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Chitosan and monochlorotriazinyl-β-cyclodextrin finishes improve antistatic properties of cotton/polyester blend and polyester fabrics

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ABSTRACT

Permanent fixation of chitosan or monochlorotriazinyl-β-cyclodextrin (MCT-β-CD) onto cotton/polyester and polyester fabrics was carried out and all parameters controlling the efficiency of both fixation reactions were studied. The amounts of MCT-β-CD or chitosan fixed onto the treated fabrics were estimated in terms of percent nitrogen content. Results obtained reveal that finishing the said fabrics with either MCT-β-CD or chitosan generally improves the water uptake capacity of the finished fabrics without harmful effect on their physico-mechanical properties. The water uptake capacities of MCT-β-CD finished fabrics were found to be higher than the corresponding values recorded for chitosan finished fabrics. The general improvement in water uptake of the finished fabrics is attributed to the hydroxyl groups introduced to the fabric structure through finishing with either MCT-β-CD or chitosan, which increase the fabric's ability to absorb more water and moisture from air. The presence of more water in such fabrics increases their electrical conductivity and thus improving their antistatic properties.

1. Introduction

Static charges usually build up on the surface of synthetic polymers such as polyester and nylon fibers because they retain low moisture content compared with the more hydrophilic cellulosic fibers. Moreover, at the same moisture content, hydrophobic fibers like nylon or polyester exhibit lower conductivity than do hydrophilic cellulosic fibers (Reck, 1985; Sereda & Feldman, 1964). Although static charges are dissipated through the air, most of the static charges developed on synthetic fibers are dissipated by an induction mechanism along the filaments to an electrical ground. This can be accomplished by either incorporating a conductive material into the polymer itself, or by the application of an antistatic coating to the yarn (Bajaj, Gupta, & Ojha, 2000; Seong, 2001).

Many researches have been conducted to develop antistatic treatment for polyester fabrics. Since polyester has hydroxyl functional groups, it was possible to crosslink them with amino groups, hygroscopic salts and some other compounds to increase the conductivity of the fiber surface and thus reducing the accumulated charges on polyester fabrics (Abdel-Fattah & Saad, 1988; Rybicki & Mielicka, 1996; Seong, 2001; Takahashi, Ohta, Kadota, & Saeki, 1989). Some antistatic finishes are based on treating polyester with metal salts in order to increase the conductivity of the fiber surface (Rybicki & Mielicka, 1996). Antistatic agents are chemicals that are applied to the surface of synthetic fabrics to control the tendency of these fabrics to accumulate static charges. Although these agents can function either by reducing the generation of electrostatic charges, by increasing the conductivity of the materials to which they are applied or by both mechanisms, most antistatic agents act through the conductivity mechanism. Antistatic finishing of textiles using nanotechnology has been reported. Nano-size zinc oxide (Zhou, Chu, Tang, & Gu, 2003) and nano-size antimony-doped tin oxide (ATO) (Wu et al., 2002) could impart antistatic properties to synthetic fibers. TiO_2, ZnO and ATO provide antistatic effects because they are electrically conductive material. Such materials help to effectively dissipate the static charges which are accumulated on the fiber surface.

An increasing demand develops for introducing active agents to textile materials (fabric and non-woven) by chemical means in order to create additional properties (functional textiles). For synthetic fibers, this functionalization routes may introduce better hydrophilic behavior (water retention and sweat transport, etc.). Chitosan, cyclodextrins and number of their derivatives constitute a group of chemicals belonging to such type of auxiliaries (Seong, 2001).

Cyclodextrins can be obtained by enzymatic degradation of starch leading to the formation of oligosaccharides consisting of six, seven or eight glocopyranose units known as α-, β- or γ-cyclodextrin, respectively. The cyclodextrin molecule consists

