## **Evaluation of Cellulosic Fabrics Surface Characteristics** after Different Treatments

# Vitalija MASTEIKAITĖ<sup>1\*</sup>, Ulzhan SMAILOVA<sup>2</sup>, Maira NURZHASAROVA<sup>2</sup>, Virginija SACEVIČIENĖ<sup>1</sup>, Tadas KLEVECKAS<sup>1</sup>

<sup>1</sup> Department of Clothing and Polymer Products Technology, Kaunas University of Technology, Studentų St. 56, LT-51424 Kaunas, Lithuania

<sup>2</sup> Department of Design, Almaty Technological University, Tole bi 100 Almaty, 050012, Republic of Kazakhstan

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Various clothes treatments during their manufacture and wear change the fabrics physical and surface characteristics. The different testing methods for fabrics quality evaluation are used in this research. The aim of this work is to examine the effect of enzyme treatment, laundering and abrasion on the appearance of different cellulosic fabrics. Six cotton and cotton blend woven fabrics used for faded garments production were chosen for this examination. Treatments such, as enzyme wash, domestic laundering, abrasion using IT-2 and Martindale (Mesdan) instruments and rubbing were applied in order to evaluate the worn look of the different fabrics. The degree of fabrics distortion was determined by using abrasion resistance and weight change characteristics. Also, the colour fastness and staining after different dry and wet treatments were analysed. The results of this research show that the surface characteristics fabrics have changed their difference in colour not only after the enzyme wash but also after successive abrading and cyclical domestic launderings. Both colour fading of the fabrics and staining of cotton white fabric may appear after rubbing in dry and especially in wet conditions. The received results show that during tested fabrics laundering, the cotton and nylon parts of multifibre fabric were stained more heavily.

Keywords: cellulosic fabrics, enzyme wash, domestic laundering, abrasion, rubbing, colour fastness, staining.

### **1. INTRODUCTION**

A fabric's appearance is one of the most important aspects of its quality. During wear the fabric and item of clothing is made from may be subject to a wide range of agencies. These can affect the colour in different ways. Poor colour fastness is a major source of consumer complaint. Therefore colour fastness is an important aesthetic consideration in choice of clothing. The fastness of a colour can vary with the type of dye, the particular shade used, the depth of shade, and how well the dyeing process was conducted [1]. At the same time, various additional chemical and mechanical treatments of ready-made garments allow the creation of very interesting and original visual effects on their surfaces. One of the current fashion trends is an aged look for everyday wear. Usually this worn appearance is applied to garments made of denim [2-4]. It may be obtained using laser, scraping, rubbing or various wet treatments with enzymes, acid, bleaching agents etc. The degree of fading may be adjusted to suit the physical properties of the fabrics [2-3, 5-7]. However, washing and fading processes, which change the aesthetic appearances of denim products, also cause deterioration in their structural and mechanical properties, especially their strength and durability [2, 7-8]. Therefore, to reduce the risk of damage to a fabric, the treatment conditions must be carefully regulated. An enzyme wash is ecologically friendly. The enzyme breaks the cellulose fibers from the fabric's surface. The broken fibres and a certain amount of dye are removed

during the washing process. This process must be strictly controlled because enzymes are extremely sensitive to temperature, time and pH. These three parameters have a significant effect on the results and large variations can cause damage to a fabric. The research [9] shows that an increase in enzyme dosage causes cotton fabric to become smoother, but also thinner. Therefore, although enzymatic treatment causes "polishing" of the surface of fibers, it reduces not only a fabric's flexural rigidity but also its breaking strength due to some destruction of the fiber structure [8]. Fading conditions affect the colour change intensity. It was determined that an increase in cellulases concentration increases a fabric's brightness. The amount of colour lost by denim fabrics after treatment with bleaching powder may achieve about 40 % [4]. In an enzyme wash process, cotton fabrics may lose 10 % of their weight and about 5%-15% of their strength [2, 9-10]. Mechanical acting, such as abrasion, may also change a fabric's appearance and colour [2-3, 5, 11]. The degree of change depends on the fabric's characteristics and the conditions of rubbing. Though abrasion may cause a more worn appearance and give a garment an aged look, it can damage the fabric and greatly reduce its mechanical properties. Back-staining, one of the most important problems in denim washing can be removed with the aid of a laccase wash applied after normal washes [3]. The domestic laundering of cellulosic materials can also change their handle parameters and surface smoothness, especially if some chemical softener is used [11].

