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The Influence of Cotton/Polyester Blend Fabric Pre-treatment on Chitosan Functionalization

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Abstract

In this work, the influence of cotton/polyester blend fabric pretreatment on chitosan functionalization was investigated. For this purpose, a cotton/polyester blend fabric in twill weave was pretreated with 1.5 M NaOH and then functionalized with chitosan. Three wash cycles were performed to determine the durability. The zeta potential was measured with an electrokinetic analyzer EKA (Anton Paar) before and after functionalization, as well as after the first and third wash cycles, so that the presence of chitosan could be determined. The changes in mechanical properties - thread density, change in mass per unit area, tensile strength and elongation, spectral properties - whiteness, yellowing index and deviation from the white standard, moisture management and antimicrobial efficacy were determined according to standardized methods. Alkali pretreatment was discovered to enhance the amount of accessible hydroxyl groups of the cotton component and carboxyl groups of the polyester component in the blend, as well as the bonding of chitosan and hence wash resistance. Chitosan improves the blend's strength and antibacterial efficiency.

***Keywords**: cotton-polyester blend, chitosan, zeta potential, moisture management.*

1. Introduction

Cotton fiber is a cellulose fiber with excellent absorbency and comfort. Natural impurities such as pectin, waxes, organic acids, proteins, and minerals are eliminated during the scouring process, but colored compounds - pigments - remain. Chemical bleaching of pigments results in fiber whiteness, although cellulose oxidative damage might occur, resulting in reduced fiber strength. Therefore, alkali treatment, i.e. mercerization process can be done to obtain better strength and brightness, and even higher absorption ability [1-4]. Another possibility is blending with synthetic fibers, i.e. polyester or polyamide for that purpose. Polyester fibers, on the other hand, have exceptional strength, resistance to chemicals, light and microorganisms, dimensional stability and fast drying, but due to high degree of crystallinity (65-85%) it has low moisture absorption and no swelling. As a result, the surface of polyester fibers can be changed using hydrolysis (alkali, enzyme), aminolysis, or plasma treatment, or combined with natural fibers such as cotton or wool [5, 6].

Since modification or treatment results in change of the number of surface-active groups of fiber, i.e. blocking and/or adding, their dissociation results in different thickness and distribution of the electric double, resulting in a change in fabric interface phenomena [1, 2, 7]. The change in the interface phenomenon between cotton and polyester fibers in monofilament fabrics has been studied in detail, but the dependence on the structure of the fabric as well as its alkaline modification in the blend was first investigated in the HRZZ project UIP-2017-05-8780 [8-12].

Today, there is an increased need for fabrics that, in addition to offering comfort, also have protective features, and given the recent COVID epidemic, antimicrobial effectiveness is particularly in demand. In addition to commercially available antimicrobial agents, natural active ingredients based on plant extracts, essential oils and animal origin are increasingly being employed in the functionalization of textiles [13].

Over the last decade, researchers' attention has shifted to the use of chitosan in textile processing, namely to offer an antibacterial impact and limit the release of textile particles into water during the washing process [10-12, 14]. Chitosan is a linear polysaccharide of natural origin obtained by partial alkaline N-deacetylation of chitin (over 50%). It consists of an acetylated part (N-acetyl-D-glucosamine) and a deacetylated part (beta-(1,4)-D-glucosamine) [15, 16]. Due to its antimicrobial activity, nontoxicity, biocompatibility, and biodegradability, it is a valued agent that is increasingly used in various fields. Its main applications are in medicine for antibacterial wound dressings, in drug delivery systems and to boost immune defenses [15-22]. Chitosan is most commonly used in the finishing of wool, cotton and polyester fibers and achieves antimicrobial activity against various bacteria and fungi [23-25]. The antimicrobial properties of chitosan result from its polycationic nature and depend on the degree of deacetylation, molecular weight and pH [26]. Positively charged amino groups of chitosan can bond to negatively charged bacterial surfaces, resulting in protein degradation and disruption of the cell membrane, increasing its permeability, and eventually leading to bacterial cell death. The higher the degree of deacetylation of chitosan, the greater its antimicrobial efficacy. The main disadvantage of chitosan as an antimicrobial agent for textiles is the dependence of its activity on temperature and the limitation of its pH activity to acidic conditions, as well as its weak adhesion to cellulose fibers. Water-soluble quaternized N-chitosan and carboxyalkylated chitosan derivatives are most commonly used as antimicrobial agents, showing antimicrobial activity in a wide pH range. Polycarboxylic acids and imidazolidinone derivatives are mainly used to form a solid bond between chitosan and cellulose fibers. In the presence of a crosslinking agent, the hydroxyl groups of chitosan and cellulose form covalent bonds with the carboxyl groups of the polycarboxylic acid, forming crosslinks between chitosan and cellulose, which significantly improves the resistance of textile materials treated in this way to care cycles [22, 27].