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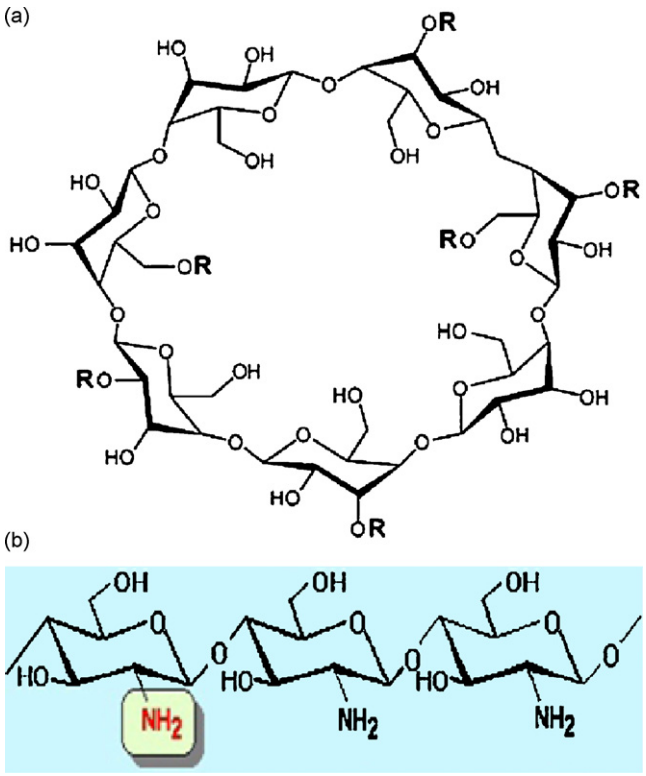


Fig. 1. Chemical structure of β-cyclodextrins (a) and chitosan (b).

of macrocyclic ring, in which all hydroxyl groups are located at the top and the bottom of the ring as shown in Fig. 1(a). Monochlorotriazinyl-β-cyclodextrin (MCT-β-CD) is the first reactive cyclodextrin derivative, manufactured on an industrial scale. Cyclodextrins and their derivatives are suitable for a wide variety of possible uses such as stabilization, masking or controlled release of hydrophobic substances. Surfaces like textiles with nucleophilic groups can be modified with cyclodextrins and their derivatives via well established methods (Abdel-Mohdy, El-Aref, Hashem, & Aly, 2005).

Chitin [poly-(1 → 4)-N-acetyl-D-glucosamine] is the second most abundant natural polysaccharide found on earth next to cellulose. Chitin is the main component in the shell of crustaceans such as shrimps, crab and lobster. It is also found in exoskeletons of insects and in the cell wall of some fungi (Rinaudo, 2006). Chitin has the same backbone as cellulose, but it has an acetamide group in the C-2 position instead of the hydroxyl group. By partial deacetylation under alkaline conditions, one obtains chitosan, which is the most important chitin derivative in terms of applications. Chitosan is a linear polysaccharide consisting of β-(1-4)-2-amino-deoxy-D-glucopyranose units and its idealized structure is similar to cellulose as shown in Fig. 1(b). In the past 30 years it has been demonstrated by a number of researchers that chitosan has a great potential for a wide range of uses due to its biodegradability, biocompatibility, antimicrobial activity, non-toxicity and versatile chemical and physical properties. The applications of chitosan include pharmaceutical and medical applications, paper production, wastewater treatment, biotechnology, cosmetics, food processing and agriculture (Fouda, 2005; Hudson & Smith, 1998). Also utilization of chitosan in textile finishes improving fabric dyeability and imparting antimicrobial properties has been reported by several researchers (Alonso et al., 2009; Giri Dev, Venugopal, Sudha, Deepika, & Ramakrishna, 2009; Jocic et al., 2005; Lim & Hudson, 2004). In addition to the aforementioned chitosan uses in textile finishing, chitosan shows high moisture regain percent even at low relative humidity and this property makes chitosan ideal candidate as an antistatic finishing agent (Seong, 2001).

Based on the above mentioned properties and functionality of both monochlorotriazinyl-β-cyclodextrin (MCT-β-CD) and chitosan, the aim of the present study is to impart antistatic properties to polyester and cotton/polyester fabrics through finishing of the said fabrics with MCT-β-CD or chitosan. The approach in this study is to modify only the fabric surface with the antistatic agent and thus keeping the mechanical properties of the bulk fibers unaffected.

