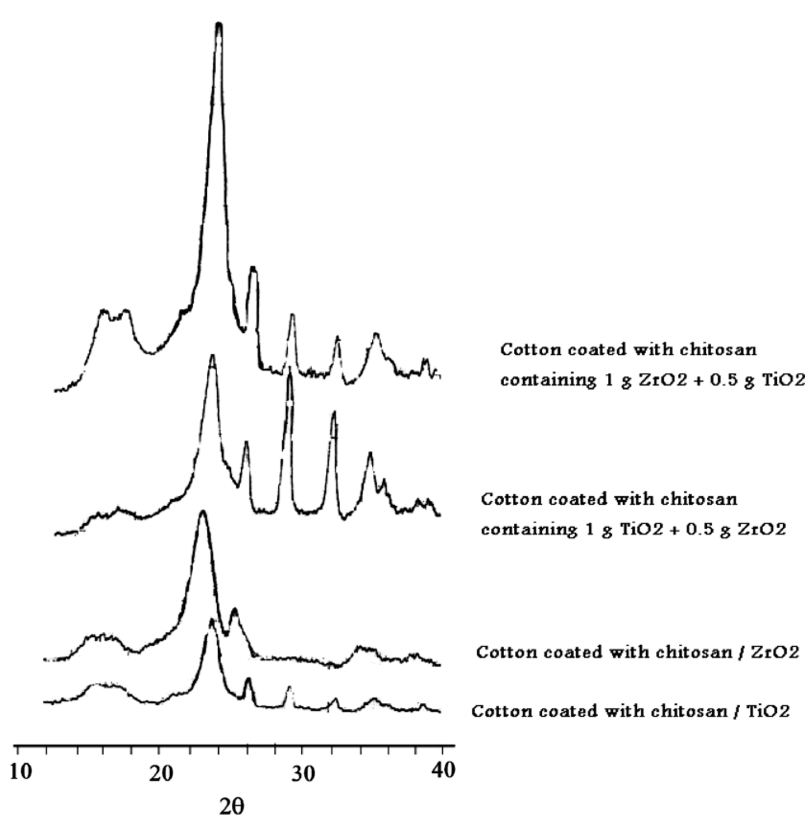


Fig. 1. X-ray diffraction patterns of cotton fabrics coated with chitosan films containing titanium, zirconium as well as its blends with different ratio.

Table 1
Effect of metal oxide type and concentration on oil stain release (OSR) and UV-protective factor properties of prepared fabrics.

Metal type and concentration	Metal concentration	OSR value	UPF rating
Blank	0	1.0	6.0
	0	1.0	6.0
	0	1.0	6.0
Chitosan film	0	1–2	17.6
	0	1–2	17.6
	0	1–2	17.6
Film + Ti-oxide	0.5 g%	3–4	>50.0
	1.0 g%	4–5	>50.0
	1.5 g%	4–5	>50.0
Film + Zr-oxide	0.5 g%	2–3	45
	1.0 g%	3–4	50
	1.5 g%	4	50
Film + (Ti-oxide + Zr-oxide)	0.5 g%	4–5	50
	1.0 g%	3–4	>50
	1.5 g%	4	>50
Film + (Zr-oxide + Ti-oxide)	0.5 g%	4–5	50
	1.0 g%	3–4	>50
	1.5 g%	4	>50

2.3.2. Morphology observations

The cross-sectional morphologies of blend films were examined using scanning electron microscopy (SEM-EDX) JEOL-JXA-840 an Electron Probe Microanalyzer. Cross-sectional samples were prepared by fracturing films in liquid nitrogen. Prior to observation samples were arranged on metal grids, using double-sided adhesive tap, and coated with gold under vacuum before observation.

2.3.3. The oily stain release rating (OSR)

OSR was assessed according to AATCC Test Method 130-1974.

2.3.4. UV-protection factor (UPF)

UPF values were assessed according to the Australian/Newzeland Standard (AS/NZS 4399-1996). According to the Australian classification scheme, fabrics can be rated as providing good, very good, and excellent protection if their UPF values range from 15 to 24, 25–39 and above 40, respectively. In no event was a fabric assigned a UPF rating greater than 50 (Srinivasan & Gatewood, 2000).

