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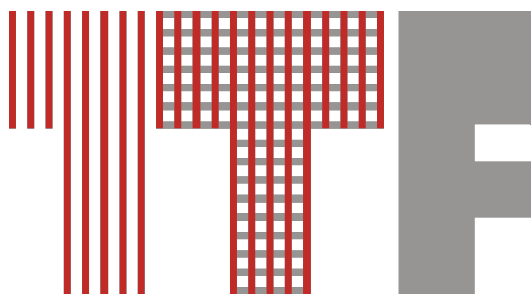
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Cumulative effects of washing on properties of cotton fabrics and their blends

KEYWORDS: washing, effects, cotton, polyester, surface modification

Abstract Apart from removing soils and preventing redeposition, chemical and mechanical activities during washing also change the structure of the textile treated. The intensity of these changes is dependent on the initial level of quality of the textiles treated, as well as upon the processing conditions (temperature, washing agent, mechanical parameters, number of washing cycles, etc). The aim of this paper was study cumulative effects of washing on properties of cotton fabrics and their blends through RAL-GZ 992 quality criteria evaluated through ash content, whiteness, tint deviation, mechanical and chemical damage. Electrokinetic potential, pilling resistance and morphology of cotton and their blends were selected as evaluation criteria for surface characterization of cotton and their blends after multiple washing cycles.

INTRODUCTION

Washing process is regulated by chemistry, mechanical agitation, temperature and time. The impact of particular factors can be represented by a washing cycle, within which is the circle dealing with water, which connects the factors in the process. Synergic effect of particular components incorporated into detergent determines its chemical activity. One of the primary requirements for a successful detergent formulation is to remove various soils and preserve an initial characteristic of textile materials as long as possible. Surfactants play a dual role in the soil removal: overcome the attraction between soil and fabric by attaching themselves to both, loosen the soil and deflocculates it at the same time, i.e. they break it up into colloidal particles and stabilize their aqueous dispersion. It is enhanced by presence of anionic and non-ionic surfactants in the washing bath. The development of new detergent formulations is targeted to low temperature washing based mostly, on the high efficient and environmental friendly components (1-5). Soil removal during washing process is also enhanced by mechanical input, proper wash time and temperature (6). Synergy of all mentioned factors impact on changes of textiles occurred in washing. The quality of water is a key parameter, thus in some cases the cumulative effect of frequent washing can be high inorganic and organic matters content on the textiles. The accumulation of deposits can cause negative effects, e.g. harsh and stiff hand, progress in degradation and tearing, as well as reduced usability of the textiles. Textiles subjected to the mostly aggressive chemicals,

mechanical agitation superimposed by high temperature and duration during washing is capable to cause damage after frequent washing. It is known that washing affects generally more to fabric damage than the usage or wear (7). Evaluating of textiles durability in washing and wear is of special interest to the laundries which, apart from washing, offer the service of renting garments. The control of secondary effects, accomplished through the usage of a reference cotton cloth cannot fulfil their expectations completely. The economic benefit, appearance and functionality during maintenance and wear are required, so quality of textiles is of the prime importance. The German Research Institute Hohenstein is authorized to accredit laundries with the quality label for proper textile care RAL-GZ 992 recognized by the German Institute for Quality Assurance named RAL. This Certificate of accreditation is awarded to laundries that reach certain criteria for determining the laundering quality (secondary laundering effects) based on standard methods. RAL Quality Mark 992 (RAL-GZ 992) subscribes testing of breaking strength, chemical wear, whiteness degree and incineration residue (8,9). The paper deals with supplement methods applied in order to provide the objective assessment of the cotton fabrics and their blends.

The frequent washing cycles can cause the fibre surface modification as result of fibre swelling capacity in the alkaline detergent bath superimposed by mechanical agitation. Cellulose textiles are exposed to a swelling in an alkali medium, which brings changes in pore structure, tendency to fibrillation and surface changes. Fibres surface charge is important parameter in the wet processing of cotton. The