2. Experimental

2.1. Materials

Polyester fabric was supplied by Misr Company for Spinning and Weaving, Mahalla El-Kobra, Egypt. The fabric was washed with aqueous solution containing 2 g/l non-ionic detergent for 15 min at 60 °C using material to liquor ratio of 1:25, then rinsed thoroughly with cold water and dried at 25 °C. Plain weave polyester/cotton blend, 67/33 of 107 g/m^2 was supplied by El-Nasr Company for Spinning, Weaving and Dyeing, Mehalla El-Kobra, Egypt. The fabric was further purified by scouring in an aqueous solution containing 2 g/l sodium carbonate and 2 g/l non-ionic detergent for 60 min at the boil using material to liquor ratio of 1:25, then rinsed thoroughly with hot water and dried at 25 °C. Monochlorotrizinyl-β-cyclodextrin (MCT-β-CD) was supplied by Wacker-Chemie GmbH, München, Germany. High molecular weight chitosan was supplied by Sigma–Aldrich Chemie GmbH, Riedstr., Germany. All other chemicals were of laboratory grade.

2.2. Fixation of MCT-β-CD on the fabrics

Fixation of MCT-β-CD on polyester and cotton/polyester fabrics was carried out according to previously reported methods (Lo Nostro, Frantoni, Ridi, & Bagllioni, 2003; Ruppert, Knittel, & Buschmann, 1997; Scalia et al., 2006). The fixation procedure was to soak the fabric samples for 5 min at 25 °C in an aqueous solution containing (2–8%, w/v) MCT-β-CD and (20%, w/v) Na_2CO_3 under continuous shaking. The samples were then squeezed to wet pick up of ca 100%. To minimize the reaction of MCT-β-CD with air moisture, the squeezed samples were cured in an oven at (110–140 °C) for (5–20 min) at atmospheric pressure, and then carefully rinsed with demineralized water to remove the unreacted MCT-β-CD. A systematic study of the parameters controlling the extent of reaction was carried out. These parameters include concentration of MCT-β-CD, temperature and time of curing. The nitrogen content of the treated fabric was estimated as per standard Kjeldhal method (Vogel, 1975) and then the amount of MCT-β-CD fixed on the fabric was calculated from the estimated nitrogen content according to the following equation:

$$\text{fixed MCT-β-CD}\,(\%) = \frac{N\%}{7} \times 100$$

2.3. Fixation of chitosan

Butane-1,2,3,4-tetracarboxylic acid (BTCA) was used to fix chitosan thermally on the surface of polyester or cotton/polyester fabric surface in the presence of sodium hypophosphite (SHP) as a catalyst. Chitosan samples of different molecular weights were dissolved (1%, w/v), separately in 1% acetic acid at 60 °C under continuous stirring for 12 h and then were filtered to get rid of any insoluble matter. The fabric samples were immersed in finishing solutions, each of them is composed of 6 g of the crosslinking agent

BTCA and 6 g of the catalyst SHP, all dissolved in 100 ml of 1% chitosan solution. The fabrics were then padded to wet pick up ca. 100%, dried at 80 °C for 5 min and finally cured for 5–20 min at 140–170 °C. The cured fabrics were cooled down to room temperature, washed with 1% acetic acid to remove unfixed chitosan, washed thoroughly with cold water until neutral and finally air dried (El-Tahlawy, El-Bendary, El-Hendawy, & Hudson, 2005). The nitrogen content of the treated fabric was estimated as per standard Kjeldhal method (Vogel, 1975) and then the amount of chitosan fixed on the fabric was calculated from the estimated nitrogen content according to the following equation:

$$\text{fixed chitosan}\,(\%) = N\% \times \frac{161}{14}$$

2.4. Tests and measurements

2.4.1. Mechanical properties

Tensile strength (kg) and elongation at break (%) of finished and untreated fabrics were measured according to ASTM D 1682, ISO 5082, ISO 5081, using Instron Series 2701 Pneumatic Grip Control, Assembled in USA. The tests were carried out at Clothing and Knitting Department, Textile Research Division, National Research Center, Egypt.

2.4.2. Surface roughness

Surface roughness of finished and untreated fabrics was measured according to JIS-94 standard using surface roughness tester Model SE 1700α. Surface roughness measurements were carried out at Spinning and Weaving Department, Textile Research Division, National Research Center, Egypt.

2.4.3. Water uptake

Finished and untreated fabric samples were immersed in water until equilibrium was reached. The excess water on the surface of wet samples was wiped out with blotting paper and the water uptake of the samples was calculated using the following equation:

$$\text{water uptake}\,(\%) = \frac{\text{swollen fabric weight} - \text{dry sample weight}}{\text{dry sample weight}} \times 100$$

2.4.4. Antistatic property measurement

Static electricity of finished and untreated fabrics was measured using electricity collect type potentiometer model KS-525 (Kasuga Denki, Inc., Japan). The antistatic property measurements were carried out at Spinning and Weaving Department, Textile Research Division, National Research Center, Egypt.

