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Improving Some Aesthetic and Performance Characteristics of Wool via Synergistic Action of a Protease and a Cationic Silicon Softener

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Abstract

Production of textile garments with pleasant aesthetic property is always a customer-demand; and the application of eco-friendlier methods to attain this target is driven by the environmental legislation all over the world. This study aims at improving garment appearance and performance attributes of wool fabric by two-consecutive benign processes using a proteolytic enzyme and a functional silicone-based softener. Two commercial proteases, from bovine pancreas Type I and another one from *Streptomyces griseus* Type XIV, were used in bio-treatment of wool under their respective appropriate conditions. The bio-treated wool fabrics were modified with an aqueous solution of a cationic softener based on poly amino siloxane (PAS). The effects of these treatments on some of the physical and mechanical properties of wool fabric were investigated. Scanning electron microscopy (SEM) shows the descaled surface of bio-treated wool, and the coated fibre surface of PAS-treated wool. Results of this investigation showed that the drapability, and hence the aesthetic property, of the treated fabric were improved. The finished fabrics were found to be durable against washing for up to 20 cycles in terms of the fabric drapability. Based on the bending length, drape coefficient, wettability, smoothness, yellowness index, and AC electrical conductivity of the treated wool fabrics, two samples were assigned to be used in prediction of the garment appearance and aesthetic using *CLO3D* simulation software. This evaluation proved that the sample treated with protease enzyme from *Streptomyces griseus* Type XIV followed by aftertreatment with PAS exhibited the best drapeability among the other treated samples.

Keywords: Wool, amino siloxane, enzyme, simulation, drape coefficient, surface properties.

1. Introduction

Wool is one of the most widely used proteinic fibres in the textile and clothing fields by virtue of its outstanding desirable properties including excellent thermal insulation, breathability, flame retardancy and comfort properties [1]. However, most of the woolen fabrics have poor drapability and used for production of fitting woolen garments. Improvement of the drapeability of wool would open the door for production of new woolen garments with low formability, such as skirts. Twitching is another example of the woolen garments especially those which are usually in contact with the human body and made of woolen grades of more than 19 µm diameter. To overcome these two problems, several methods have been reported to improve fabric smoothness [2, 3] and drapability via subtractive and additive methods [4, 5]. Various types of softeners;such as. cationic, anionic, and nonionic softeners, were used to enhance the smoothness of wool fabrics [6].

Silicon-based softeners and their functionalized forms have been widely used in lessening the surface roughness of wool [7]. Softeners based on fatty acids

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were also utilized in softening of wool and other substrates [8]. On the other hand, research studies have been reported for the feasibility of using certain proteolytic enzymes to improve the surface characteristics of wool fabrics;such as smoothness [9], wettability [10], felting resistance [11], anti-pilling [12], and drapability [13]. Application of silicon-based polymers for surface smoothness of wool is an additive method, while treatment of wool with proteases is a subtractive one; a property which would favour the use of the former than the latter to impart specific property for wool for better aesthetic characteristics of wool garments.

Functional and aesthetic properties are two important aspects that affect the garments quality, and both properties are highly influenced by the fabric drape [14]. On the other hand, the fabric drapability is always a function of its bending stiffness, which is considered as the most effective factor influencing the subjective sensory image of fabric [15].

One of the brightest customer-demand in the clothing field is the garment appearance which is a major selling factor in the textile retails. Attaining the best garment appearance can be regarded as the net result of some parameters; the most important of which is the garment design. The garment manufacturing process should adopt energy and time-saving cost effective procedures [16]. Leung *et al.* stated that the final garment appearance can be optimized by controlling the fabric formability and bias extension [17]. Some objective tests and subjective evaluation are allocated for measuring the aesthetic features of fabrics [18].

Sanad *et al.* correlated between the drapability and the garment appearance by using dresses suspended on a mannequin. They developed a graphical user interface to undergo the image analysis and to assess the drape values. TheFAST(Fabric Assurance by Simple Testing)properties, drape coefficient and drape values of selected knitted, woven, and nonwoven fabrics were compared based on visual assessment. Limited agreement was encountered among individuals in terms of the appropriate drape coefficient [19].