To improve the bonding of chitosan, for the purposes of this study a cotton-polyester blended fabric in twill weave was pretreated with 1.5 M NaOH and then functionalized with chitosan. Three wash cycles were performed to determine the durability. The zeta potential was measured with an electrokinetic analyzer EKA (Anton Paar) before and after functionalization, as well as after the first and third wash cycles, so that the presence of chitosan could be determined. The changes in mechanical properties - thread density, change in mass per unit area, tensile strength and elongation, spectral properties - whiteness, yellowing index and deviation from the white standard, moisture management and antimicrobial efficacy were determined according to standardized methods. Alkali pretreatment was found to increase the number of available hydroxyl groups of the cotton and carboxyl groups of the polyester component in the blend, and to increase the bonding of chitosan and thus wash resistance. Chitosan contributes to better strength and antimicrobial efficacy of the blend.

2. Material and Methods

The cotton/polyester blended fabric 50/50 supplied by Čateks d.o.o. (Čakovec, Croatia) was used. It is a 3/1 twill weave fabric with 38 ends per cm and 19 picks per cm with a mass per unit area of 160 g/m^2. The fabric was scoured and bleached under industrial conditions.

The fabric was pre-treated with 1.5 M NaOH with addition of 4 g/l surfactant Subitol MLF (CHT-Bezema) by exhaustion method at 80 °C for 20 min in the drum of Turbomat P4502 (Mathis) at LR 1:10. Afterwards, the fabrics were rinsed in hot, warm and cold distilled water; neutralized with 1% HCl, then again rinsed in distilled water and air dried.

Chitosan functionalization was performed using the pad dry cure method on Benz Stenter. Fabric was impregnated in a bath of 3 g/l chitosan dissolved in acetic acid, then dried at 110 °C for 4 min and cured at 170 °C for 45 s. After treatment, the fabrics were washed in distilled water at 60 °C for 30 min to remove unbound chitosan.

Three washing cycles were performed according to ISO 6330:2021 *Textiles - Domestic washing and drying procedures for textile testing* in Polycolor, Mathis, at 60 °C, 40 min with the program Washtest 60 using 2 g/l of EMPA ECE reference detergent 98 without optical brightener (Testfabrics Inc.).

Fabric labels and treatments are listed in Table 1.

Table 1. Fabric labels and treatments

Label	Treatment
19M	Cotton/polyester blended fabric
L	Pretreatment with 1.5 M NaOH
K	Chitosan functionalized fabric
0	Washed with distilled water
19MK1	1 washing cycle
19MK3	3 washing cycles

Fabric characterization was accomplished using electrokinetic analysis with the Electrokinetic Analyzer EKA (Anton Paar) and the stamp cell [28]. By using the streaming current approach, the zeta potential (ZP) was determined as a function of the pH (2-9) of the electrolyte, 0.001 mol/l KCl. The Helmholz-Smoluchowski equation [7] was used to compute the zeta potential. The IEP (isoelectric point) was also calculated [7, 28].

The tensile properties were used to examine the changes in mechanical characteristics by determining maximum force and elongation at break according to ISO 13934-1:2013 *Textiles - Tensile properties of fabrics - Part 1: Determination of maximum force and elongation at maximum force using the strip method* using the TensoLab dynamometer (MESDAN-LAB). The fabric count was determined according to ASTM D3775-17e1 *Standard Test Method for End (Warp) and Pick (Filling) Count of Woven Fabrics,* and the mass per unit area according to ISO 3801:1977 *Textiles - Woven fabrics - Determination of mass per unit length and mass per unit area* by weighing on a digital balance ALJ 220-5DNM (KERN) with an accuracy of 0.0001 g.