3. Results and discussions

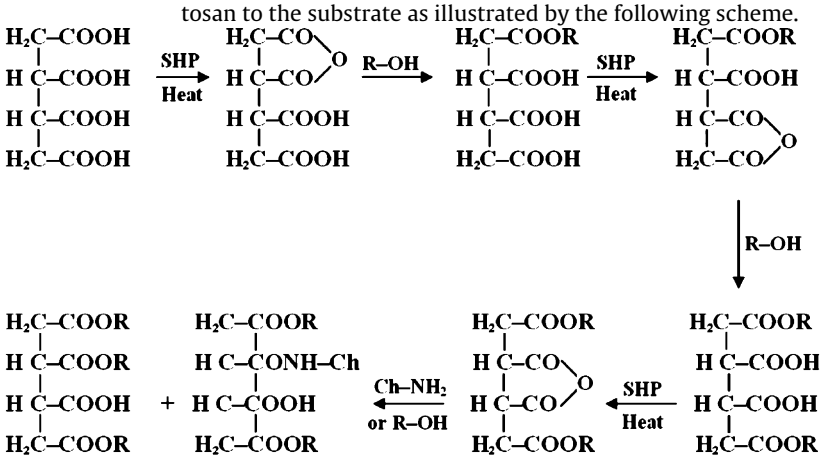
3.1. Fixation of chitosan and MCT-β-CD onto polyester and cotton/polyester fabrics

3.1.1. Tentative mechanism for fixation

Cellulose is composed of glucose molecules condensed and linked together linearly by means of 1,4-glucosidic bonds. Cellulose has three functional hydroxyl groups per repeated glucose unit. The hydroxyl groups in positions 2 and 3 are secondary hydroxyl groups, whereas the one in position 6 is primary hydroxyl group. The primary hydroxyl group in position 6 is the first to react in esterification reaction.

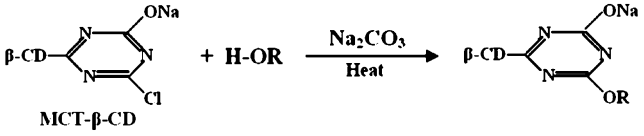
Polyester is a synthetic polymer prepared by polycondensation of terephthalic acid and ethylene glycol.

Butane-1,2,3,4-tetracarboxylic (BTCA) acid is known to be used as crease recovery finishing agent for cotton and cotton/polyester fabrics. The role of BTCA in this finishing process is to acts as crosslinking agent in presence of catalyst like sodium hypophosphite (SHP) (El-Tahlawy et al., 2005; Hsieh, Chen, & Wei, 2003). The crosslinking reaction proceeds via esterification mechanism between the carboxylic groups of BTCA and the hydroxyl groups of cellulose and/or polyester. During the drying and curing steps, part of the crosslinking agent (BTCA) is consumed in linking chitosan to the substrate as illustrated by the following scheme.



(R-OH is cell-OH in case of cotton or PE-OH in case of polyester and Ch-NH_2 is chitosan)

The fixation of MCT-β-CD onto polyester or cotton/polyester fabrics proceeds via etherification mechanism as illustrated by the scheme given below.



(R-OH is cell-OH in case of cotton or PE-OH in case of polyester)

3.1.2. Factors affecting the amounts of MCT-β-CD and chitosan bonded to the fabrics

The amounts of MCT-β-CD and chitosan bonded to the fabrics were mostly dependent on MCT-β-CD and chitosan concentrations,