electrokinetic properties of the fibre are possible to characterize with a double layer model created at the interface fibre/electrolyte solution. Electrokinetic double layer is characterized by zeta potential which depends on the nature of fibre functional groups, type and the number of the dissociating groups, hydrophylicity, as well as ion and water sorption from the solution according (10). Most textile fibres possess negative charge in neutral aqueous solutions (11). The zeta potential on a material's surface in contact with a polar medium, is governed by the dissociation of surface groups, the preferential adsorption of cations or anions, the adsorption of polyelectrolytes and surfactants, the isomorphic substitution of cations and anions, and the accumulation or depletion of electrons (12). It provides insight into the charge and adsorption characteristics of solid surfaces. The zeta potential is an experimentally-accessible parameter that can be determined using several types of electrokinetic phenomena: electro-osmosis, streaming potential, electrophoresis, and sedimentation potential (13). Streaming potential/ current is the most convenient for the characterization of textile materials. Zeta potential is highly valuable when the washing bath contains special detergent ingredients such as antiredeposition agents or enzyme cellulase (14). Antiredeposition agents have a particular orientation towards cellulose, in several layers, which increases the number and the density of negatively charged carboxylic groups, resulting in a negative charge. Enzyme cellulase significantly impacts the topography of the fabric surface (15).

The investigations presented here have supplemented and broadened the RAL-GZ 992 (quality system) requirements by introducing indicative testing methods, evaluating susceptibility to pilling, breaking force and tearing work, as well as surface characterization of washed textiles by pilling resistance, electrokinetic potential and SEM images.

EXPERIMENTAL

Material

Technical characteristics of two types of cotton and cotton/ polyester blend are presented in Table 1.

Composition	Label	w (percent)	Q (g/m ²)	Density (w/w)	Weave
Cotton	CO 1	100	185	44/24	Twill 1/3
Cotton	CO 2	100	198	46/23	Cloth
PES/cotton	CO/PES 1	55/45	177	56/27	Twill 1/3
PES/cotton	CO/PES 2	35/65	173	47/24	Twill 1/3

Table 1. Technical characteristics of the tested fabrics.

Procedures

Selected fabrics together with 80 kg heavily soiled linen were exposed to 25 washing cycles in a batch washer following the programme presented in Table 2.

Washed fabrics were not dried between repetition of washing cycles.

Methods

The breaking strength of the cotton fabrics was measured by the strip method in the direction of the warp (EN ISO 13934-1,

Programme	t (min)	BR	T (°C)	Agent	(g/kg)
Soaking	5	1.4	15-35	a	2.00
1 st Prewashing	6		35	b	5.00
2 nd Prewashing	12		60	c	25.00
Draining	1				
Soaking	1	1.4	15		
Prebleaching	16	1.4	60	d	13.00
Draining	1				
Washing	10	1.4	73	e	6.60
Cooling down	5		73 - 40	f	9.00
Draining	1				
1 st rinsing	3	1.6	40		
Draining	1				
2 nd rinsing	3	1.6	15		
Draining	1				
3 rd rinsing	3	1.6	15	g	3.00
Centrifuging	4				
Water hardness= 139.4 ppm					

Table 2. Washing programme. ^a Nonionic surfactant (15-30 percent); enzymes (< 5 percent); solubilisers; stabilisers. ^b Nonionic surfactant (>30 percent), FWA, solubilisers. ^c surfactants-anionic, nonionic, soap (> 5 percent); sequestering agent (>30 percent); enzymes. ^d NaOCl (10-25 percent). ^e Nonionic surfactant (15-30 percent); sequestering agent (>30 percent); phosphonates, silicates; carbonates, foam inhibitors, decomposition inhibitors. ^f oxygen based bleach (>30 percent), peracetic acid (5-15 percent); phosphonates (< 5 percent). ^g Acetic acid (80 percent).

1999) using a Tensolab 3000 dynamometer, Mesdan s.p.a. The decrease in breaking strength (DF) was calculated as a percentage of the difference between fabric washed after 3 and 25 cycles. The Schulz-Blanschke equation was used to determine the limiting viscosity value (η) and the Eisenhut equation for the calculation of chemical wear (s). The limiting viscosity value of each cotton washed fabric (η) was determined by the viscometric method (DIN 54270-2, 1977), measured with, Capillary Ubbelohde viscometer, type ASTM 1c. The incineration residue (A) was determined according to the standard method (ISO 4312). Cotton fabric was incinerated in a muffle furnace for 2 h at 800°C [9]. The whiteness quality was determined by measuring under D-65 light (Spectroflash SF 600 Plus, Datacolor, Lucerne) and calculating lightness (Y), CIE degree of whiteness (W_{CIE}) and CIE tint value TV_{TD} .