2.3.5. Antibacterial activity

Antibacterial activity against Gram-positive bacteria (*Staphylococcus aureus*) and Gram-negative bacteria (*Escherichia coli*) was tested quantitatively by AATCC Test Method 100-1999.

2.3.6. Durability

The durability to wash was determined according to AATCC Method 124. The obtained results of the aforementioned analysis and test methods are the average of triplicate tests.

3. Results and discussion

3.1. Structure and morphology characterization

3.1.1. X-ray diffraction studies

Fig. 1 shows the diffraction patterns of cotton fabrics coated with chitosan films containing titanium, zirconium as well as its blends with different ratio.

The data show that, the diffractive angle ($2\theta°$) and *d*-spacing of the corresponding plane are presented in Table 1. Fibres processed according the coating of chitosan–titanium showed a diffraction plane of ($1 1 \bar{0}$), (1 1 0) and (0 2 0) appearing at a lower angle side with broader *d*-spacing plane. Moreover, there were two additional small peaks in the X-ray pattern of fabric from this coating at $2\theta = 24°$ and 31°, which are attributable to chitosan and titanium, respectively. This may be a result of chitosan–titanium film coating. This can be attributed to a relatively greater inclusion of chitosan–titanium film in the ($1 1 \bar{0}$) and (1 1 0) planes. The presence of such film would lead to a little expansion of that plane and a decrease the in *d*-spacing of the (0 2 0) plane, accompanied by a greater angle shift and decrease in diffraction intensity. Owing to the inclusion of chitosan–titanium film in the ($1 1 \bar{0}$) and (1 1 0) planes of cellulose fibre, the intermolecular hydrogen bonding between the sheets might be prevented, lowering the crystallinity index. This can be explained by the strong interaction between titanium film and cellulose fibre that destroyed the close packing of the cellulose molecules. Whereas in case of chitosan–zirconium film, the diffraction intensity increase of (0 2 0) plan with a smaller angle shift. Owing to the little inclusion of chitosan–zirconium film in the ($1 1 \bar{0}$) and (1 1 0) planes of cellulose fibre, heightening the crystallinity index that explained by a weak interaction between the fibre and film. Moreover, the steric effect of zirconium atom

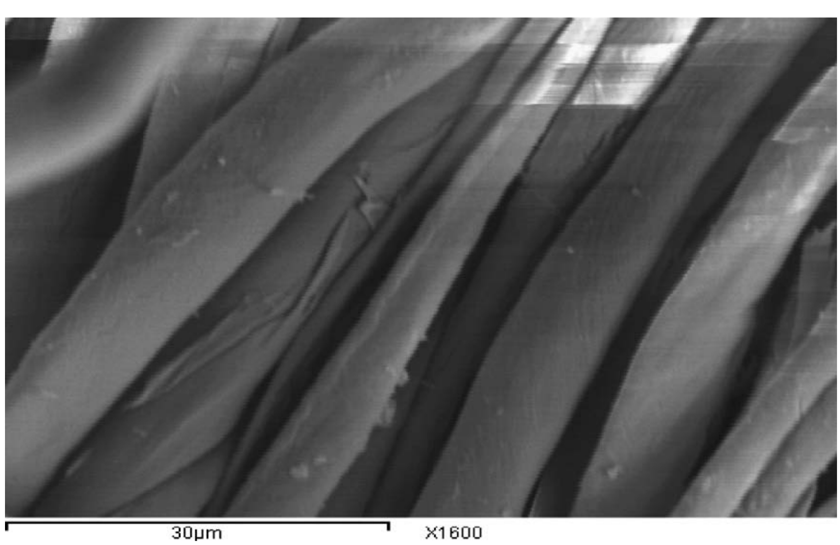


Fig. 2. Scanning electron microscope for untreated cotton fabric as blank.

size (larger than titanium) decreases the affinity of this film toward the cellulose chain.

Moreover, the presence of both zirconium and titanium in the film showed the highest crystallinity index of the fibre owing to week interaction occurred between the fibre and zirconium–titanium–chitosan film that keeps the close packing of the cellulose molecules.