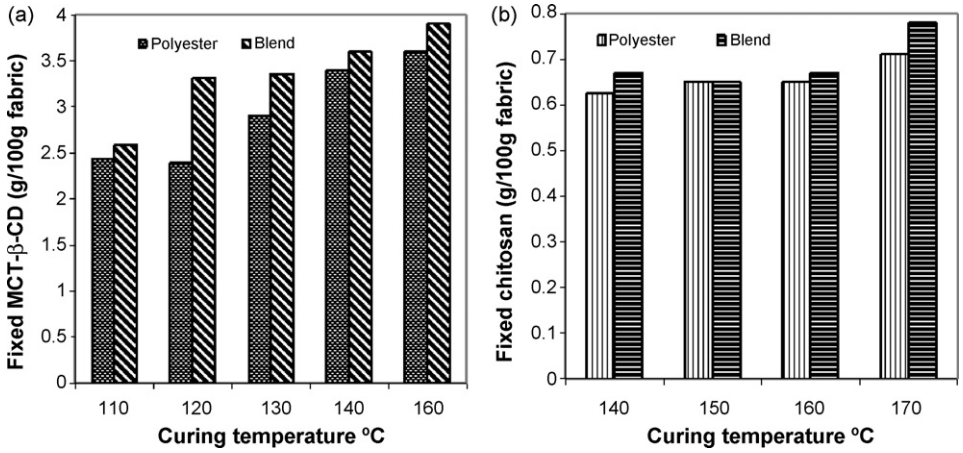


Fig. 2. Effect of curing temperature on the fixed amount of MCT-β-CD (a) and chitosan (b). Reaction conditions: (a) [MCT-β-CD], 5% (owf); wet pickup, 100%; curing time, 15 min and (b) [chitosan], 1% (owf); chitosan MW, 0.75×10^5; wet pickup, 100%; curing time, 10 min.

the catalyst type and concentration, the curing temperature and the curing time.

3.1.2.1. Effect of curing temperature. The effect of curing temperature on the amounts of fixed MCT-β-CD and chitosan is shown in Fig. 2(a and b). The fixed amounts of MCT-β-CD and chitosan were found to be nearly proportionally related to the curing temperature. On the other hand, for chitosan fixation (b), fabrics cured at temperatures higher than 150 °C were found to show remarkable yellowness.

3.1.2.2. Effect of curing time. Fig. 3(c and d) shows the effect of curing time on the percent fixed MCT-β-CD and chitosan. The figure demonstrates that the longer the curing time, the higher is the amount of MCT-β-CD or chitosan fixed onto the fabrics. From the figure one can notice that for MCT-β-CD fixation (c), there is continuous increase in the fixed amount of MCT-β-CD onto both fabrics with increasing the curing time. For chitosan fixation (d), the amounts of fixed chitosan on both fabrics increase until the curing time reaches 10 min and then remain almost constant after 10 min.

3.1.2.3. Effect of finishing agent concentration. The effect of MCT-β-CD or chitosan concentrations on their amounts fixed onto polyester and blend fabrics is shown in Fig. 4(e and f). From the figure one can notice a continuous increase in the amounts of fixed MCT-β-CD (e) and chitosan (f) by increasing their concentrations in the finishing solutions. Higher concentrations of MCT-β-CD or chitosan mean greater availability of their molecules in the reaction medium, and therefore, higher probability of molecular collisions is expected, which ultimately leads to higher reaction efficiency.

3.1.2.4. Effect of crosslinking agent type on the amount of fixed chitosan. The effect of crosslinking agent type on the amount of fixed chitosan onto both polyester and blend fabrics is shown in Fig. 5. It is clear from the figure that maximum chitosan fixation is obtained on using BTCA as crosslinking agent.

3.1.2.5. Effect of chitosan molecular weight on the amount of fixed chitosan. The effect of chitosan molecular weight on the amount of fixed chitosan is represented in Fig. 6. It was found that keeping the same reaction conditions, increasing chitosan molecular weight leads to decreasing the amount of fixed chitosan onto both polyester and blend fabrics. This is because the higher the chitosan molecular weight, the higher will be the apparent viscosity of the finishing bath and consequently the lower the ability of the active ingredients to penetrate to the bulk of the fabric. Due to low penetration ability of the viscous finishing solution, there will be good chance for chitosan side reaction. This means that there will be higher probability for the crosslinking agent to react with chitosan (mobile phase) rather than to react with the fabric hydroxyl groups (immobile phase).