Herein, we adopted two-consecutive processes for treatment of wool fabric using proteolytic enzyme followed by functional silicon-based polymer to improve the fabric drapability and smoothness. The synergetic effect of the enzyme and the silicon-based softener on the performance characteristics of wool fabrics would result in woolen garments with improved aesthetic properties.

2. Experiment

2.1. Materials.

Pure crossbred twill weave wool fabric was purchased from Misr Company for Spinning and Weaving, Egypt. Wool fabric specifications are shown in Table 1.

The cationic softener Magnasoft 88 amino-silicon micro-emulsion was kindly supported by Momentive Performance Materials Inc., NY, USA. Two proteases; one from bovine pancreas Type I (activity≥5u/mg) and another one from Streptomyces griseus Type XIV (activity≥3.5u/mg) were purchased from Sigma- Aldrich. The acid dye C.I. Acid Blue 203 was kindly supplied by Egypt Colors Co., Cairo, Egypt. All used chemicals were of laboratory grades and used without any further purification.

2.2. Methods

3.2.1. Scouring

Wool fabric was scoured in an aqueous bath containing 1.5 g/L sodium carbonate at 50 °C for 30 min; the material liquor ratio (MLR) was 1:30, followed by thorough rinsing with cold water and finally air-drying at an ambient temperature [20].

3.2.2.Bio-treatment of wool

The scoured fabric was treated with different concentrations of protease enzyme from bovine pancreas Type I (0.5, 1, 1.5, 2% o.w.f.) at pH 7.5 at 37°C for 1 h, and the MLR was 1:30. In another trial, wool fabric was treated by 1.5 % (o.w.f.) of enzyme at 37 °C over night using the same pH and liquor ratio. The bio-treated wool sample was then rinsed with running tap water followed by washing at 80°C for 10 min to deactivate the enzyme.

The scoured wool fabric was treated also using another protease enzyme from Streptomyces griseus Type XIV. the treatment was carried out by using 1.5% (o.w.f) of enzyme and adjusting pH at 7.5, 1:30 liquor ratio at 37 °C over night then the sample was washed followed by deactivation of the enzyme by washing at 80°C for 10 min.

Table 1: Specifications of the used woven wool fabric

Structure	ends/cm	picks/cm	Warp count (Nm)	Weft count (Nm)	Weight (g/m2)	Thickness (mm)
Twill 2/2	16	18	28	15.28	231	0.68

3.2.3. Softener treatment of wool

An aqueous micro-emulsion (1.5 %, w/v) Magnasoft 88 poly amino-silicon (PAS) was prepared at room temperature with continuous stirring for 20 min. The untreated as well as bio-treated wool fabric was treated by the prepared micro-emulsion with a MLR 1:20. The pH of the treatment bath was keptat 6 for 1h at 80 °C. The treated fabric was picked up from the bath, rinsed thoroughly with tap water and finally left to dry at room temperature**.** Table 2 summarizes the fabric samples' codes and the treatment for each sample.

2.3. Analyses and Testing

2.3.1. Fabric Weight

The fabric weight of the treated as well as untreated wool samples was determined according to [21]. The weight loss (%) of enzyme-treated wool was calculated according to the following equation:

$$Weight\ loss\ \% = \frac{W_1 - W_2}{\dots} \times 1 \quad \text{(Equation 1)}$$

where W1: is the dry weight of sample before treatment and W2: is the dry weight of sample after treatment.

2.3.2. Fabric wettability

The fabric wettability was assessed in terms of drop disappearance in accordance with the standard test method [22], measured by allowing a drop of water to fall on the sample and recording the time required for drop disappearance.

2.3.3. AC electrical conductivity

The electrical conductivity of untreated and treated fabrics before and after sewing was measured using LRC-bridge (Hioki model 3531zHiTester, Japan). The relative dielectric permittivity was calculated using the following equation:

$$\varepsilon' = Cd/\varepsilon_o A \quad \text{(Equation 2)}$$

where "C" is the capacitance of the measured sample in Farad, "d" is the thickness of the thin film sample in meters, A is the cross-section area of the electrode, and εo is the permittivity of free space (8.854x10–12 Fm–1). The dielectric loss (ε") was calculated from the following relation:

$$\varepsilon'' = \varepsilon' \times \tan\delta \quad \text{(Equation 3)}$$

where tan δ is the loss tangent. The AC resistivity of the bio-treated samples was deduced from the dielectric parameters.