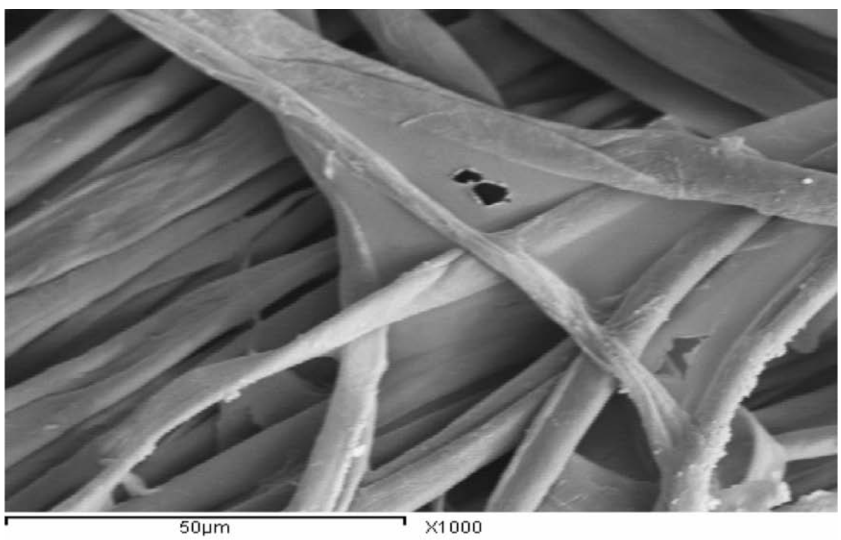
3.1.2. Morphology observations

From SEM-EDX (Figs. 2–7), chitosan–titanium film sorption on cotton is the highest where the N% contents are 7.16%, 3.96% and 9.67% corresponding to chiosan film, chitosan–zirconium and chitosan–titanium film sorption on cotton, respectively, due to a favorable electrostatic balance between the chitosan–titanium film and cotton.

SEM figures show the cross-section of chitosan film and chitosan–titanium film sorptions on cotton are smooth and homogenous rather than chiotsan–zirconium film. Also, this result indicates good compatibility between the chitosan–titanium film and cotton.

3.2. Oily stain release rating (OSR)

From Table 1, it is clear that: (i) incorporation of the metal oxides in the chitosan film have practically positive effects on the increasing of OSR values, regardless of the used metal oxides and its mixture; (ii) the increase in the OSR values depends on the film, metal oxide type as well as its mixture, i.e. Ti > Zr > Ti/Zr > Zr/Ti > chitosan coated fabric > none. These may be due to chemical composition, location and extent of distribution, functionality



Element	Weight%	Atomic%
C	60.60	60.19
N	7.61	7.16
O	31.79	32.65
Totals	100.00	

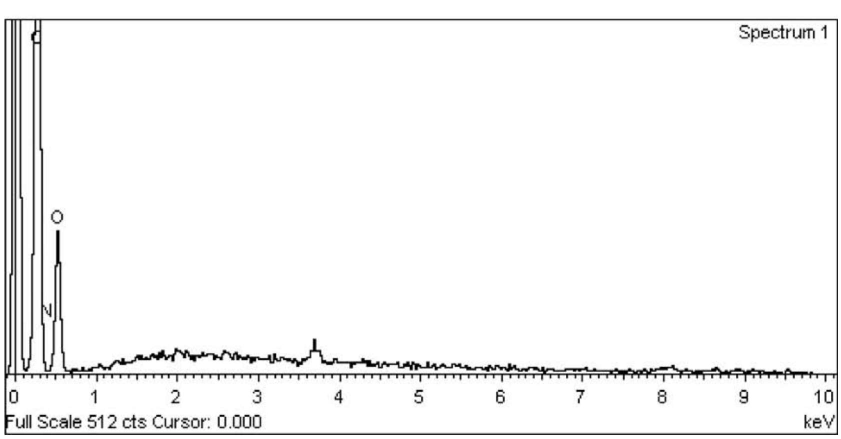
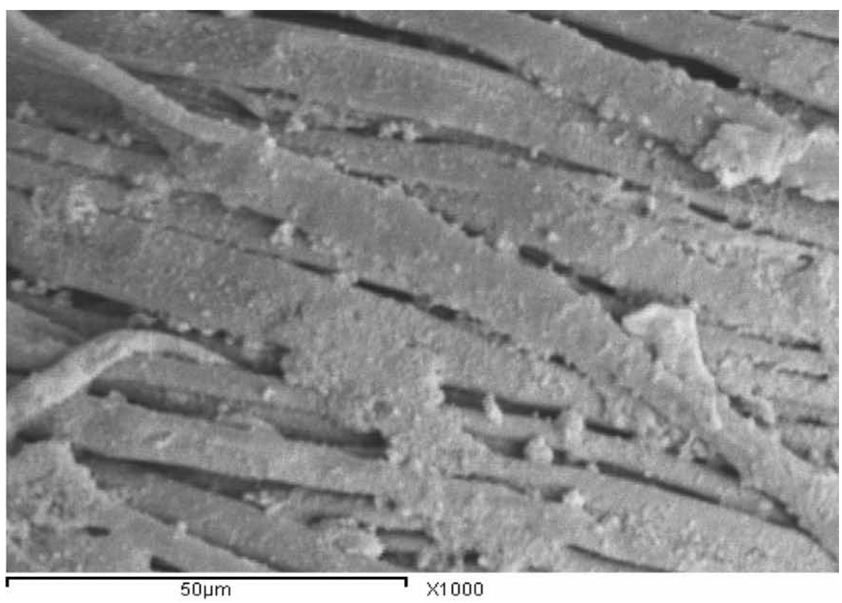