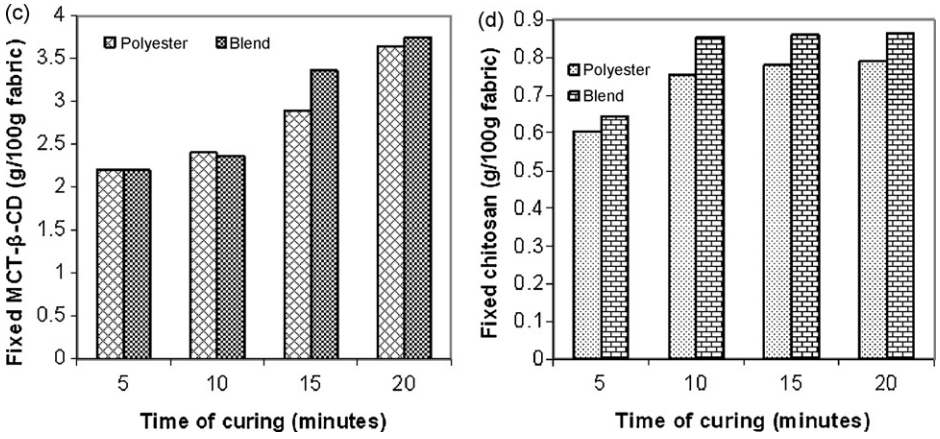


Fig. 3. Effect of curing time on the fixed amount of MCT-β-CD (c) and chitosan (d). Reaction conditions: (c) [MCT-β-CD], 5% (owf); wet pickup, 100%; curing temperature, 130 °C and (d) [chitosan], 1% (owf); chitosan MW, 0.75×10^5; wet pickup, 100%; curing temperature, 150 °C.

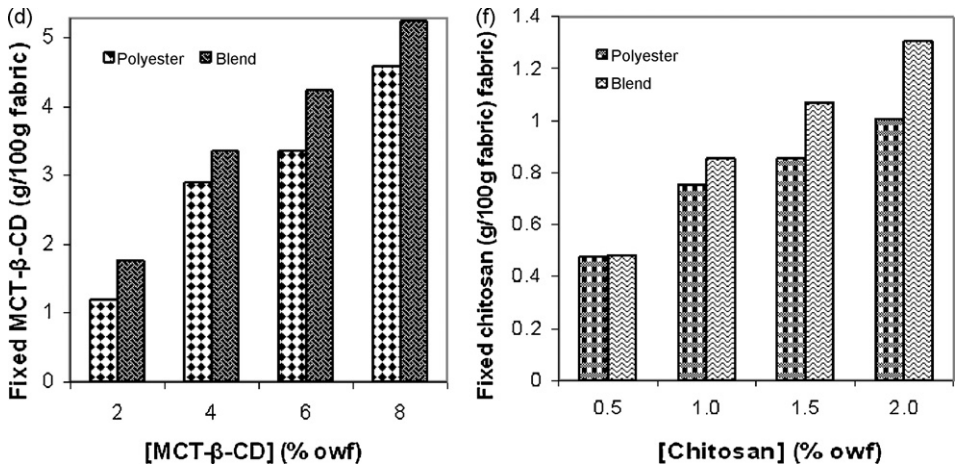


Fig. 4. Effect of finishing agent concentration on the fixed amount of MCT-β-CD (e) and chitosan (f). Reaction conditions: (e) wet pickup, 100%; curing temperature, 130 °C; curing time, 15 min and (f) Chitosan MW, 0.75×10^5; wet pickup, 100%; curing temperature, 150 °C; curing time, 10 min.

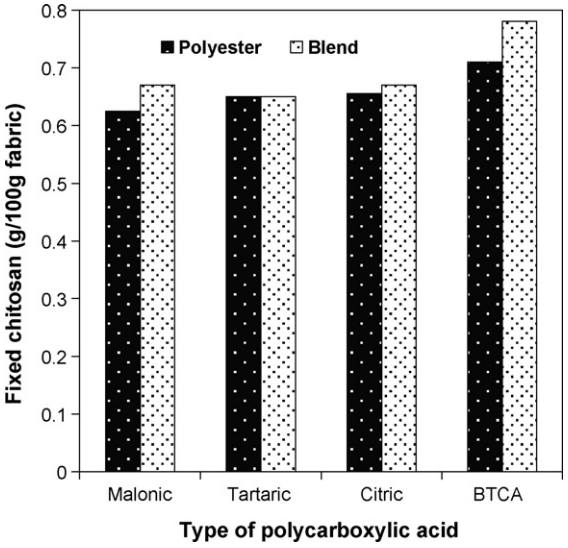


Fig. 5. Effect of crosslinking agent type on the amount of fixed chitosan. Reaction conditions: [chitosan], 1% (owf); Chitosan MW, 0.75×10^5; wet pickup 100%; curing temperature, 150 °C; curing time, 10 min.