Impact of multiple washing cycles on cotton fabrics and their blends was monitored through tear work and surface characterization through pilling resistance, zeta potential and morphology. The tear work (W) is determined using the Elmendorf apparatus, according EN ISO 13937-1, while breaking force (Fp) was calculated. The method of testing according to the ISO 12945-2 standard offers an estimation of susceptibility to pilling, since loads of 1000, 3000 and 5000 cycles simulate end-use quite well. The testing is done on a

Martindale tester, a device for testing fabric susceptibility to pilling and wear.

The zeta potential was determined by the streaming potential method using the EKA, Electrokinetic Analyzer (Anton Paar, GmbH, Austria) with rectangular cell suitable for the textile fabrics. The zeta potential of the unwashed and washed fabrics is measured in 1 mmole/l KCl in the pH range from 10 to

2. Detailed mounting procedure is described in the previous paper. In this case, determining an apparent zeta potential follows the approximation of the Helmholtz-Smoluchowski equation (16-19).

Surface characterization by scanning electron micrographs (SEM) was selected as a useful technique to analyse topography and changes of textiles in washing. The scanning electron microscope, Tescan SEM VEGA 5136 MM was used for surface examination of the samples after 3 and 25 washing cycles. The samples were fixed on the sample holder and

sputter-coated with a palladium-gold alloy. The samples were examined using 2 types of signals produced by SEM including secondary electrons (SE) with acceleration voltage of 20.0 kV and magnification 3.5 kx.

RESULTS AND DISCUSSION

Cotton and their blends were batch washed in a two-chamber machine, with the load of heavy soiled workwear. Chemicals were dozed at maximum level, since most sturdy soils were supposed to be removed in a washing. Results of tested criteria are presented in Table 3.

Label	WC	ΔF (percent)	s	A (percent)	W _{CIE}	TV	TD	Y	W (cN×cm)		F (cN)	
									w	f	w	f
CO 1	3	-	-	0.17	132.9	0.6	G1	89.0	2033.3	2333.3	236.4	271.3
	25	3.6	1.0	0.17	135.5	1.1	G1	90.1	1833.3	2133.3	213.1	246.0
CO 2	3	-	-	0.28	128.9	0.2		88.4	3233.3	3233.3	321.7	375.9
	25	7.5	1.2	0.06	132.0	1.1	G1	89.4	3100.0	3100.0	290.7	360.5
CO/PES 1	3	-			132.7	0.1		84.9	1566.6	1433.3	182.1	166.6
	25	5.1	n/a	n/a	131.2	0.3		85.4	1166.7	1333.3	135.6	155.0
CO/PES 2	3	-			128.8	0.8	G1	84.0	2233.3	1733.3	256.7	201.5
	25	0.4	n/a	n/a	129.5	0.7	G1	84.2	2133.3	1633.3	245.0	187.9

Table 3. Properties of cotton and their blends after 3 and 25 washing cycles (WC).

The analysis of the results in Table 3 showed that the incineration residue (A) on all the tested samples was below the allowed value of 0.7 percent, meaning water was properly prepared and washing agents were selected so as to take those that prevented deposition of inorganic components onto the fabric during a process. Decrease in breaking strength of the tested fabrics after 25 washing cycles never exceeded the allowed value of 15 percent. It was obvious that fabric construction properties influenced the intensity of the changes that occurred in washing, i.e. twill weave fabrics were more prone to changes. The fabrics made of CO/PES blend, and especially the CO/PES 2 fabric, exhibited lower decrease in breaking strength than cotton fabrics. It was to be expected, since the blends are less susceptible to swelling, which makes them less exposed to the influence of washing bath components. The chemical wear (s) occurred on cotton fabrics (CO) in washing was high, and the value exceeded the allowed value of 0.5. However, these values were not related to breaking strength decrease. Higher level of damage is usually attributed to the presence of heavy metal ions, as catalysts in hydrogen peroxide decomposition. However, having in mind the preparation of processing water and the content of sequestering agents, the damage could be attributed to the sensitivity of cotton to higher concentration of sodium hypochlorite. This bleach, in high concentrations and at the temperature of 60 °C, exhibits increased chemical potential and can cause damages in washing of cellulose materials. The chemical wear was not determined for the fabrics made of the CO/PES blend, since the method is not appropriate for blends. Whiteness degree (W_{CIE}), tint value and deviation (TV, TD) and whiteness with no UV stimulation (Y) were expressed as CIE. The values obtained indicate that whiteness quality in washing was not sufficient (7). Since whiteness degree after 25 cycles increased for only a couple of units, as compared to the textiles laundered in 3 cycles, redeposition did not occur. To achieve a higher whiteness degree it would be necessary to increase the concentration of fluorescent

whitening agent. Sodium hypochlorite reduces the effect of optical brightening, meaning inadequate increase in whiteness could be attributed also to the impact of this chemical. Basic whiteness (Y) of all the fabrics tested met the required criteria. Tint value for all the fabrics washed also met the required standards, although a slight shift of the tint deviation towards greenish was registered with some fabrics (G1).