Fig. 3. SEM-EDX for cotton coated with chitosan film.

and mode of interaction (physically and/or chemically), hydrophilic/hydrophobic nature of the formed film, ability to pick-up and retain the oily stain, extent of modification and lubrication of the fabric (Hashem & Ibrahim, 2002; Ibrahim, Refai, Ahmed, & Youssef, 2005; Ibrahim, Refai, Youssef, & Ahmed, 2005).

3.3. UV-protection

To evaluate the potential effectiveness of certain metal oxides incorporated into chitosan film and coated to cotton fabric, for enhancing the UPF values, two metal oxides were used for their ability to upgrade the inherent UV-protection against the harmful UV-radiation. Results of Table 1 indicate that: (i) increasing the metal concentration results in the higher UPF values as well as the better sun-protection category are attributed to the presence of higher metal content compare to the untreated substrate, (ii) the extent of improvement in the UV-protecting properties is determined by the type of metal oxide and follows the descending order: Ti > Zr > Ti/Zr > Zr/Ti > coated fabric > none, and (iii) the variation in the UV-screening power of treated fabric samples in presence of the aforementioned metal oxides, reflects the differences among them in: molecular weight, content, particle size, surface area, location and extent of distribution, blocking property and absorbing capacity (Ibrahim, Refai, Ahmed, et al., 2005; Ibrahim, Refai, Youssef, et al., 2005; Yadav et al., 2006).



Element	Weight%	Atomic%
C K	66.12	66.89
N K	4.19	3.96
O K	29.03	29.05
Zr L	0.66	0.10
Totals	100.0	