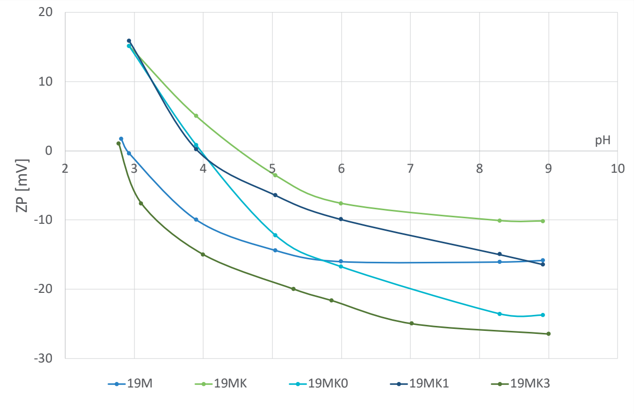
The spectral remission was evaluated using a remission spectrophotometer Spectraflash SF 300 (Datacolor). Degree of whiteness according to CIE (W_{CIE}) was calculated automatically according to ISO 105 J02:1997 *Textiles - Tests for colour fastness - Part J02: Instrumental assessment of relative whiteness* and Yellowing Index (YI) according to DIN 6167:1980-01 *Description of yellowness of near-white or near-colourless materials*. Tint deviation (TD) from the white standard and its coloristic meanings according to Griesser [29] were determined as well.

The moisture management properties were determined according to AATCC TM 195-2017 *Liquid Moisture Management Properties of Textile Fabrics* using the Moisture Management Tester MMT M290 (SDL Atlas).

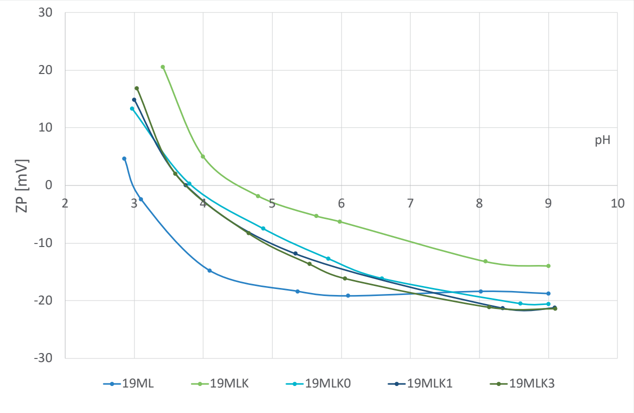
The antimicrobial activity was determined according to AATCC TM 147-2016 *Antibacterial Activity of Textile Materials: Parallel Streak*. Activity was determined to Gram-positive bacteria *Staphylococcus aureus* ATCC 6538 (*S. aureus*), Gram-negative bacteria *Escherichia coli* ATCC 8739 (*E. coli*), and microfungi *Candida albicans* ATCC 10 231 (*C. albicans*).

3. Results and Discussion

In this work, the influence of cotton/polyester blend fabric pretreatment on chitosan functionalization was investigated. The characterization of fabrics was performed by electrokinetic analysis through the results of ZP presented in Figure 1. From the results, it can be seen that the ZP of the cotton/polyester fabric (19M) at pH 9 is about -15 mV. The negative charge is due to the dissociation of the hydroxyl and carboxyl groups of the fibers. In cotton due to the dissociation of the hydroxyl groups and in polyester due to the carboxyl groups and its hydrophobic surface [7]. Cotton shows an IEP at pH lower than 2.5, whilst polyester has IEP at pH 4. Blending fibers in the fabric causes a change and the IEP is at pH 3. With alkaline hydrolysis (19ML), the ZP decreases to -18 mV because the swelling of the material is greater, and the IEP shifts to pH 3.5.



a.



b.

Fig. 1. Zeta potential (ZP) of cotton/polyester blended fabrics vs. pH of 0.001 M KCl, (a.) before and (b.) after pretreatment and chitosan functionalization up to three washing cycles

According to the literature [7], hydrophilic surfaces may adsorb water (hydration) and swelling, and so have a greater zeta potential. Cotton swelling [4] and the availability of active groups are both caused by alkali pretreatment. When polyester is hydrolyzed, the number of surface-active groups increases.

With the addition of chitosan, the surface of the fabric becomes more positive (ZP -10 mV), indicating a decrease in free active groups, and the positive charge of the chitosan comes to the fore (-NH_3 groups dissociate). The IEP rises to 4.5-5. This effect is particularly noticeable in materials that have been alkali pretreated/hydrolyzed, indicating greater chitosan bonding (19 MLK). Washing removes unbound chitosan, although in most cases, the ZP stays more positive after washing. This is supported by the IEP values, which have remained nearly constant.