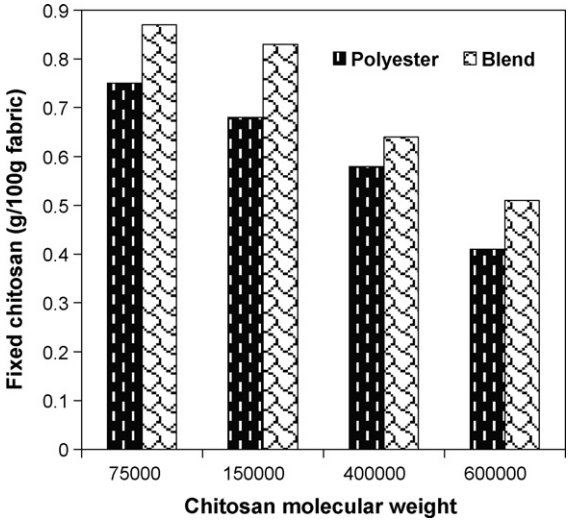


Fig. 6. Effect of chitosan molecular weight on the amount of fixed chitosan. Reaction conditions: [chitosan], 1% (owf); wet pickup, 100%; curing temperature, 150 °C; curing time, 10 min.

3.2. Physico-mechanical properties

Physico-mechanical properties of fabrics finished separately with MCT-β-CD and chitosan were measured and listed in Tables 1 and 2, respectively.

3.2.1. Tensile properties

For MCT-β-CD finished fabrics (Table 1), as a general remark, the finished fabrics (both polyester and blend fabrics) showed reasonable loss in tensile strength compared with the untreated fabrics. However, increasing the amount of fixed MCT-β-CD did not lead to great loss in tensile strength. This implies that finishing of both polyester and blend fabrics with MCT-β-CD does not lead to high molecular degradation. The results of Table 1 also show that for both fabrics, the percent elongation at break in general decreases after finishing and slightly decreases by increasing the amount of fixed MCT-β-CD. This could be ascribed to the rigidity imposed to

Table 1
Effect of percent MCT-β-CD fixed on the fabrics on physico-mechanical properties, water uptake and antistatic properties of treated fabrics.

Fabric	Fixed CD (g/100 g)	Tensile strength (kg)	Elongation at break (%)	Roughness	Water uptake (%)	Antistatic properties
PE	0	93.23	33.95	14.99	44.06	−6
	7.22	89.26	27.12	16.6	55.09	−2
	7.8	89.16	24.29	17.25	57.37	−1
	8.5	87.55	24.95	18	58.2	−0.2
PE/CO	0	64.9	14.58	13.33	46.74	−2.5
	9.19	63.92	14.33	15.73	67.93	0
	11.57	60.79	14.3	15.93	68.33	0
	12	59.5	13.29	16.71	69.33	0

PE: polyester fabric; PE/CO: polyester/cotton blend; CD; MCT-β-CD.

Table 2
Effect of percent citosan fixed on the fabrics on physico-mechanical properties, water uptake and antistatic properties of treated fabrics.

Fabric	Fixed chitosan (g/100 g)	Tensile strength (kg)	Elongation at break (%)	Roughness	Water uptake (%)	Antistatic properties
PE	0	93.23	33.95	14.99	44.06	−6
	1.55	93.15	32.20	17.25	48.46	−3.4
	1.88	92.52	30.62	17.8	48.98	−2.2
	2.2	91.96	28.41	18.83	49.06	−1.4
PE/CO	0	64.9	14.58	13.33	46.74	−2.5
	1.08	48.74	11.91	16.71	58.40	0
	1.17	46.51	11.37	17.73	58.86	0
	1.32	45.82	11.29	18.95	60.51	0

PE: polyester fabric; PE/CO: polyester/cotton blend.

the fabric structure due to the presence of excessive amounts of MCT-β-CD.

With respect to chitosan finished fabrics (Table 2), for the tensile strength, the trend is same in case of polyester fabrics and somehow different in case of blend fabrics. In case of blend fabrics, the results showed general high loss in tensile strength of the finished fabrics compared with unfinished blend. Moreover, the loss in tensile strength was found to increase slightly by increasing the amount of fixed chitosan. The general high loss in tensile strength of polyester/cotton fabric and the small loss in tensile strength of polyester fabric when they are finished with chitosan under almost the same conditions imply that the cotton component of the blend fabric undergoes high molecular degradation under the effect of finishing conditions and thus leads to general loss in the tensile strength of the blend. The change in percent elongation at break for chitosan finished fabrics follows the same trend as in case of MCT-β-CD finished fabrics and can be discussed in the same manner.