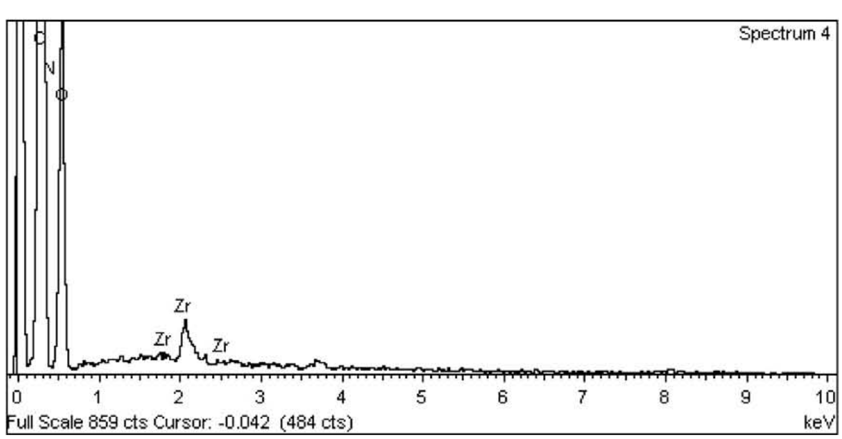


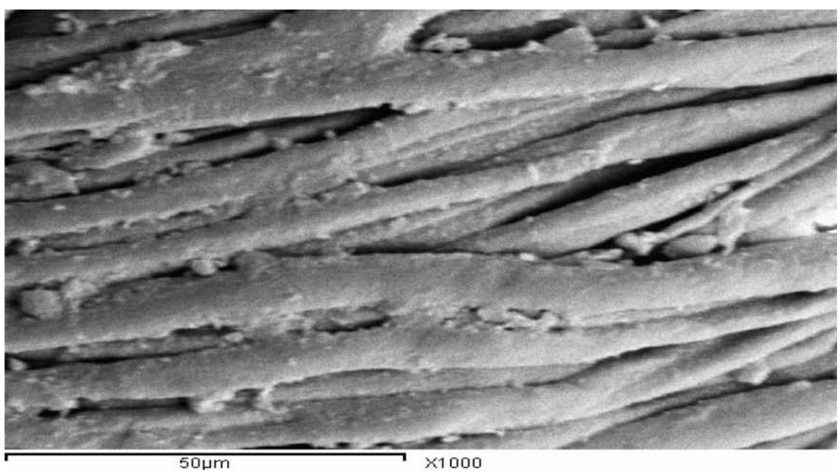
Fig. 4. SEM-EDX for cotton fabric coated with chitosan containing Zr.

3.4. Antibacterial activity

For a given treatment conditions, the data in Table 2 signify that: (i) antibacterial activity of the corresponding metal oxides have a positive impact on enhancing the antibacterial activity, (ii) the extent of improvement in the % RBC of both the G +ve and the G −ve bacteria is determined by the type of metal oxide and follows the descending order: Zr-oxide > Ti-oxide > mixed oxides > chitosan film coated fabric > none, i.e. the higher the % RBC values, the higher the antibacterial activity of the coated fabric samples, (iii) the outstanding effect of the metal oxides, i.e. Zr-oxide, reflecting their ability to react with cellular proteins thereby inactivating and killing them more effectively than Ti-oxide and chitosan film alone (Purwar & Joshi, 2004). (iv) The synergistic effect of: the combination of heavy metals with the protein-thiol group, the interaction of metal oxide with the moisture retained by the cellulose to generate H_2O_2 which is harmful to the cells of living microorganisms, in addition to the ability of heavy metals to absorb UV-light and its contribution to enhance the antibacterial activity (Ghule, Ghule, Chen, & Ling, 2006; Kim et al., 2006), reflects the high potentially and usefulness of them as antibacterial/UV-protecting agents, and (v) the durability of deposited metal oxides to severe washing (30-laundering cycles) as shown in Table 3 most probably due to their fixation onto cellulose structure through hydrogen bonding (Kim et al., 2006).

3.5. Laundering cycles

Coated fabric samples were laundered to 10–30 cycles in a home laundering machine and adjusted to antibacterial activity tests according to AATCC test Method 124. The results are given in Table 3 and reveal that, increasing laundering cycles up to 10 cycles has practically no effect on the antibacterial activity of the coated samples. Further increasing in laundering cycles up to 20 cycles has a negative impact on the retained antibacterial activities depending on the type of metal oxides and its mixtures, ranged



Element	Weight%	Atomic%
C K	67.01	66.81
N K	9.76	9.67
O K	16.13	21.45
Ti K	7.10	2.07
Totals	100.00	

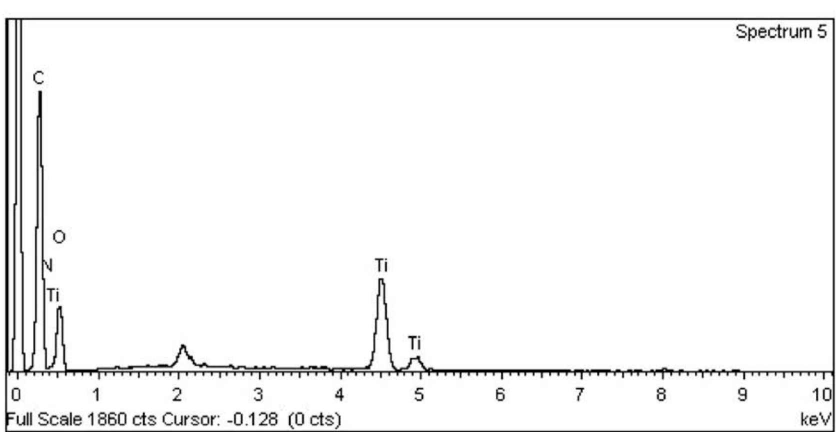


Fig. 5. SEM-EDX for cotton fabric coated with chitosan containing Ti.

from (80–83%) for Ti, (90–93%) for Zr and (89–90%) for the mixtures (Ti/Zr and Zr/Ti) depending on the type of microorganisms. After 30 laundering cycles the antibacterial activity against gram positive and Gram-negative bacteria remains at over 80% most probably due to their fixation onto the cellulose structure through hydrogen bonding (Ghule et al., 2006).