2.3.4. Air permeability

Air permeability was measured on FX 3300 air permeability tester 152 (TEXTEST AG, Switzerland) at a pressure of 100 Pa according to ASTM D737 standard method[23].

2.3.5. Water permeability

The water permeability of the treated as well as untreated wool was assessed according to the ASTM–D 1913 155 (American Test Method for Water Repellency; Water Spray Test new edition 156 2010)[24].

2.3.6. Yellowness index

The degree of yellowness of untreated as well as bio-treated wool fabrics was performed on Datacolor Colorimeter 3980 (Datacolor Marl). Each value is an average of five measures determined at different positions on the examined fabric.

2.3.7. Bending length

The bending length of untreated as well as treated wool fabrics was determined according to the ASTM D1388-2018 standard test method[25].

Table 2: Fabric samples' codes

Sample code	Description
B	Untreated wool
I	Wool treated by protease from Streptomyces griseus Type XIV
II	Wool treated by protease from bovine pancreas Type I
III	Wool treated by softener
IV	Wool treated by protease from Streptomyces griseus Type XIV followed by softener
V	Wool treated by protease from bovine pancreas Type I followed by softener

2.3.8. Tensile strength and elongation

Tensile strength and elongation at break of wool samples were measured according to ASTM-D5035-11[26]

2.3.9. Durability test

Adopting the standard AATCC 61-1989 method, the washing durability of the finished fabrics was examined. The treated specimens were washed for 1, 5, 10, and 20 wash cycles. The treated fabric (5x15 cm) was mounted in a launder-o-meter with a detergent solution (200 mL) at 40 °C for 45 min. The fabric drapability of the washed samples were assessed to indicate the washing durability of the finished wool fabric [27].

2.3.10. Fabric drapability

The real and virtual drapability of the fabrics was examined using the Cusick drape meter device, where the sample circle diameter is 25 cm; hold on a 10 cm circular disk. The shadow of the fabric circular sample was digitally captured and the area of draped shadow was analyzed using Adobe photoshop to investigate the drape coefficient percentage (Emulator–How can we help you? clo3d.com, 2022). To validate the method, the fabric drapability test was simulated as the real test on the simulated object, and the shadow of the said sample was analyzed according to the following equation [28].

$$DC(\%) = \left|\frac{A_s - A_d}{\quad}\right| \times 1 \quad \text{(Equation 4)}$$

Where: “DC” is the drape coefficient, “As” is the area of the shadow obtained from the projection of the draping fabric specimen by cm^2, “Ad” is the area of the shadow obtained from the projection of the sample holder in the initial position by cm^2, and “AD” is the area of the shadow obtained from the projection of the fabric specimen in the initial position by cm^2 [28].

2.3.11. CLO3D simulation

The pressure values were investigated using fabric pressure map mode on the software. The CLO3D-Avatar system was used for virtual garment fitting. The pattern pieces were drafted using tools available in the software. The system contributes to improve the quality of designs by checking silhouette and fit sooner in the development process [29].

2.3.12. Scanning electron microscopy

ZEISS LEO 1530 Gemini Optics Lens SEM of 30 kV scanning voltages was used to investigate the morphological structure of the treated as well as untreated samples. EDX measurement conditions were 20 kV accelerating voltage, 21 mm working distance, and 1 nA sample.

3. Results and Discussion

In our attempt improve the fabric drape of wool, we adopted a subtractive method using protease enzyme from either bovine pancreas Type I (Enzyme I) or Streptomyces griseus Type XIV (Enzyme II), additive treatment using poly amino siloxane (PAS), or a combination thereof. Although different studies have been reported about chemical processing of wool [30–32], yet and up to our knowledge, none of these investigations has been directed towards improvement of the fabric drape for the sake of better aesthetic properties of the final garment.

