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# The Synergetic Effects of Alternative Methods in Wool Finishing

## Abstract

*Knitted wool fabrics were treated with atmospheric argon plasma, enzyme (protease), chitosan and a combination of these processes. The treated fabrics were evaluated in terms of their dyeability, colour fastness and shrinkage properties, as well as bursting strength. The surface morphology was characterised by SEM images. In order to show the functionalisation of a wool surface after plasma treatment, XPS analysis was done. Our results reveal that atmospheric plasma has an etching effect and increases the functionality of a wool surface, which is evident from SEM and XPS analysis. Atmospheric plasma treatment enhances the adhesion of chitosan to the surface and improves the hydrophilicity of the fibre. Enzymatic treatment improves the coating ability of chitosan. All these processes produce high dyeing efficiency and shrink-resistance in wool fabric. Plasma treatment has no significant effect on bursting strength, unlike enzymatic treatment. The loss of bursting strength after plasma and enzymatic treatment is recovered, to a certain degree, by chitosan treatment. Significant changes in the fastness properties of the treated fabrics were not observed.*

**Key words:** wool, knitted fabric, finishing plasma treatment, enzymatic treatment, chitosan.

## Introduction

Wool is composed of 82% keratinous protein (characterised by a high concentration of cystine), 17% non-keratinous protein, which has a relatively low cystine content, and a 1% by mass of non-proteinaceous material consisting of mainly waxy lipids, plus a small amount of polysaccharide materials. The hydrophobic nature of wool is known to be due to the specific properties of exocuticle and epicuticle. Exocuticle is hydrophobic because it contains a high degree (35%) of disulphide crosslinkage. Epicuticle surrounds each cuticle cell of wool fiber, and it consists of fatty acid (25% by mass) and protein (75% by mass). The surface morphology of wool plays an important role in wool processing. Unwanted effects, such as felting and a diffusion barrier are most probably due to the presence of wool scales. The presence of scales on a wool fiber surface also introduces a number of problems, such as felting and a surface barrier to dyestuffs, in the wool industry. In the past, chemical methods were the major treatment for eliminating these problems. Modifications of wool surface morphology were conducted either by chemical degradation of the wool scales or by deposition of polymer on the scales [1 - 5].

Among the traditional chemical and mechanical processes, biotechnological application in textile processing is gaining more and more interest. Nowadays, the use of protease enzymes to achieve wool shrinkage, better whiteness, improved handle and dyeing is of considerable interest. Also, synthetic polymers can be

applied to wool fabrics to improve such properties as shrink resistance (silicone polymers etc), stain and water repellency (fluoropolymers), and flame resistance. Much attention is focused on natural polymers as possible substitutes for synthetic polymers. The application of biopolymers has attracted a great deal of scientific and industrial interest as possible substitutes [6 - 8]. As a substitute for synthetic polymer, chitosan is proposed because of its unusual combination of chemical and biological properties. Chitosan is a modified biopolymer obtained by the deacetylation of chitin (the second most abundant polysaccharide found in nature) and has a structure similar to that of cellulose. Chitin itself is widespread in nature and is available for industrial application in large quantities, i.e. as a waste product from crab fishing. Chitosan is obtained by means of deacetylation in a hot concentrated NaOH solution [2].

In recent years the application of chitosan in industry and science has been widely promoted. The most important chemical properties of chitosan are attributed to its polyamine character, which makes the polymer water-soluble (at acidic pH) as well as positively charged and confers bio-adhesive properties. The biological properties of chitosan include biocompatibility and biodegradability. These chemical and biological properties form the basis of a number of potential applications in food processing, agriculture, hair and skin care products, membranes and microcapsules, biomedical and waste water treatments [2]. In the field of textiles, chitosan has been/is used as a shrink-resisting agent and as an agent

for improving the dyeability of wool, peroxide, wastewater treatment, removing neps or odour, and as an antimicrobial agent. The positive charge (at pH < 6.5) gives CHT its unique chemical and biological properties and makes it attractive for a wide number of textile applications, whereas its solubility in acidic solutions makes it easily available for industrial purposes. In aqueous solutions chitosan, in its protonated form, has an affinity of interaction with oppositely charged molecules or surfaces, such as enzymes and wool, respectively [7, 8].

Even such a low quantity of chitosan sorbed on a fiber surface during aqueous washing, reduces the frictional coefficient of the fibers. The main problem for chitosan applications to wool is that it is weakly bound to the substrate as it is supposed to interact electrostatically with available anionic groups. For instance, the shrink-resist properties obtained with plasma treatment do not impart a machine-washable finish, which is one of the most important end-user demands. In order to improve shrink-resistance or anti-felting properties, further treatment, such as a biopolymer deposition on the wool fiber, is required. To enhance chitosan's adsorption on wool fibers and increase the uniformity of its distribution, it is convenient to increase the anionic character of the wool surface. Therefore, to enhance CHT binding, it could be useful to promote the formation of new anionic groups on the fiber and to increase the surface energy of wool fibers. To achieve this, one of today's established methods is the LTP treatment of wool [7, 9 - 12].