For the colour measurement of textiles, the Grey scale, CIELAB and Hunter colour systems are widely used in the textile industry [1, 3, 4, 11, 13]. The methods of fabrics

<sup>\*</sup>Corresponding author. Tel.: +370-611-11327, fax: +370-37-353989. E-mail address: *vitalija.masteikaite@ktu.lt* (V. Masteikaitė)

colour fastness evaluation may be divided into two main groups. The first group allows the change in the shade of the specimen to be determined. Using the second group methods it can be observed the degree of staining the white fabric with which the tested fabric was in contact. More often the action called crocking is using for this purpose. The transference of dye from a fabric to another surface happens fairly frequently in over-dyed or heavily dyed fabrics like denims, cottons and silks. The test for colour fastness to washing using multifiber fabric is one of the most basic colour fastness tests used by customers to evaluate end products [5].

The aim of this work is to use various acting and testing methods to examine changes in the appearance of different cellulosic fabrics after enzyme treatment, domestic laundering and abrasion.

#### 2. EXPERIMENTAL

Six cotton and cotton blend woven fabrics used for faded garments production were chosen for this study. Their main characteristics are shown in Table 1. In fabrics A2 and A3 only the warp yarns are coloured, while only the face side of fabric A4 was dyed. The remaining fabrics were dyed after they were woven.

Fabric thickness was determined under a pressure of 0.196 MPa, in accordance with the FAST system [14]; density, in accordance with EN 1049-2:1998; mass per square meter, in accordance with EN 12127:1997. Fabrics dimensional change after washing and drying was measured using the procedure specified in ISO 3759:2011. To calculate area shrinkage after enzyme treatment S (%), following formula was used:

$$S = \{ [l_1 \times l_2 - l_{e1} \times l_{e2}] / l_1 \times l_2 \} \times 100,$$
(1)

where  $l_1$  and  $l_2$  are the distances lengthwise and widthwise between data lines before fabrics treatment, and  $l_{e1}$ ,  $l_{e2}$  are the same distances, respectively, but after enzymatic wash.

In order to evaluate the changes in surface characteristics and resistance of fabrics to some actions we examined the effects of enzyme wash, domestic cyclical laundering, abrasion, and dry and wet rubbing. Specimens, which received no treatment were called 'control'.

**Enzyme wash.** The industrial enzyme wash conditions were used in this work. The specimens were:

- laundered for approximately 30 minutes in a solvent at a temperature of 50 °C-55 °C, using 1.5 % detergent "Beinzym UL", pH 4.5-5.5;
- 2. cold rinsed with soda ash to pH 8-9 for approximately 10 minutes;
- 3. softened with 1 %-3 % "Tubingal Cis" for 15 minutes at 40 °C, pH5-6;
- 4. tumble dried at 100 °C and then allowed to cool.

The specimens were treated in a professional drum machine, "Primus Texcolour NC45DM".

**Domestic laundering.** Domestic cyclical laundering was added for the purpose of analysing the degree to which fabrics faded when worn. Specimens treated with enzymes (e) and untreated specimens (0) were subjected to domestic laundering one, five and ten times. The fabrics were laundered in a home automatic washing machine for 30 minutes at 40 °C with the detergent "Ariel" and then rinsed

for 30 minutes at 10 °C. The specimens were dried on a flat surface, at room temperature.