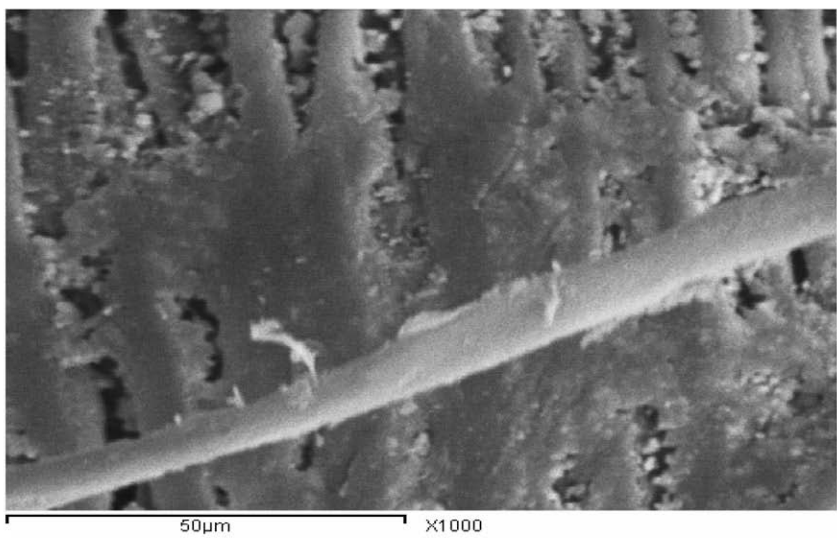
4. Conclusion

Cotton fabrics coated with chitosan film containing titanium oxide and/or zirconium oxide for enhancing multifunctional properties were prepared. The prepared fabric samples have a positive impact on UV-protection and the extent of protection follows the decreasing order: Ti > Zr > Ti/Zr > Zr/Ti > chitosan film > none.

Table 2
Antibacterial activity of prepared coated fabrics containing metal oxides.

Samples	*Staphylococcus aureus*		*Escherichia coli*	
	Count/ml	RBC (%)	Count/ml	RBC (%)
Blank		–		–
Chitosan film	3×10^7	80.0	5×10^7	70.5
Zr-oxide	25×10^5	97	7×10^6	95
Ti-oxide	3×10^7	86.0	4×10^7	84
Zr/Ti-oxides	8×10^6	96	15×10^6	93
Ti/Zr-oxides	15×10^5	96	5×10^6	93

Cotton fabrics coated with chitosan film incorporated with titanium and/or zirconium oxides have remarkable positive effects on the increasing of OSR values and the increasing of the OSR values



Element	Weight%	Atomic%
C K	53.89	60.26
N K	8.85	9.19
O K	26.69	27.43
Ti K	9.94	3.02
Zr L	0.63	0.1
Totals	100.00	