3.1. Effect of enzyme concentration and bio-treatment time

During modification of textile substrates using subtractive (using enzymes), additive (using polymers), or even combination thereof; the net weight loss or gain will be of prime importance. In Table 3, the effect of enzyme concentration on the drape coefficient as well as the weight of the bio-treated wool fabric, was elucidated. The data of this table shows that treatment of wool with enzyme I resulted in noticeable improvement in the fabric drapability (as indicated by the decrease in the drape coefficient). As theenzyme concentration increased from 0.5 up to 1.5 % (o.w.f.), the drape

coefficient decreases. No appreciablechange on thefabric drape coefficient was monitored upon increasing the enzyme concentration from 1.5 % to 2 %.The limited decrease in the bio-treated fabric drapability could be attributed to the partial removal of the scales of wool surface which constitutes the hardest part of wool [33]; thus the fabric elasticity and drapability would be improved accordingly. On the other hand, a maximum loss in weight of 2.1 % was recorded upon bio-treatment of wool with the said enzyme.

The effect of bio-treatment duration on the fabric drape coefficient and weight of wool fabric was investigated, and the results were summarized in Table 4. It is obvious from this table that as the treatment duration time increased, the fabric drapability increased. Bio-treatment of wool fabric with 1.5 % (o.w.f.) enzyme I for 24h resulted in improvement of the fabric drape by a factor of ca. 6.5 % with a loss in weight within the acceptable limits (4.5 %).

For comparison, wool fabrics were treated with the enzymes I and II, each separately, at the same conditions; 1.5 % (o.w.f.) of enzyme for 24 h at 37 °C. The extent of improvement in the fabric drape coefficient (7.87 %) which is higher than the corresponding value in case of enzyme I. The loss in weight in case of enzyme II-treated wool is significantly higher than that in case of wool treated with enzyme.

It is worthy to mention that loss in weight of the bio-treated wool fabric was compensated by post-treatment with the PAS; thus, there will be no appreciable net weight loss at the final stage of the finished wool fabric.

3.2. Physical properties

The effects of bio-treatment and/or PAS-treatment of wool fabrics on some of its inherent physical characteristics were monitored by testing their yellowness index, wettability, air permeability, water permeability, and electrostatic charge. The results of this investigation, summarized in Table 5, revealed the followings:

- Bio-treatment of wool with Enzyme I or II resulted in decreasing its degree of yellowness; presumably due to descaling of the fibre surface under the influence of the proteolytic enzyme [34]. On the other hand, the yellowness index of enzyme-treatedwool was increased again under the influence of heat during curing of the PAS-post-treated wool [35].
- The remarkable improvement in the wettability of the bio-treated wool could be attributed to the removal of the outermost lipid barrier from wool surface as a result of partial descaling of wool [36]. The fabric wettability was even enhanced by post-treatment of wool with the amino groups-containing softener. Scheme 1 illustrates the bonding between PAS and wool keratin macromolecule through hydrogen bonds and/or salt linkages.

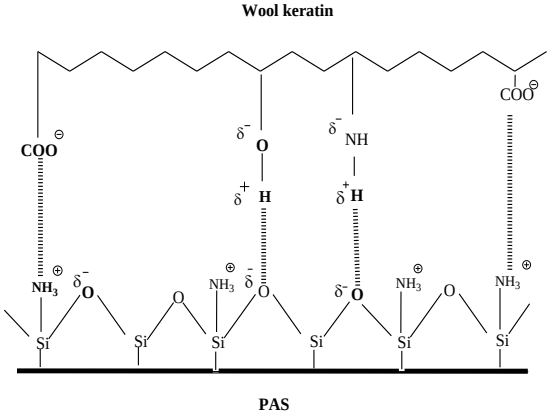
Table 3: Effect of enzyme concentration on the bending stiffness and weight of biotreated wool fabric using Enzyme I (Treatment conditions: 1h at pH 7.5 at 37°C; MLR: 1:30)

Enzyme conc. (% o.w.f.)	Drape coefficient (%)	Weight loss %
Untreated	43.44	0.0
0.5 % for 1h	42.87	0.84
1.0 % for 1h	42.29	1.17
1.5 % for 1h	42.02	1.22
2.0 % for 1h	42.00	2.10

Table 4: Effect of bio-treatment time on the bending stiffness and weight of biotreated wool fabric using Enzyme I [Treatment conditions: 1.5 % (o.w.f.), pH 7.5 at 37°C; MLR: 1:30]