**Abrasion.** In order to evaluate the resistance to abrasion till fabrics complete distortion, a flat abrasion device IT-2 with contact pressure of 9.3 kPa on the specimen was used. The specimen was abraded until a hole appeared and as characteristic of abrasion resistance the number of cycles N was recorded. The hole appearing was checked by the device automatically. Abrasive paper was used as the abradant. Abrasion resistance was indicated for the control fabrics as N(0) and for the fabrics washed with enzyme as N(e).

A Martindale instrument (EN ISO 12947-1:1998) was chosen for fabrics colour and weight change estimation. The advantage of this instrument is that it is designed to abrade a fabric uniformly at every point on the specimen. It allows measurement not only of any change in the colour of the specimen but also of staining of the abrasive woven worsted cloth. Therefore the colour fastness of the fabric, abrasive staining and weight loss  $\Delta w(a)$  according to EN ISO 12947-3:1998 were measured after abrasion. A round specimen with a diameter of 38 mm was used. A force of 12 kPa was applied to the top of the specimen. 10,000 cycles were used. The weight change characteristic was expressed as a percentage comparing the weight of the control specimens with the weights of specimens after enzyme wash w(a) and after successive abrading w(a) + w(b). These characteristics were marked as  $\Delta w(a)$  and  $\Delta w(b)$ , respectively.

**Rubbing.** Colour fastness to rubbing in wet and dry conditions was evaluated according to EN ISO 105-X12:2001. A constant load of 7 N on the specimen was used during the test. The distance of hand crank movement was 104 mm  $\pm$ 3 mm. Three specimens, each measuring 50 mm × 50 mm, were used for this test. Each specimen was given ten strokes in their warp direction. The degree to which white 100 % cotton fabric used as pocketing was stained by dyestuff was determined. All the abrasion tests were made on the face fabrics side.

**Colour fastness to washing.** The level of dye transfer after one cycle of fabrics domestic washing was evaluated using multifiber adjacent fabric SDC DW (acetate-cotton-nylon-polyester-acrylic-wool) according to EN ISO 105-C06:2010. Every specimen was cut to measure 40 mm  $\times$  100 mm and jointed with multifibre adjacent fabric. Such composite specimens were washed in the wash wheel pot using Grade 3 water, the appropriate detergent and additives with 10 steel balls at a temperature of 40 °C for 30 minutes and then rinsed and dried at a temperature of 60 °C. The degree of staining on the multifibre adjacent fabric was evaluated by determining difference in colour of its parts before and after washing.

The colour fastness of tested fabrics was evaluated using two different characteristics. One was the degree to which a fabric changes colour when subjected to a particular treatment or environment, and the other was the tendency for a textile to cause staining of other fabrics with which it came into contact also after some treatment. The colour change in fabrics during different treatments was measured using a Datacolor reflectance spectrophotometer interfaced to a personal computer. Illuminant D65 and observer 10° were employed during measurement.

Table 1. Characteristics of fabrics investigated

Fabric symbol	Colour	Composition	Density, thread per cm		Weave	Mass per square	Thickness,	Area
			Warp	Weft	structure	meter, g/m <sup>2</sup>	mm	shrinkage <i>S</i> , %
A1	Dark brown	100 % cotton	28	14	Velvet	339	0.99	2.8
A2	Dark blue	65 % cotton, 35% PES	26	19	Plain weave	227	0.36	2.8
A3	Dark blue	100 % cotton	32	21	1⁄2 Twill	298	0.54	8.5
A4	Grey	<ul><li>98 % cotton,</li><li>2 % elastane (in weft)</li></ul>	56	24	1⁄2 Twill	292	0.55	8.9
A5	Camel	43 % cotton, 57 % flax	20	13	Plain weave	228	0.43	9.0
A6	Dark gold	100 % cotton	44	14	Plain weave	293	0.52	6.8

The aperture size of 9 mm and standard aperture plate LAV were used for this test. The average CIELAB values  $L^*$  (lightness),  $a^*$  (redness-greenness),  $b^*$  (yellownessblueness) and  $\Delta E^*$  (colour difference between untreated and treated sample as well as between treated in different ways sample) were determined. The lightness of the specimens is represented by  $L^*$  on a scale ranging from zero for black to 100 for white. The total colour difference  $\Delta E^*$  was determined according to equation (2):

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$
(2)

Weight change. Weight change of fabrics was calculated from the difference in fabric mass per square meter before and after the treatment.