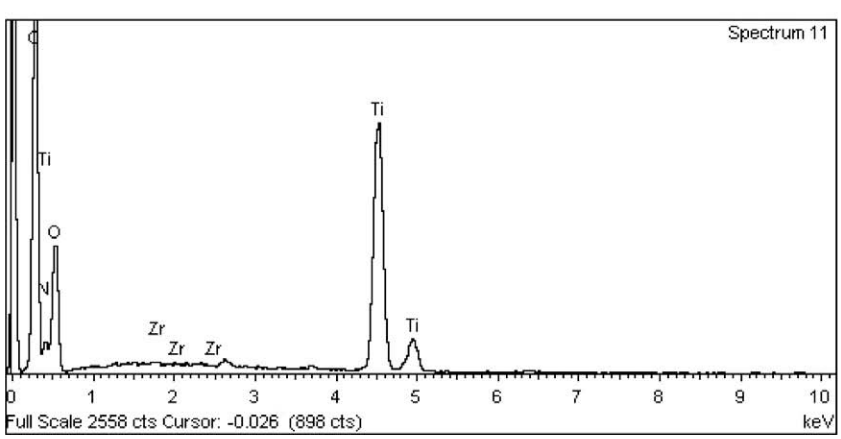
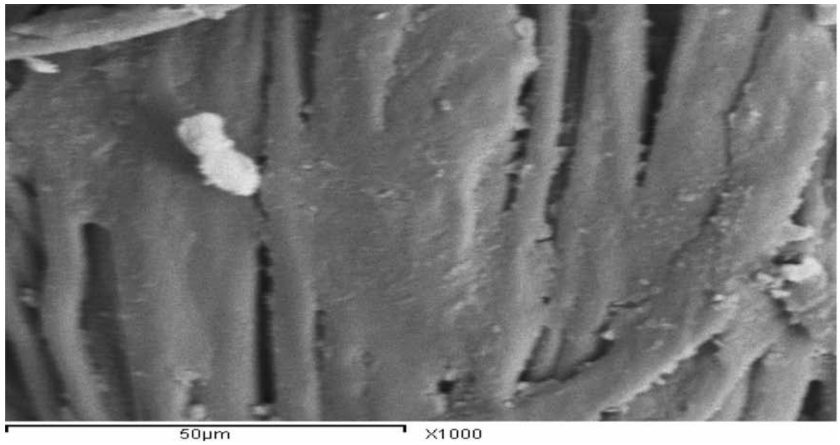


Fig. 6. SEM-EDX for cotton fabric coated with chitosan containing 0.5 g Zr + 1 g Ti.



Element	Weight%	Atomic%
C K	62.58	63.93
N K	8.51	8.36
O K	20.42	25.80
Ti K	4.53	1.31
Zr L	3.96	0.60
Totals	100.00	

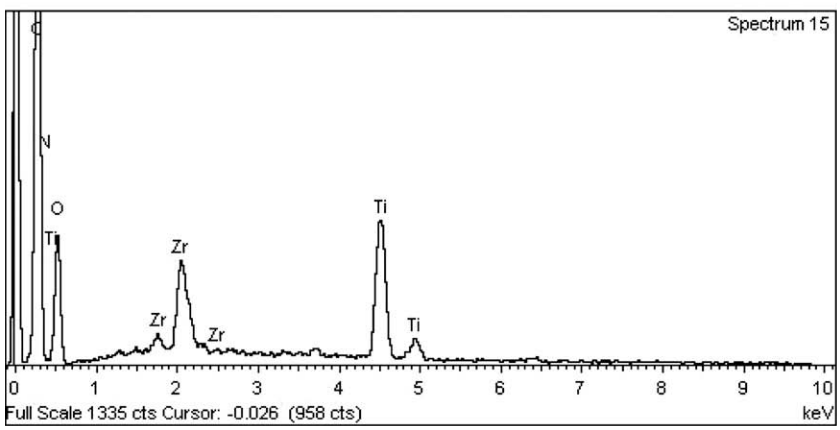


Fig. 7. SEM-EDX for cotton fabric coated with chitosan containing 0.5 g Ti + 1 g Zr.

Table 3
Effect of laundering cycles on the antibacterial activity of the prepared coated fabric samples.

Laundering cycles	Percent of reduction bacterial count (% RBC)							
	Staphylococcus aureus				*Escherichia coli*			
	Ti-oxide	Zr-oxide	Ti/Zr-oxides	Zr/Ti-oxides	Ti-oxide	Zr-oxide	Ti/Zr-oxides	Zr/Ti-oxides
0	86	97	96	96	84	95	93	93
10	86	97	96	96	84	95	93	93
20	83	93	90	90	80	90	89	89
30	80	90	88	88	78	88	86	86

depending on the type of metal oxides incorporated (i.e. Ti > Zr > - Ti/Zr > Zr/Ti > chitosan film > none).

Furthermore the antibacterial activities of the corresponding metal oxides have a positive impact on the Gram-positive bacteria (*S. aureus*) and Gram-negative bacteria (*E. coli*). The extent of improvement in the reduction of bacterial count percent is determined by the type of metal oxides and follows the decreasing order: Zr > Ti > Ti/Zr = Zr/Ti > chitosan film > none.

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