Treatment duration (h)	Drape coefficient (%)	Weight loss %
Untreated	43.44	0.0
1 h	42.02	1.22
5 h	41.74	2.17
10 h	41.37	3.05
24 h	40.62	4.50
1.5 % Enzyme II for 24 h	40.02	8.5

- The air permeability (AP) of wool fabric was enhanced to a limited extent upon bio-treatment with the said enzymes, indicating that no alteration in the fabric porosity was taken place upon finishing of wool with enzyme I or II. Treatment of wool with the PAS led to remarkable decrease in its AP, most probably due to a decrease in the fabric porosity by virtue of the encapsulation of wool fibres with a thin layer from the used PAS. Similar trend was observed regarding the water permeability of the treated wool fabrics. These findings assure that the principal parameters which influence the fabric air and water permeability were not drastically affected; such as yarn and fabric type and structure.



Scheme 1: The mode of action between wool keratin macromolecule and the applied polymer

- Whereas the electrical conductivity of the bio-treated fabrics is almost the same as the untreated one, the PAS-treated fabric exhibited higher ability to impede the formation of electrostatic charges, as indicated by the higher AC conductivity value for the latter. This may be attributed to the induction of new hydrophilic groups (the amino groups) into the PAS-treated fabric, a factor which is of great importance in preventing formation of electrostatic charge on fabric surface [37].

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3.3. Mechanical properties

The effect of treatment of wool fabric with protease/PAS softener on some of its mechanical properties was monitored. Results of this investigation, tabulated in Table 6, elucidate that bio-treatment of wool fabric with enzyme I or II decreased its tensile strength, while post-treatment with PAS re-increased the tensile strength, compared to the untreated sample. This finding is a direct consequence of the degradative nature of bio-treatment and additive nature of polymer deposition. On the other hand, the elongation at break of either bio-treated or polymer-treated samples was not adversely influenced.3. Keywords list all keywords in order of importance, separated by semicolons and should be typed in Times New Roman, 10-point, non-italic and non-boldface.

Results in Table 6 reveal also the bending length of wool fabric treated with protease/PAS system is lower than that of the biotreated or PAS-treated fabrics, and each of the latter has lower bending length than the untreated sample. Whereas the removal of the rigid cystine-rich scales of bio-treated wool might be the reason of the decrease in the bending length [38]; the softening action imparted to wool fabric by application of PAS may rationalize the reduced bending length of the PAS-treated fabric [39]. These results are in harmony with the drape coefficient values of the treated sample (c.f. Table 6).

3.4. Fabric drapability

The drape coefficient of the bio-treated and PAS-treated wool fabric was assessed and the results were abridged in Table 7 The results in this table can be correlated with the bending length of the treated fabric shown in Table 5. The fabric weight is another factor which is crucial in determining the fabric drape. It has been reported that the fabric drape is the extent of distortion in the fabric shape as a result of the change in its weight; or the way in which the fabric falls under the influence of gravitational force, if only a part of it is firmly buttressed [40]. Treatment of wool fabric with PAS increased its weight by a factor of 7 %; hence the DC decreased remarkably.

Thorough inspection of Table 7 reveals that the number of pleats in the tested samples increased from 4 to 5 upon bio-treatment with enzyme I or PAS, and to 6 upon treatment of wool with either enzyme I or II followed by post-treatment with PAS. These results indicate that wool fabric treated with protease/PAS system has a more harmonic drape than the untreated or those treated with enzyme or PAS only.

Table 5: Effect of bio-treatment of wool fabric with Enzyme I and/or PAS on some of its inherent physical characteristics

Sample code	Yellowness index	Wettability (min)	Air perm. (cm3/s/cm2)	Water perm. (cm3/cm2.L)	AC conduct. (S/cm)
B	22.40	Up to 30 min	74.08	1.12	3.99 x 10–5
I	15.30	5.7	76.88	1.13	4.00 x 10–5
II	16.44	5.1	79.37	1.03	4.00 x 10–5
III	24.09	12.5	60.14	9.74	4.00 x 10–5
IV	23.30	1.2	70.23	9.44	9.40 x 10–5
V	19.16	1.0	71.46	9.37	1.17 x 10–4

Table 6: Effect of bio-treatment of wool fabric with Enzyme I and/or PAS on some of its mechanical characteristics