The criteria for cumulative washing effects according to RAL were supplemented and broadened for the purpose of testing workwear made of CO/PES blend. Breaking force and tearing work, judged to be important, as they show how much work and how high a force was necessary to continue with tearing damaged fabric. The values are particularly important for table linen, where mechanical damages are often encountered by sharp items. The tear work and breaking force for the unwashed CO 1 fabric were considerably different warpwise from weftwise, due to impact of higher density of warp yarns. Washing of workwear resulted in fabric shrinkage,

both in warp and weft, thus reducing the difference of these values after 3 and 25 washing cycles. Reduced work and breaking force indicated damages that occurred on the CO 1 fabric in washing.

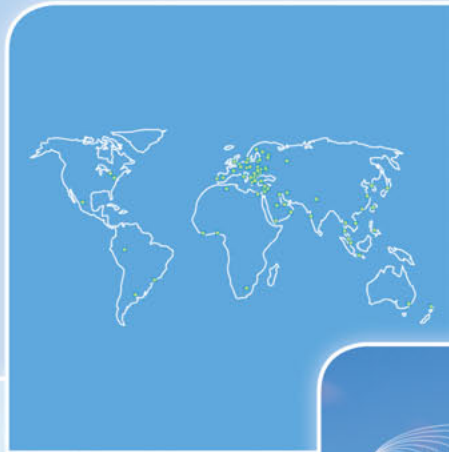
The results indicated slight differences in breaking force and tearing work warpwise and weftwise for the unwashed CO 2 fabric, as compared to the CO 1 fabric. Washing reduced a breaking force and tearing work of the CO 2 fabric, reduction being more prominent in the warp direction. Fabric characteristics had an impact on the changes of these properties.

Breaking force and tearing work after 3 and 25 washing cycles were considerably lower for the CO/PES 1 fabric than for the previously analysed fabrics CO 1 and CO 2. This reduction was attributed to the conditions of washing (temperature and the amount of washing agents), adapted to the cotton fabrics. The sensitivity of the polyester component in the blend, when rinsing (cold water) after laundering at elevated temperature (73°C), should be noted. The presence of the polyester component in the blend is reasonable for a different washing procedure, at lower temperatures, with a cool down system (gradual cooling), which resulted in more favourable washing effects.

Breaking force and tearing work for the CO/PES 1 fabric in the direction of the warp differed from that in the direction of the weft. The changes were more prominent in the direction of the warp, the highest being caused by the first washing cycles, while additional washing cycles caused only minor further changes. The results of measuring tear work and breaking force indicated that these values were reduced after 25 cycles, as compared to 3 cycles. The highest value was recorded for the CO 2 fabric, at the same time the only fabric where tearing work weftwise was higher than warpwise. High tear work was not correlated with breaking strength decrease (DFp=7.5 percent) measured only warpwise. High values of tear work after 25 cycles could be attributed to shrinkage in washing.

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Surface characterization

Pilling resistance

Susceptibility to pilling was also tested. The fabrics were multiple washed and were not worn between cycles. It was thus interesting to test tendency to pilling evaluated according to the etalons SM50-twill background from 1

to 5, using various types of surface provocation. The appearance of the fabric CO 1 made in twill weave (3/1) after repeating washings indicated pilling tendency even on the surface of unwashed CO 1 fabric. The appearance of the fabric indicated and warned of the danger of creating pilling in further washing cycles. It was especially prominent after 3000 and 5000 turns. The fabric washed in 25 washing cycles, after 5000 turns, exhibited prominent pilling, evaluated by the grade 2, Table 4.