From three to five separate measurements were taken in each case. The coefficients of variation varied from 0.1 % to 2.8 %. All measurements were taken after the specimens had been conditioned in standard atmospheric conditions for 24 hours ( $20 \,^{\circ}C \pm 2 \,^{\circ}C$  temperature, 65 % ±4 % RH).

#### **3. RESULTS AND DISCUSSION**

Abrasion resistance. The received results show that tested fabrics differ in their abrasion resistance. After the enzyme wash resistance to mechanical acting reduces but only for some fabrics (Fig. 1). The decrease ranged between 0.64 % and 37.92 %. A possible explanation is that after enzyme wash, the structure of a fabrics becomes looser, entanglement of individual fibers weakens and destruction of polymers that are the components of fibers takes place [4, 7]. The largest abrasion resistance N was determined for the thickest fabric A1. Of course, the thicker fabrics would stay more abrasion resistant, but there is no strong dependence between these two characteristics. The worst characteristic of N occurred with cotton/flax blend fabric A5, thought the thinnest was fabric A2. The low abrasion resistance of fabric A5 may be due to its comparatively low density. It is known that after enzymatic treatment, linen fabrics became more extensible, weaker, and softer but their surface also became smoother [15] and denser because high shrinkage. Maybe there is a reason that enzyme treatment has no influence on the abrasion resistance of fabric A5. We also found that enzyme treatment has practically no effect on the resistance to abrasion of fabric A1. Its characteristic N remained almost unchanged. Though the resistance to

abrasion of control fabric' A6 was relatively high, after enzymatic treatment it decreased considerably.

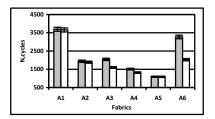


Fig. 1. Abrasion resistance of control fabrics (■) and after their enzyme wash (□)

Weight change. Works by earlier researchers show that the amount of weight a fabric loses generally depends on the enzyme concentration, intensity of mechanical agitation and length of treatment [8, 9]. However, after wet treatment, a fabric may shrink considerably [2]. It is known that fabric shrinkage causes an increase in its area density. Therefore it can be expected a larger specimen of the same area  $(g/m^2)$ weight. As is evident from Fig. 2, there was a marked increase in the weight of fabric A3 after enzyme wash. The total shrinkage in its warp and weft reached 8.5 % (Table 1). The weight increase of fabric A6 after enzyme wash occurred for the same reason. Although fabrics A4 and A5 may also be characterized as very shrinkable, their weight increase is not significant. This effect may be related to the specimens' more intensive lint and dye loss during chemical and mechanical treatment. The latter treatments may cause a decrease in fabric weight.

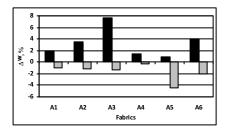
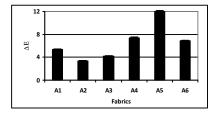


Fig. 2. Fabrics weight change after enzyme wash (■) and successive abrading using Martindale instrument (■); N = 10000 cycles

According to received results, the weight loss  $\Delta w(b)$ , after 10,000 cycles of abrading using a Martindale instrument, varied from 0.99 % to 4.38 % for enzyme-washed fabrics. The maximal weight loss was observed in

fabric A5. A higher value of  $\Delta w(b)$  can also indicate damage to the fabric. This is an unfavourable effect.

**Colour fastness after enzyme wash and abrasion.** Fig. 3 shows the total colour difference  $\Delta E^*$  after enzyme washing of the fabrics. Characteristic  $\Delta E^*$  is within the 3.35-11.96 range. One might expect colour change to be less evident in brighter fabrics.