The results from the measurement of the degree of whiteness (W_{CIE}) shown in Table 2 show that the treatment did not affect the whiteness of blended textiles. Fabrics are whiter after alkali pre-treatment, but chitosan functionalization remains the same. It is to emphasize that the white standard has no discernible difference in tint.

Tables 3 and 4 present the findings of mechanical parameters, such as breaking force and elongation, fabric count, and mass per unit area of cotton/polyester fabrics before and after alkali pre-treatment.

Table 2. Fabric whiteness (W_{CIE}), Yellowing Index (YI), Tint Deviation (TD) and its coloristic meaning

Fabric	W_{CIE}	YI	TD/ coloristic meaning
19M	79.9	0.14	No appreciable deviation in tint from the white standard
19MK	75.9	1.59	
19MK0	77.9	0.80	
19MK1	78.5	0.62	
19MK3	79.0	0.45	
19ML	86.8	-0.03	
19MLK	82.7	1.45	
19MLK0	81.3	1.68	
19MLK1	83.9	0.89	
19MLK3	83.1	1.09	

Table 3. Fabric count and mass per unit area

Fabric	warp [No/cm]	weft [No/cm]	m [g/m^2]
19M	38.3	17.8	157
19MK	38.8	18.3	161
19MK0	39.4	18.4	165
19MK1	37.8	18.3	163
19MK3	37.8	18.7	163
19ML	38.5	18.1	188
19MLK	39.2	18.3	192
19MLK0	39.8	18.7	196
19MLK1	39.8	18.4	193
19MLK3	39.6	18.2	192

The declared warp count was 38 ends/cm, and it did not alter after the alkali pre-treatment or chitosan functionalization. Slightly lower values were discovered for the claimed weft count of 19 picks/cm and mass per unit area. However, the fabric shrank, but the number of weft threads and mass per unit area rose. The cause of this is swelling of the cellulose component during wet processing. Because just the cellulose component of the blend swells, the increase is small, only 2-4%. It is also clear that chitosan treatment does not affect the mass of the fabric. During the first wash, there is more shrinkage and an increase in mass, confirming that the treatment is wash-resistant.

Table 4. Fabric count and mass per unit area

Fabric	F [N]	ΔF [%]	e [%]	Δε [%]
19M	1202	0.00	12.29	0.00
19MK	1204	0.17	14.70	19.59
19MK0	1278	6.32	15.45	25.69
19MK1	1241	3.24	16.60	35.05
19MK3	1283	6.74	17.05	38.70
19ML	1069	-11.06	14.69	19.52
19MLK	1151	-4.24	16.35	33.01
19MLK0	1195	-0.58	18.69	52.08
19MLK1	1156	-3.83	17.62	43.35
19MLK3	1152	-4.16	18.54	50.86

Polyester fibers are known to have excellent mechanical properties. According to the breaking force data, alkali treatment of polyester causes hydrolysis, and the tensile strength is reduced by 11% (19ML). The cotton component swells, whereas the polyester component shrinks due to alkaline hydrolysis.. The elasticity of the two components, and hence of the fabric itself, rises by 19% (19ML). The reason for this is regulated hydrolysis of polyester, cotton swelling, and shrinkage, all of which contribute to fabric strength retention.

Fabric exhibits improved strength and elongation following chitosan functionalization. The tensile strength increases because chitosan is coated in functionalization, which strengthens the fabric. It is also important to emphasize that with an increasing number of wash cycles, there is no loss of strength, but rather an increase in elongation.

Table 5 displays the findings of liquid moisture management qualities as mean values and coefficients of variation (CV) for each measured parameter for the Top surface (T) and Bottom surface (B). The results obtained are: Wetting Time (WT), Absorption rate (AR), Maximum wetted radius (MWR), Spreading speed (SS), Accumulative One-way Transport Capability (R) and Overall (liquid) Moisture Management Capability (OMMC).