3.2.2. Surface roughness

The surface properties of the antistatic treated fabrics are very important because these fabrics are used mainly for garments in direct contact with human skin, that surface roughness has strong effect on the wearer comfort. Surface roughness results of polyester and blend fabrics treated with MCT-β-CD and chitosan are given in Tables 1 and 2, respectively. The results showed slight increase in the surface roughness of the finished fabrics compared with the untreated fabrics and that the higher the amount of finishing agent fixed on the fabric surface, the greater is the surface roughness. Based on the measured mechanical properties and surface roughness results, one has to control the amount of fixed finishing agent and keep it just enough to give reasonable antistatic effect in order not to lead to sever loss in tensile strength or high surface roughness which affects the wearer comfort.

3.3. Water uptake and antistatic properties

The water uptake results and the corresponding antistatic properties of polyester and polyester/cotton finished with MCT-β-CD are given in Table 1. The results show that the water uptake percent of finished polyester and blend fabrics increases as the amount of MCT-β-CD fixed onto the fabric surface increases. In case of polyester fabric the increase in water uptake reaches about 30% of the value recorded for the untreated polyester, while in case of polyester/cotton blend, the increase reaches 50% of the value recorded for the untreated fabric. For the water uptake percent and antistatic properties of polyester and polyester/cotton finished with chitosan (Table 2) one can notice the same trend but the increase in water uptake is reduced to 11% for polyester and 30% for polyester/cotton blend, related to the values recorded for the untreated fabrics. Based on the water uptake results listed in both tables one can conclude the following: (a) the water uptake of polyester/cotton blend is in general higher than the water uptake of the polyester fabric, which is attributed to the presence of hydrophilic cellulosic component in the blend fabric, (b) the water uptake capacity imparted by MCT-β-CD finishing is in general higher than the water uptake capacity imparted by chitosan finishing and this can be explained in terms of greater number of hydrophilic hydroxyl groups introduced to the fabric structure in case of MCT-β-CD finishing than the number of hydroxyl groups introduced through chitosan finishing.

The influence of absorbed water on the antistatic behavior of the finished fabrics is not difficult to interpret. The resistance of organic polymers to transfer electricity is in the order of 10^{16} Ω-cm, therefore they are considered to be insulators for electricity. Pure water has a specific electrical resistance of 10^8 Ω-cm, or 10^8 lower than the pure polymer. In addition, water that contains electrolytes (such as tap water) has electrical conductivity as much as 10^3 times greater than pure water. When exposed to an atmosphere of high relative humidity, fabrics finished with MCT-β-CD or chitosan absorb significant amounts of water. The presence of water in these fibers increases their electrical conductivity and thus decreases their tendency to accumulate static charges, and the greater the moisture content of the fabric, the better will be its antistatic properties.

4. Conclusion

A process for imparting durable antistatic properties to polyester and polyester/cotton blend was carried out by chemical fixation of either monochlorotriazinyl-β-cyclodextrin (MCT-β-CD) or chitosan onto the said fabrics. Pad-dry-cure technique was used in the finishing process, through which fixation of MCT-β-CD proceeds via etherification mechanism, while chitosan fixation proceeds via crosslinking and esterification mechanism in presence of butane-1,2,3,4-tetracarboxylic acid (BTCA) and sodium hypophosphite (SHP) as a catalyst.

The amounts of MCT-β-CD and chitosan bonded to the fabrics were mostly dependent on MCT-β-CD and chitosan concentrations, the curing temperature and the curing time. The physico-mechanical properties of the finished fabrics were measured and compared with the corresponding values measured for the untreated fabrics. Fabrics finished with either MCT-β-CD or chitosan showed general improvement in the water uptake capacity, compared with the untreated fabrics, without serious loss in the tensile strength or much increase in the surface roughness. The water uptake capacity imparted by MCT-β-CD finishing was found to be higher than the water uptake capacity imparted by chitosan finishing and this can be explained in terms of greater number of hydrophilic hydroxyl groups introduced to the fabric structure in case of MCT-β-CD finishing than the number of hydroxyl groups introduced through chitosan finishing. The presence of water in such finished synthetic fabrics increases their electrical conductivity and thus improving their antistatic properties.

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