**Fig. 3.** Control fabrics colour change  $\Delta E^*$  after their enzyme wash

Our results for the characteristic  $L^*$  (Table 2) show that fabric A3 is darkest ( $L^*$  is minimal) and fabric A5 – brightest ( $L^*$  is maximal) in the tested specimens. However, the greatest colour change occurs in fabric A5.

The marked difference between the shade of the control and the washed fabric's A4 specimens may be related to the way this fabric was dyed. Dyeing only the face side of a fabric does not ensure a strong bond between dye and fibres. Therefore, during wet treatment the dye was partially washed out of the surface of the fabric. The minimal change in the fabric's weight after treatments confirms this conclusion (Fig. 3). It is also worth mentioning that fabric A4 contained elastane yarns. It is known that elastane is one of the hydrophobic fibres that cannot be dyed thoroughly. The dye molecules only attach to the surface of the fibres [1]. It is known that the action of cellulases and mechanical agitation, simultaneously or sequentially, abrades the surface of the cellulose fibre and causes defibrillation on the surface. In our tests, an analysis of the colour change which occurred after abrading using a Martindale instrument was made only for enzyme-washed fabrics. This allowed evaluation of the degree of fading after mechanical acting in fabrics that had already treated with enzymes. The characteristics of surface colour are presented in Table 2. The characteristic  $L^*$ characterizes lightness of the sample. The level of darkness can have an influence on fabric's staining characteristic.

**Table 2.** The colour lightness and colour change in fabrics and abrasives after abrading using a Martindale instrument

	Colour	Total colour difference			
Fabrics symbol	lightness	Fabric's	Abrasive's		
- )	$L^*$	$\Delta E^*$	$\Delta E^*$		
A1	25.86	0.91	0.92		
A2	27.86	2.70	6.87		
A3	20.15	6.80	18.14		
A4	49.13	1.68	2.45		
A5	61.75	6.07	1.96		
A6	47.67	8.40	2.91		
Abrasive	78.66	_	_		

According to the received results, a higher fading effect was experienced with fabric A6 while abrading of the surface of fabric A1 had practically no effect on the shade. The results show considerable variation in how much the total colour difference  $\Delta E^*$  value of all the specimens increased after abrasion. A higher  $\Delta E^*$  was determined for fabrics A3, A5 and A6. Such results indicate that fading of clothes made from these fabrics may continue during wear. Fading intensity may be observed in the surfaces of clothes which suffer frequent abrasion forces. It is known that colour difference values increase as the number of abrasion cycles rises but there is an upper limit at which the colour difference values cease to grow [16]. Further surface fragmentation of the fabrics occurs.

Staining is one of the biggest problems with faded garments. Our research shows that there was a higher degree of abrasive fabric staining after the abrasion of fabric' A2 and especially of fabric' A3 ( $\Delta E^* = 18.14$ ). Of course the staining intensity may depend on not only the colour fastness of fabrics but also on their colour darkness. Fabric A3 was the darkest of the tested fabrics (characteristic  $L^*$ ) and the results after abrasive staining show this fabric as having the lowest colour fastness (Table 2). In spite of the facts that after enzyme wash this fabric have changed its colour at some degree (Fig. 3), the subsequent abrasion removed the dye particles once more.

**Colour fastness to rubbing.** The amount of colour transferred from the surface of the tested textile materials to the surface of white cotton fabric as a result of rubbing was evaluated using characteristic  $\Delta E^*$ . The results of the colour fastness test after rubbing in dry and wet conditions are shown in Fig. 4. After rubbing, colour from all tested specimens was transferred to the white test cloth.