Sample code	Tensile strength (Kg)	Elongation (%)	Bending length (Cm)
B	21.4	55.0	4.0
I	17.9	50.2	3.6
II	17.4	48.9	3.8
III	22.4	48.5	3.3
IV	19.8	53.6	3.0
V	19.7	54.1	2.8

3.5. Fabric aesthetic features

Fabric drape is an important feature which highly influences the garment aesthetic appearance [41]. Based on the results of fabric drape coefficient of the treated fabric; we chose samples IV and V to estimate their aesthetic property on Avatar using *CLO3D* software. Figure 1 shows the front, back, and side views of dress made from untreated wool fabric as well as those samples treated with enzyme I or enzyme II followed by post-treatment with PAS. It is obvious from this figure that the sample V has the best drapability, compared to the untreated and IV samples.From the aesthetic point of view, sample V affords fabric fall and shape which is distinct to the designer's inspiration towards distinguished garment.

3.6. Fabic durability

The fabric drapeability of the finished wool fabric were evaluated after various washing cycles and the results are shown in Table 7. The data therein revealed that the treated fabrics have adequate durability .against washing until 20 cycles. The fabric drapability of the wool fabrics treated with protease/PAS, after washing, are almost the same as the corresponding unwashed samples. This emphasizes that the applied PAS was tightly bonded to the wool fabrics

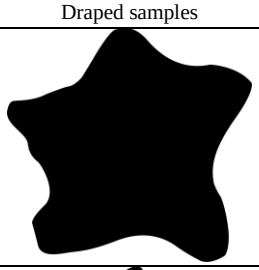
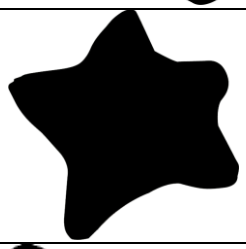
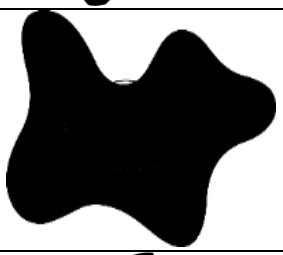
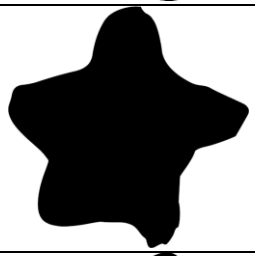
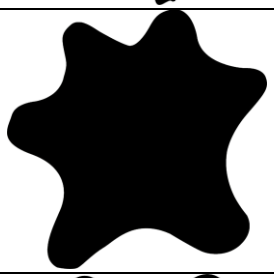
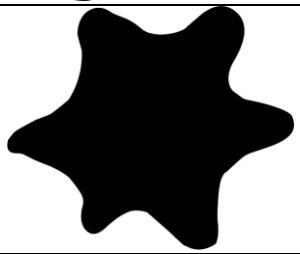
3.7.Fibre morphology

The alteration in the morphological structure of wool fabric after being bio-treated with protease enzyme I or II, treated with the cationic softener PAS, and combination thereof was investigated and illustrated in Figure 2 This figure shows that bio-treatment of wool with the protease enzyme I or II caused partial descaling of the fibre surface without severe fibre deterioration. Controlled wool fibre descaling is one of the methods usually adopted for improvement of the resistance of wool to felting shrinkage [42], pilling [43], surface wettability [44] and smoothness [45]. The scanning electron micrographs of untreated or bio-treated wool fabrics after being post-treated with PAS show a thin layer of the used polymer on the fibres surface which would enhance the fabric smoothness and reduce the tendency of the fibres to felt-shrink [46].

4. Conclusions

Based on the above finding, it is concluded that there is the synergism between a protease enzyme and a cationic softener based on amino siloxane, is useful in improving the wool fabric drapability towards aesthetically appealing woolen garments. The used proteolytic enzymes were successful candidates to partially descale the wool fibre surface without degradative action under the adopted experimental conditions. The used cationic softener was effective in improving the fabric drapability by virtue of its smoothening action.