The evaluation of proneness to pilling for the washed CO 2 fabric after 1000, 3000 and 5000 cycles is evidently less pronounced on the surface of the CO 2 fabric than was the case with the CO 1 fabric. Proneness to pilling of this twill-weave fabric could be due to construction properties of the yarns used and of the fabric itself. A disadvantage of fibre blends can be different friction effects between cotton and polyester fibres that can lead to creation of surface pills. Despite the presence of the polyester component in the blend, the CO/PES 1 fabric exhibited lower proneness to pilling than cotton fabrics previously tested – CO 2 and CO 1, and the CO/PES 1 fabric was attributed high grade of surface pilling resistance. The CO/PES 2 fabric also exhibited proneness to pilling, especially after 25 washing cycles, provoked by 5000 cycles, which resulted in the fabric surface grade of 2. The unwashed CO/PES 2 fabric also exhibited proneness to pilling due to increased polyester component

Fabric	Washing cycles	1000 cycles	3000 cycles	5000cycles
		Grade		
CO 1	3	4	3	3
	25	4	3	2
CO 2	3	4	3	3
	25	4	3	3
CO/PES 1	3	5	4	4
	25	5	4	4
CO/PES 2	3	4	4	3
	25	4	3	2

Table 4. Assessment of pilling tendency of tested fabrics after 3 and 25 washing cycles.

in the blend when compared with PES/CO 1 blend fabric. The results requires for further analysis of the yarn, since its level of twist could be the cause of higher proneness to pilling. Tendency to create pilling on the surface after provocation was most prominent with the cotton

fabric CO 1 and their blend CO/PES 2. The lowest tendency of the sort was exhibited by the CO/PES 1 fabric, the one with the highest density.

Scanning electron microscopy

The intensity of damage in washing depends on different parameters as follows: textiles characteristics (construction parameters, polymerization degree of textile fibres, swelling capacity) as well as washing conditions (chemicals, temperature, time, mechanical agitation). The changes of textiles in washing can be

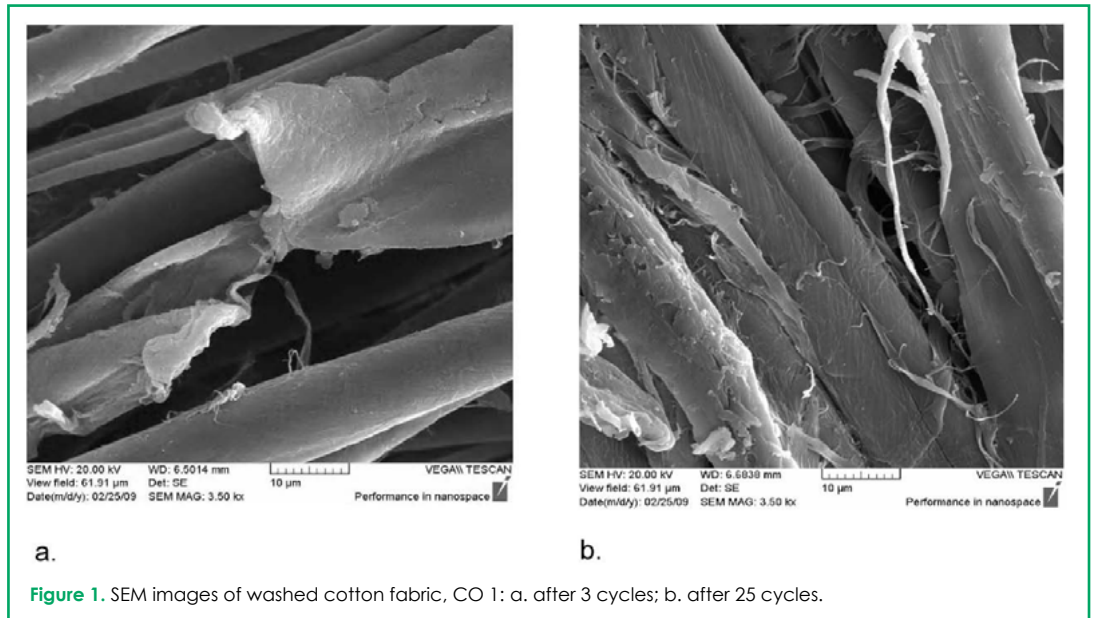


Figure 1. SEM images of washed cotton fabric, CO 1: a. after 3 cycles; b. after 25 cycles.