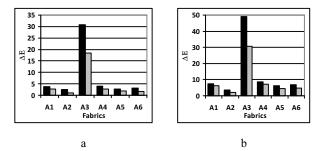


Fig. 4. The degree of adjacent white fabric staining after dry (a) and wet (b) rubbing of control (■) and enzyme washed (■) fabrics

As expected, the staining after fabrics rubbing in wet conditions was of a higher intensity. Enzyme wash, on the other hand, reduced adjacent fabric staining. The higher staining results have shown denim fabric A3. Although the degree of fabric staining decreased considerably after an enzyme wash, it nevertheless remained relatively high.

The comparison of results received using a Martindale instrument for abrasion and rubbing show that the staining degree of most of the tested fabrics is similar, with the exception of fabric A2. The test shows that the level of Martindale abrasive staining was about 6 times higher than that for the white cotton fabric' after its rubbing in dry conditions.

The colour difference  $\Delta E^*$  of multifibre fabric's parts is presented in Fig. 5. The received results show that acetate, acrylic and wool of multifibre adjacent fabric are particularly prone to staining. The polyester part of multifibre adjacent fabric was stained only by fabric A3 and slightly stained by specimens of control fabrics A4 and A5.

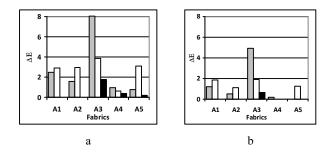


Fig. 5. The multifibre fabrics staining after control (a) and enzyme-washed (b) fabrics washing; ■ - cotton, □ nylon, ■ - polyester

It is worth mentioning that staining of the multifibre adjacent fabric zone was determined after fabric A6 wash. Of course the degree of staining in fabric A3 after wash is considerably lower than after it's rubbing in wet conditions. But this may be a problem which occurs when clothes made from this fabric are worn. The multifibre swatch used in the colour fastness test is the same size as that of the specimen, while the size of sections of a garment's coloured material may be comparably larger than that of the bright parts. Therefore bright material with fewer squares may pick up much more colour than standard tests have shown.

An enzyme wash can achieve a desirable worn look for clothes. However enzyme-washed clothes experience further treatments during wear and there may be additional changes in their appearance. Significant changes in the physical properties of fabrics may be caused by laundering and especially by the repeated laundering of clothes that are worn often.

Bearing in mind the high staining degree of fabric A3 the laundering test was not performed for this fabric.

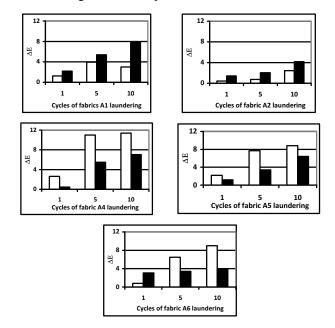


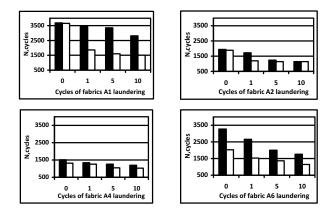
Fig. 6. Control (■) and enzyme-washed (□) fabrics colour fastness after their cyclical laundering

According to received results, repeated laundering of clothes may cause additional fading of fabrics (Fig. 6).

The first laundering cycle caused no significant change in the colour of tested specimens. But when the number of laundering cycles was increased, noticeable changes in colour occurred in some specimens. By comparing the colour fastness results of control, untreated fabrics, it was found that there was a considerable degree of fading in fabrics A4, A5 and A6. After cyclical laundering of enzyme-washed specimens, fabrics A1, A4 and A5 faded more intensively. The results show that the most noticeable fading occurs after the five laundering cycles and further this process slackens.

The influence of cyclical laundering on the abrasion resistance N of tested fabrics was also determined. According to received results, enzyme washed fabrics were the least resistant to cyclical laundering (Fig. 7). Considerable abrasion resistance loss was determined for fabric A1 after its very first laundering. It should be noted that the laundering process has no influence on fabric A5 (the results are not shown). It remains at the same level not only for the control but also for enzyme-washed specimens.