Table 5. Moisture management properties of cotton/polyester fabrics before and after alkali pre-treatment, chitosan functionalization and washing

Fabric		**19M**		**19ML**	
Parameter		**Mean**	**CV**	**Mean**	**CV**
WT (s)	**T**	2.375	0.0994	2.25	0.0418
	B	2.5	0.0781	2.3437	0.0399
AR (%/s)	**T**	56.4877	0.0197	53.534	0.008
	B	62.0323	0.0087	55.833	0.0048
MWR (mm)	**T**	25	0	25	0
	B	25	0	26.667	0.1083
SS (mm/s)	**T**	6.7875	0.031	7.1172	0.0178
	B	6.7519	0.0291	7.1183	0.0429
R (%)		162.527	0.0986	115.42	0.0999
OMMC		0.6307	0.0295	0.5611	0.0229
Type		MMF		MMF	

Fabric		**19MK**		**19MLK**	
Parameter		**Mean**	**CV**	**Mean**	**CV**
WT (s)	**T**	14.7917	0.367	4.1663	0.1751
	B	8.6877	0.0856	4.193	0.0901
AR (%/s)	**T**	8.986	0.1362	24.6493	0.2524
	B	30.628	0.2314	38.9672	0.0999
MWR (mm)	**T**	15	0	20	0
	B	18.3333	0.1575	20	0
SS (mm/s)	**T**	0.8578	0.3295	3.4827	0.1448
	B	1.1997	0.2553	3.6911	0.1102
R (%)		613.373	0.0658	267.976	0.3033
OMMC		0.5776	0.0632	0.655	0.0793
Type		MMF		MMF	

Fabric		**19MK0**		**19MLK0**	
Parameter		**Mean**	**CV**	**Mean**	**CV**
WT (s)	**T**	9.7707	0.1662	3.0937	0.0302
	B	8.5157	0.1568	3.1877	0.0509
AR (%/s)	**T**	10.9331	0.1623	45.4939	0.0264
	B	40.9454	0.1321	49.553	0.0463
MWR (mm)	**T**	11.6667	0.2474	21.6667	0.1332
	B	20	0	20	0
SS (mm/s)	**T**	1.1233	0.1184	5.4066	0.0832
	B	2.0801	0.0765	4.9708	0.0244
R (%)		506.918	0.1134	161.667	0.0625
OMMC		0.676	0.0418	0.5951	0.0155
Type		MMF		MMF	

Fabric		19MK1		19MLK1	
Parameter		Mean	CV	Mean	CV
WT (s)	T	2.6567	0.0539	2.6663	0.0914
	B	3.0003	0.0312	2.854	0.0525
AR (%/s)	T	53.6087	0.006	51.502	0.0087
	B	57.8656	0.0255	54.2083	0.0338
MWR (mm)	T	20	0	25	0
	B	25	0	23.3333	0.1237
SS (mm/s)	T	5.9568	0.0162	6.3929	0.043
	B	6.1673	0.0481	6.0904	0.1402
R (%)		151.316	0.1717	119.379	0.3465
OMMC		0.6066	0.0536	0.561	0.0909
Type		MMF		MMF	

Fabric		19MK3		19MLK3	
Parameter		Mean	CV	Mean	CV
WT (s)	T	2.474	0.0403	2.9067	0.1116
	B	2.63	0.0033	3.1563	0.0684
AR (%/s)	T	54.5092	0.0131	45.9961	0.043
	B	58.1552	0.0009	51.0711	0.0218
MWR (mm)	T	23.3333	0.1237	20	0
	B	25	0	21.6667	0.1332
SS (mm/s)	T	6.8659	0.0509	5.3753	0.0743
	B	6.8977	0.0137	5.5287	0.1458
R (%)		175.328	0.0966	124.4	0.1131
OMMC		0.6341	0.0295	0.5579	0.0301
Type		MMF		MMF	

**Variation coefficient (CV); Wetting Time (WT); Absorption rate (AR); Maximum wetted radius (MWR); Spreading speed SS for top (T) and bottom (B) surface; Accumulative One-way Transport Capability (R); Overall (liquid) Moisture Management Capability (OMMC)*

The wetting time measured at the MMT is the time it takes for the top and bottom surfaces of the cloth to start wetting [31, 32]. Table 5 shows that the untreated cotton/polyester blend fabric (19M) has an extremely short wetting time WT<2.3 and a large wetting radius. In this blend these two components combine the best of both: the good absorbency of cotton and the high capillarity of the hydrophobic surface of polyester. In the case of fabrics treated with alkali, the wetting time is < 2.5 s. This is due to the change in the surface and the better absorption of the polyester components, so that a small amount of water bonds to the new surface groups of the polyester fibers. Measurements confirm the hydrophilicity of all cotton/polyester samples: the wetting time is very fast and the maximum wetting radius MWR is 25 mm. For this reason, the spreading rate, which represents the accumulated rate of surface wetting from the center of the sample where the discharge solution descends to the largest wetted radius, is somewhat lower. The accumulative one-way transport capability (R) represents the difference between the areas of the upper and lower curves of the moisture content of the surface of the sample as a function of time. All fabrics benefit from efficient transport.