Table 7: Drape coefficient (DC) of wool fabric treated with protease enzyme followed by poly amino silicone softener

Sample	Draped samples	DC (%)
Blank		43.44
I		40.62
II		40.02
III		39.33
IV		38.94
V		34.53

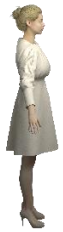
	Blank	IV	V
Front			
Back			
Side left			
Side right			

Figure 1: From, back, and sides views of dress made the untreated as well as treated fabric samples IV and V

Table 7: The drapeability of the finished wool fabrics after different washing cycles

Wool sample	Fabric drape (%)				
	0 wash	1 wash	5 wash	10 wash	20 wash
I	40.62	40.63	40.66	40.75	40.75
II	40.02	40.09	40.12	39.14	39.18
III	39.33	39.43	39.61	39.88	39.00
Iv	38.94	38.98	39.10	39.19	39.21
V	34.53	34.50	34.64	34.71	34.75

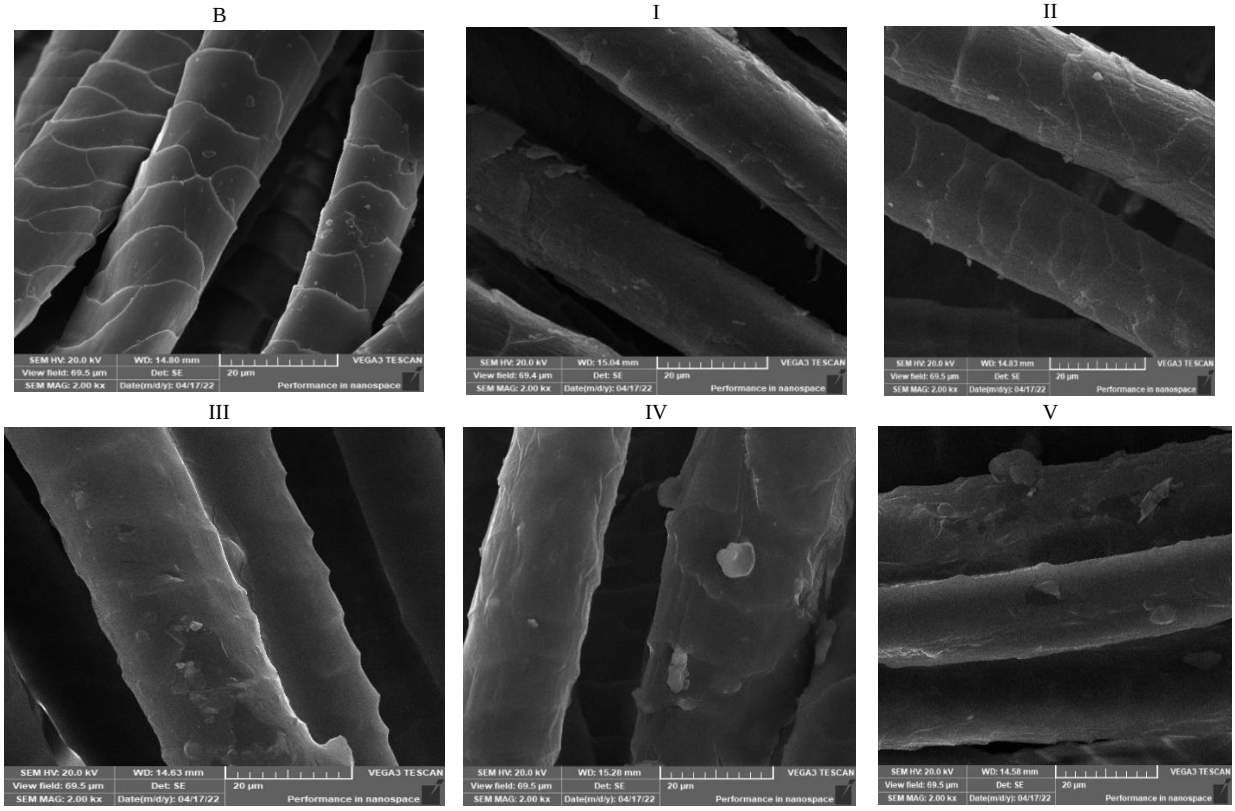


Figure 2: Scanning electron micrographs of untreated as well as treated wool fabrics

It is worth to conclude that treatment of wool with the system protease/cationic softener should be carried out on before dyeing to avoid the expected decrease in the fabric dyeability, the unevenness, and the change in hue if the fabric was treated then dyed.

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