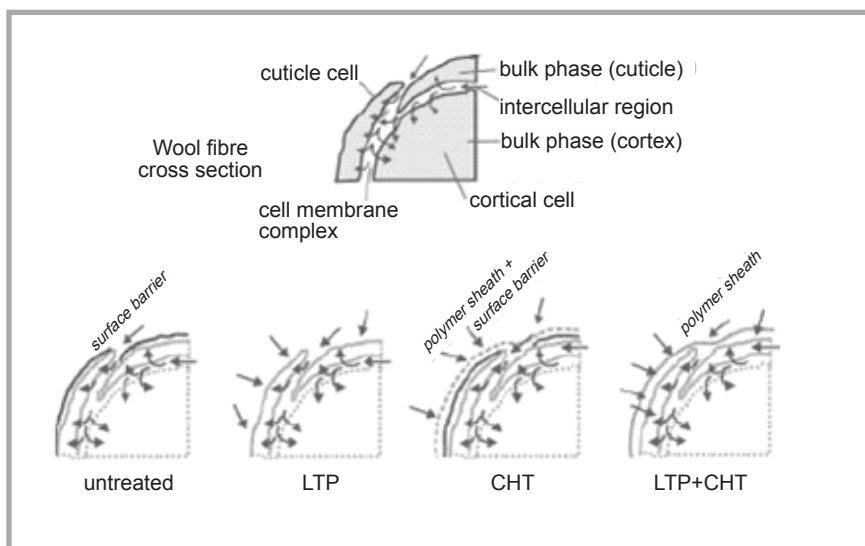


Figure 1. Dye penetration pathways [6].

Cold plasma treatment is a physical method which is used for surface modification, as it affects the surface both physically and chemically without altering the bulk properties of the material. Low temperature plasma treatments of wool only modify the cuticle surface of the fibers, generating new hydrophilic groups as a result of hydrocarbon chain oxidation, reducing the chain length of fatty acid, improving their surface wettability, dyeability, fiber cohesion, and shrink resistance. The oxidation process also promotes cystine oxidation in the exocuticle, converting it into cysteic acid, and thus reducing the number of crosslinkages in the fiber surface [7, 9 - 12]

The dye penetration in wool are presented in Figure 1 [6].

In this study, knitted wool fabrics were treated with enzyme, argon atmospheric plasma and chitosan in an attempt to observe some characteristics of wool fibers, such as dyeability, fastness, shrinkage properties and bursting strength.

## Experimental

### Materials

Woollen knitted fabrics with an interlock weave (supplied by Yünsa A.Ş.) and an area weight of 251 g/m<sup>2</sup> were the test objects. The Chitosan used with a 82.7% degree of deacetylation and (Sigma-Aldrich) molecular weight (375,000). Enzyme (protease) was kindly supplied by Dr. Petry, Germany. Supranol Rot BL01 (milling type acid dye, pH = 5.5 - 6) was obtained from DyStar.

### Treatment of the fabric samples

The woollen knitted fabrics had the treatments stated below:

#### Atmospheric plasma treatment

In this study, a dielectric barrier discharge (DBD) atmospheric plasma device was used. The distance between the electrodes was 0.2 cm. The samples were placed between the electrodes and passed continuously with the speed of 0.45 m/min. In all treatments, argon was used as process gas under a constant power of 200 W. The fabrics were passed 15 times (15 passages) from the plasma chamber.

#### Enzymatic treatment

Enzyme treatments were (2 g/l) applied by the exhaustion method for 1 hour at a liquor ratio of 30:1 using an ATAC LAB DYE HT 10 (Ataç, Turkey) machine at 70 °C and pH = 8 with a 0.3 - 0.5 g/l non-ionic surfactant. Following the enzymatic treatment, the fabrics were rinsed with water at pH= 4, temp. 40 °C and maintained at the same temperature for 10 minutes to denature the enzyme. Finally, the fabric was rinsed several times with deionized water to remove any remaining enzyme from the treatment.

#### Chitosan treatment

For the chitosan treatments, chitosan solutions were freshly prepared by dissolving the desired amount in distilled water containing acetic acid (3%). The Treatments were performed by padding wool fabrics (100% WPU). After treatment, the samples were squeezed and dried at 40 °C.

## Characterisation of modification

### Dyeing

The dye used in this study was Supranol Rot BL01 (milling type acid dye, pH = 5.5 - 6). The liquor to fabric ratio was 15:1. Fabrics were dyed for 15 min at 50 °C, and the temperature was then raised to 98 °C after 30 min. and kept at this temperature for 60 min. After dyeing, the fabrics were rinsed with cold-hot-cold-hot water and then dried at room temperature. The *K/S* values of the dyed fabrics were measured using a HunterLab ColorQuest II spectrophotometer instrument over a wavelength range of 390 - 700 nm. In a typical test, reflectance values (*R*) were measured and relative colour strength (*K/S*) values were then established according to the following Kubelka-Munk equation:

$$K/S = [(1 - R) / 2R],$$

where *K* and *S* are the absorption and scattering coefficients, respectively.