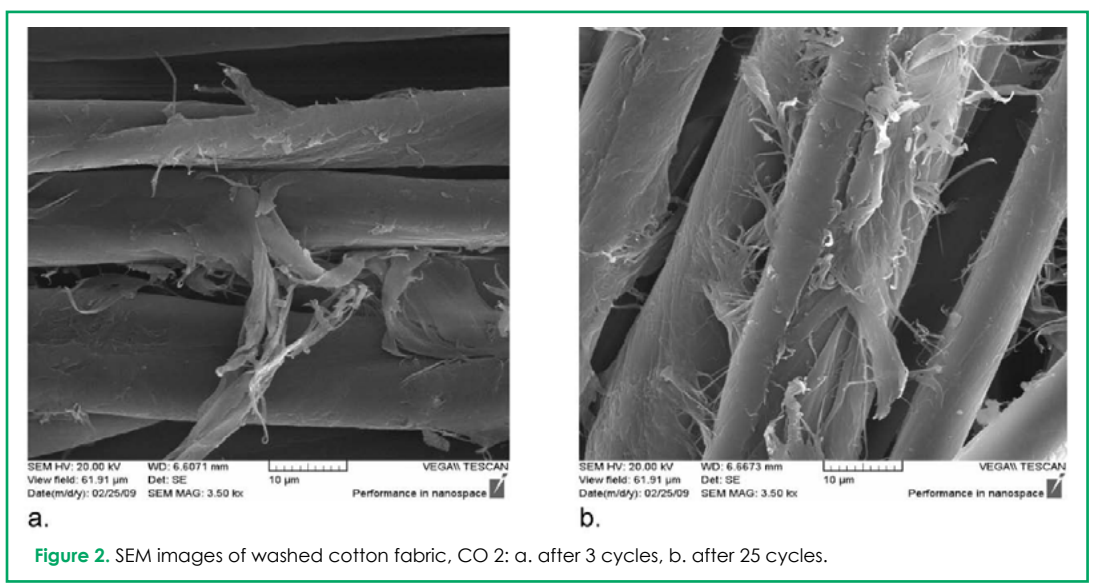


Figure 2. SEM images of washed cotton fabric, CO 2: a. after 3 cycles, b. after 25 cycles.

analyzed by microscopic methods as well, one of the best known being the SEM. The images of the fabric before washing indicate an integrated structure and recognizable appearance of the cotton fibres. Washing caused changes in cotton, due to its high swelling in the alkali media, the impact of bleaching agents and mechanical influences. The damaged fabric is much more prone to soiling than the undamaged one. In general SEM images of tested fabrics show a signs of damage in the case of cotton fabrics almost after 3 washing cycles. A serious fibrillation is present after 25 cycles at levels which may effect changes in appearance as well as mechanical properties of fabrics to be washed (Figures 1-4).