According to received results the higher colour fastness to laundering has shown plain weave cotton/PES fabric A2. After analysing our results, it can be said that this comparably dark ( $L^* = 27.86$ ) fabric has average abrasion resistance, which changed more noticeably not after enzyme wash but after the first laundering cycle.



**Fig. 7.** Control ( $\blacksquare$ ) and enzyme-washed ( $\square$ ) fabrics abrasion resistance *N* after their cyclical laundering

The colour difference  $\Delta E^*$  after enzyme wash for this fabric was minimal. Noticeable staining was determined only after abrasion of this fabric using a Martindale instrument.

The pure cotton fabric A1 of similar darkness has a different structure: it has the highest thickness due to the pile in the surface (velvet). Therefore its abrasion resistance was the highest of the tested fabrics. However, this resistance markedly decreased after laundering, especially for enzyme-washed specimens. The colour change after enzyme wash was average but higher than for fabric A2. The fabric A1 enzyme-washed specimens continued to lose their colour intensity during successive cyclical laundering. The fabric's A1 abrading has no effect on the Martindale instrument's abrasive staining. Some degree of staining was observed after its wet rubbing.

Despite the low cotton/flax blend of fabric A5, abrasion resistance remains stabile during its enzyme wash and cyclical laundering. As for the colour fastness characteristics, the received results show that enzyme wash and rubbing have a considerable effect on this fabric's

colour difference  $\Delta E^*$ . Although fabric A5 was the brightest of the tested fabrics ( $L^* = 61.75$ ), the experiment shows that cyclical laundering also had a considerable effect on the intensity of the colour change in this fabric. The staining after rubbing was not so noticeable but after washing with multifibre fabric it was found to be higher than the colour change in the nylon part.

The cotton fabric A4 containing elastane is dyed only on its face side. The research results show that its abrasion resistance was not high and after cyclical launderings decreased even more. This fabric markedly faded after enzyme wash and stained the white fabric after wet rubbing, but the degree of colour change in the multifibre fabric was minimal. There was also a marked fading in fabric' A4 after its cyclical laundering.

The plain weave pure cotton fabric A6 lost considerable resistance to abrasion after enzyme wash and a further decrease in resistance was evident after cyclical laundering. Enzyme wash and rubbing also have an influence on the degree of colour change in this fabric but staining after abrading and rubbing was not significant. It should be noted that A6 was the only fabric which did not change the multifibre fabric's colour.

Fabric A3 is traditional denim fabric and during manufacture it is specially prepared for fading. The results show that despite high shrinkage after enzyme wash and considerable increase in weight, its abrasion resistance decreases markedly. The colour change after enzymatic wash was average compared with other tested fabrics. However, staining was very high not only after abrading with a Martindale instrument but also after dry and especially after wet crocking. It is also worth mentioning that staining in the multifibre fabric' was high, especially in its cotton section. Taking into account the received results, fabric A3 may be characterized as unsuitable for clothing due to its poor quality, i.e. considerable possibility of staining other fabrics, especially bright ones.

#### **4. CONCLUSIONS**

Based on the results obtained, it was determined that the surface characteristics of cellulosic fabrics depend on their properties and type of treatment. Most of the tested cellulosic fabrics lost their colour not only after enzyme wash but also after successive rubbing and cyclical domestic launderings. The highest degree of fading after enzyme wash occurred in grev cotton/flax blend fabric. Successive abrasion and cyclical laundering also changed this fabric's fade because the weight loss and decrease in abrasion resistance are significant. Colour change after enzyme wash of typical dark blue denim fabrics was average and only more remarkable after successive abrasion, but degree of staining was high. The higher colour fastness after different treatments show the cotton/PES blend fabric while the pure cotton velvet type fabrics more considerable fade achieved only after enzyme wash. The received results show that during laundering of the tested fabrics, the cotton and nylon sections of multifibre fabric were stained more intensively. It should be noted that successive treatments, such as abrasion and laundering, may cause not only further surface colour changes but also staining and the weakening of a fabric's structure.

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