Total (Liquid) Moisture Management Capability (OMMC) is estimated by adding three measurable qualities together: Liquid absorption rate at the bottom, one-way liquid transport capability, and maximum moisture spreading rate at the bottom. This is a measure of the fabric's ability to carry liquid moisture. OMMC is present in all fabrics and is a great indicator of moisture management fabric and does not change with alkali treatment.

The addition of chitosan changes the properties. It is evident that the wetting time increases following chitosan treatment. Additionally, there is a distinction between untreated and alkali-pretreated samples. The MWR value decreases for non-alkali pre-treated samples while remaining nearly unchanged for alkali pre-treated samples. Regardless of these differences, excellent transport is achieved, and all fabrics are of the "Moisture Management Fabric" (MMF) type. The fabric type is also unaffected by the wash cycles.

Since the presence of chitosan was also detected after 3 washing cycles, the antimicrobial efficacy was evaluated according to AATCC TM 147-2016 for *S. aureus*, *E. coli,* and *C. albicans*. The activity as example is shown in Fig. 2 and the results are listed in Table 6.

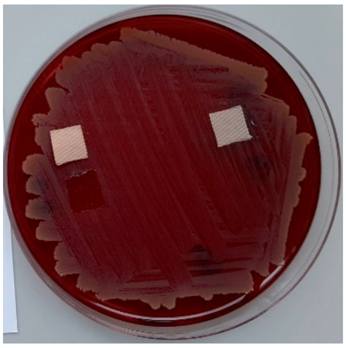


Figure 2. The antimicrobial activity – an example

It is clear from the results in Table 6 that both nonfunctionalized fabrics (19M, 19ML) showed no activity against Gram-positive bacteria *Staphylococcus aureus*, Gram-negative bacteria *Escherichia coli*, and microfungi *Candida albicans*. It is also clear that chitosan functionalization resulted in high antimicrobial activity. Although there is no the zone of inhibition because chitosan is not leaching from fabric, there are no bacterial colonies directly under the sample in the contact area, indicating that the fabrics have antimicrobial activity (Fig. 2).

Table 6. The antimicrobial activity of fabrics before and after functionalization with chitosan and 3 washing cycles

Fabric	*S. aureus*	*E. coli*	*C. albicans*
19M	-	-	-
19MK	+/-	+/-	+/-
19MK0	+/-	+/-	+/-
19MK1	+/-	+/-	+/-
19MK3	+/-	+/-	+/-
19ML	-	-	-
19MLK	+/-	+/-	+/-
19MLK0	+/-	+/-	+/-
19MLK1	+/-	+/-	+/-
19MLK3	+/-	+/-	+/-

\+ *antimicrobial activity (zone of inhibition can be observed);*
+/− *antimicrobial activity (no colonies beneath);*
− *no antimicrobial activity*

The antimicrobial activity of chitosan is attributed to its polycationic nature. It most likely interacts with the anionic membrane of the microorganism, resulting in a change in permeability that causes cell death by leakage of intracellular plasma. Therefore, the electropositive character of chitosan plays an important role in antimicrobial activity. It should be emphasized that the antimicrobial activity obtained remains after 3 washing cycles, indicating a sufficient amount of chitosan in the fabric structure.

4. Conclusions

In this work, the influence of cotton/polyester blend fabric pretreatment on chitosan functionalization was investigated. For this purpose, a cotton/polyester blend fabric in twill weave was pretreated with 1.5 M NaOH and then functionalized with chitosan. Three wash cycles were performed to determine the durability.

Alkali pretreatment was found to increase the number of available hydroxyl groups of the cotton and carboxyl groups of the polyester component in the blend, and to increase the bonding of chitosan and thus wash resistance. Chitosan contributes to higher strength and antimicrobial efficacy of the blend. The results confirm hydrophilicity of cotton/polyester fabrics and fabric type is “Moisture Management Fabric”.

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