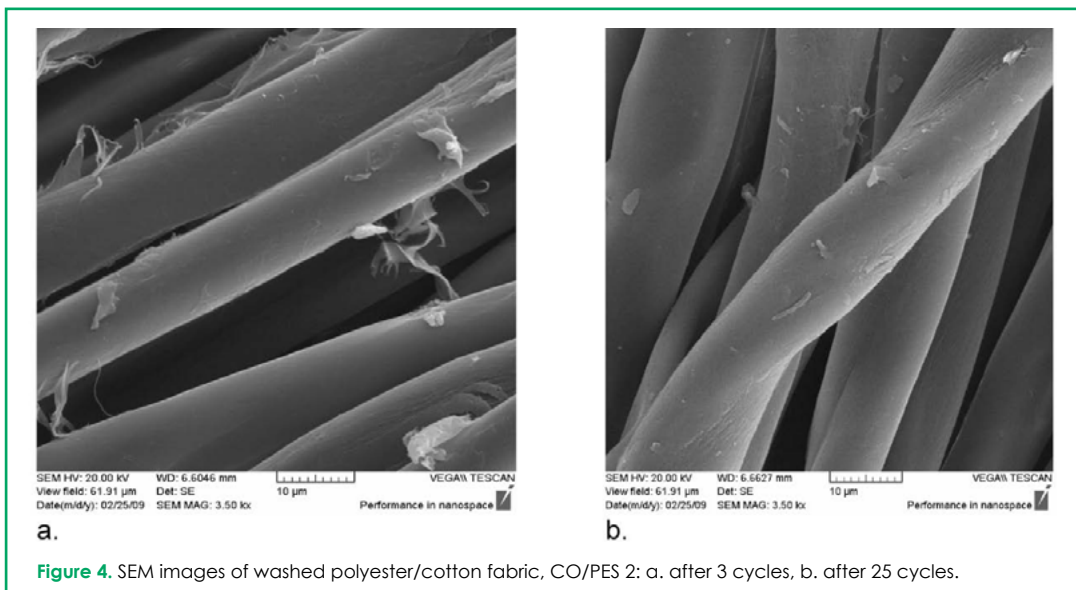
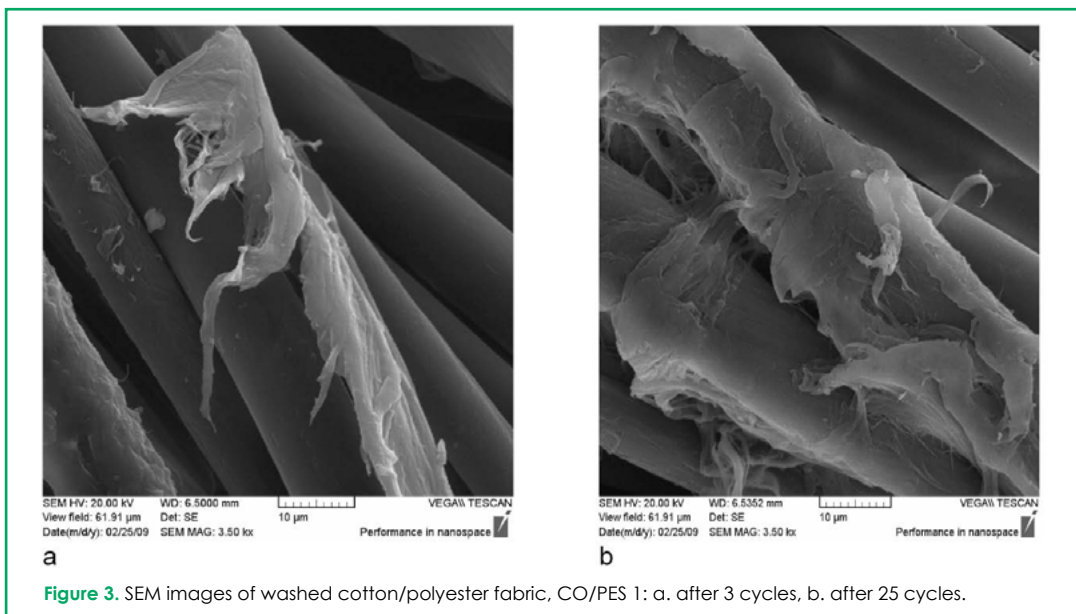
Polyester fibres are highly crystalline, hydrophobic and negligible swollen in water. Due to less swelling capacity of cotton / polyester blends there is negligible change of the fibre surface after repeat washing cycles when compared to cotton fabrics. This specific behaviour is confirmed by micrographs images, Figures 3-4.

Electrokinetic potential

The intensity of textile surface changes in washing depends upon a number of parameters. The changes are less prominent if laundering conditions are optimised. Sensitivity of textiles is the result of their high swelling capacity in alkali and bleaching agents, further increased by mechanical agitation in washing. Surface changes can be characterised in a number of ways, one of them being measuring surface charge, characterized by zeta potential. It depends upon construction properties and the degree of hydrophilicity. Fibres characterised by high hydrophilicity exhibit lower surface charge than hydrophobic ones. Accordingly, the results of measuring zeta potential for the tested fabrics depend upon pH value of the electrolyte solution were presented in Figures 5 a-d.

The curves show differences in zeta potential of the tested fabrics after 3 and 25 washing cycles. The cotton fabric CO 1 had lower negative zeta potential after 25 cycles than the same fabric after 3 cycles, although the

differences of zeta potential in the alkali region were minor. The relation could be attributed to shrinkage and more closed structure of the fabric after repeated washing. The dependence of the CO 2 fabric zeta potential after 3 and 25 cycles on pH can be seen in Figure 5 b. A difference values indicate surface modifications caused by the alkali medium and the aggressive washing conditions, although this could not be avoided since heavy stains from the workwear had to be removed. The presence of the polyester component blended with cotton resulted in higher zeta potential in the entire pH range, Figure 5 c & d, as compared to cotton fabrics, Figure 5 a & b. Higher content of polyester (65 percent) than cotton (35 percent) in the blend resulted in higher negative zeta potential of the CO/PES 2 fabric (Figure 5 d), as compared to the CO/PES 1 fabric



(Figure 5 c) after 3 cycles. However, impact of multiple washing cycles on surface changes of blends was minor in relation to the cotton fabrics.

CONCLUSIONS

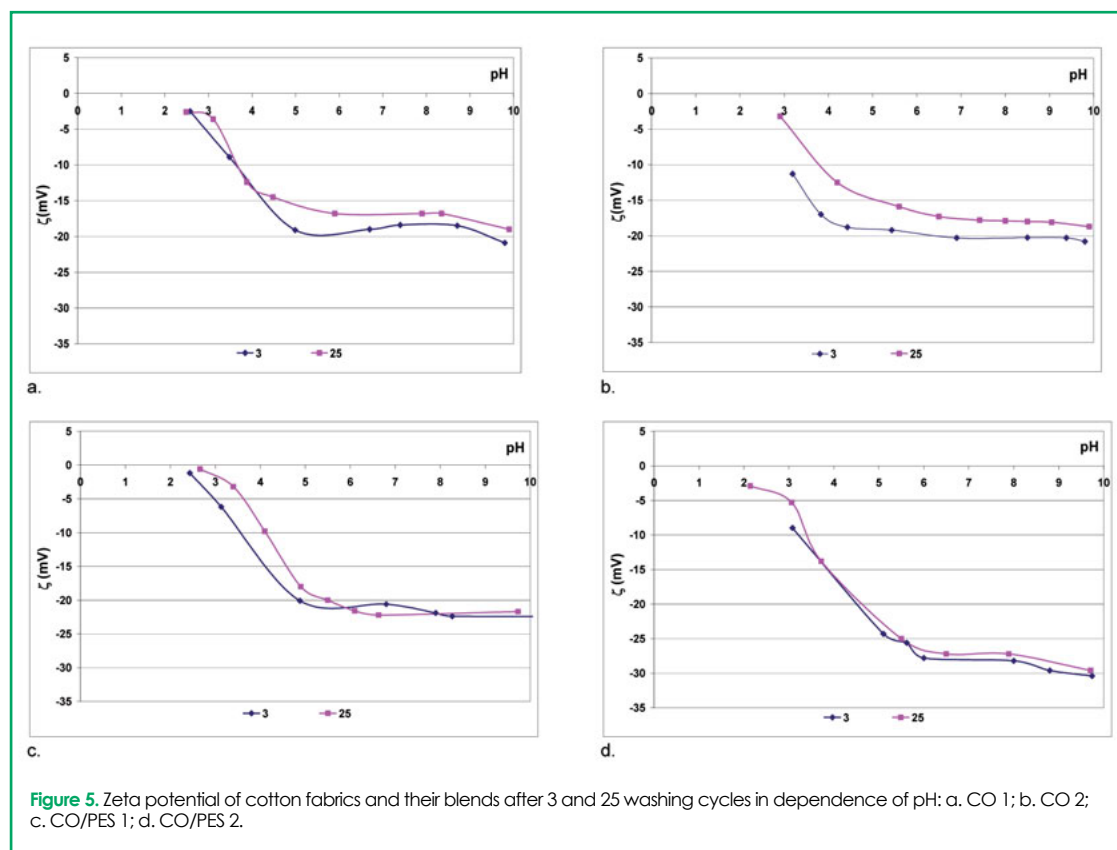
Cumulative effects of washing were analyzed on cotton fabrics and blend of cotton with polyester fibres. The impact of multiple washing was performed through criteria usual for evaluation of cotton textiles including incineration residue, whiteness degree, mechanical and chemical wear. Most quality systems prescribe no criteria for cotton/polyester blends. Therefore, it was interesting to study convenience of some other control criteria including breaking force, tearing work, pilling resistance, electrokinetic potential as well as morphology by SEM characterization. If cumulative effects are tested on washed fabric (with no reference fabric), the relevant mechanical damage indicators of the PES/cotton blend fabrics after 25 washing cycles under highly alkaline conditions at elevated temperature, can be tearing work and breaking force, as they point at the sensitivity of the polyester component in the blend. Cumulative washing impacts on surface charge of cotton and their blends with polyester as well as their proneness to pilling. Surface changes of textiles characterized by zeta potential being dependent on pH. Change in magnitude of zeta potential of cotton fabrics was reduced after cumulative washing. The impact of multiple washing cycles on change in surface charge of blends is negligible. Specific tendency to fibrillation of cellulose materials was characterized by SEM micrographs. SEM images of cotton/polyester blends indicated no surface modification due to their less swelling capacity when compared to cotton fabrics.

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