### Colour fastness

The colour fastness to washing was determined according to BS EN ISO C06 at 40 °C, the colour fastness to rubbing according to BS EN ISO 105 X12, and the colour fastness to light according to BS EN ISO B02.

### Determination of shrinkage

Wash shrinkage tests were carried out in a Wascator using the ISO 6330 5A programme, as described in IWS Test Method 31.

### Bursting strength

The bursting strength was determined for samples after conditioning for 48 h at 20 °C and 65% relative humidity. The Bursting strength was obtained according to ISO 2960, appropriate/according to the diaphragm method. The bursting strength values were measured in kPa (kG/cm<sup>2</sup> × 10<sup>-2</sup>).

### XPS Analysis

X-ray photoelectron spectroscopy was used to monitor the modifications produced in the outermost (5 - 10 nm) wool fibre surface. In our studies, the measurement depth was 7 nm. The wool fabric samples were analysed using the SPCS XPS system with a Mg K $\alpha$  X-ray source operated/operating at 10 kV and 200 W. The pressure of the internal analysis chamber was 1.33 to 0.133  $\mu$ Pa. Survey scans were taken with/at a pass energy of 48 eV. The peak positions were corrected

for charging relative to the C-C bond, which was assigned a binding energy of 284.5 eV.

### SEM Analysis

For surface observation, changes in the fabric surface were evaluated using scanning electron microscopy (SEM). SEM observations were made with a Phillips XL-30S FEG scanning electron microscope. Samples were mounted and gold sputtered in a vacuum prior to observation.

## Results and discussion

To observe the dyeing properties of fabrics, acid dyes were used. The *K/S* values and fastness properties of the fabrics were evaluated.

### Dyeing properties

Plasma, enzyme and chitosan treatments increase the colour strength of wool fabrics, even if they are not combined [6, 13, 11, 14]. The *K/S* values of treated fabrics are given in Figure 2. Plasma + enzyme + chitosan combined treatment has the highest dyeing efficiency, as stated above.

The colour difference between chitosan and plasma + chitosan treated fabrics is caused by the increased surface energy after plasma treatment. Plasma treatment enhances chitosan binding by promoting/introducing new anionic groups on the fiber, as can be seen from the XPS results.

Due to the chemical effect of plasma species, new functional groups occur which cause some changes in the surface composition. It can be seen from Figure 3 that the carbon content is significantly reduced from 77.0 to 64.2 after plasma treatment. This reduction is probably due to the etching effect of atmospheric argon plasma treatment [15 - 21]. As we can see from Figure 3, there are significant differences between untreated and plasma treated wool fabrics. The peak at 284.2 eV corresponds to the -CH peak, 288.02 eV to C=O and 291 eV to -COO-. An increase in carboxylic acid after LTP treatment is a result of the oxidation of hydrocarbon chains located on the wool surface, which is in accordance with the degrees of the amount of covalently bound surface lipids present in wool [15, 22, 23]. It is obvious that the intensity of the oxygen peaks of an/the Ar plasma treated surface is much stronger than that

of an untreated surface. The oxygen content is increased from 15.5 to 31.2, and the O/C ratio of samples from 0.2 to 0.48. These changes can be attributed to the partial oxidation of hydrocarbon chains of the F-layer without the removal of the epicuticle, and it can be expected that the rich surfaces of oxygen containing groups were produced after Ar-plasma treatment [15, 19, 21, 22]. The increased amount of oxygen causes an improvement in the hydrophilicity of wool fiber. As a result, dye uptake and chitosan adhesion during finishing was also enhanced. These results are compatible with the dyeing and shrinkage results.

When plasma is used prior to enzymatic treatment, the accessibility of the wool surface to the enzyme action was increased. As a result of this, the dyeability of wool increases. The *K/S* values of

enzyme + chitosan is greater than that of plasma + chitosan. This difference can be attributed to their surface modification effects. Plasma treatments can modify the chemical composition and/or partially remove material on the wool fiber surface. On the other hand, enzymes can remove the hydrophobic layer of wool more efficiently.

Jocic et al. stated that during dyeing, the dye adsorbed in chitosan should migrate to the fiber surface and diffuse in the fiber bulk with time [6]. We assumed that this diffusion is affected by the composition of the cuticle layer. If we compare the effects of plasma and enzymatic treatment on the cuticle layer, it is obvious from SEM pictures that plasma modification is more limited than enzymatic treatment. Therefore, the combination of enzymatic treatment and chitosan gives higher

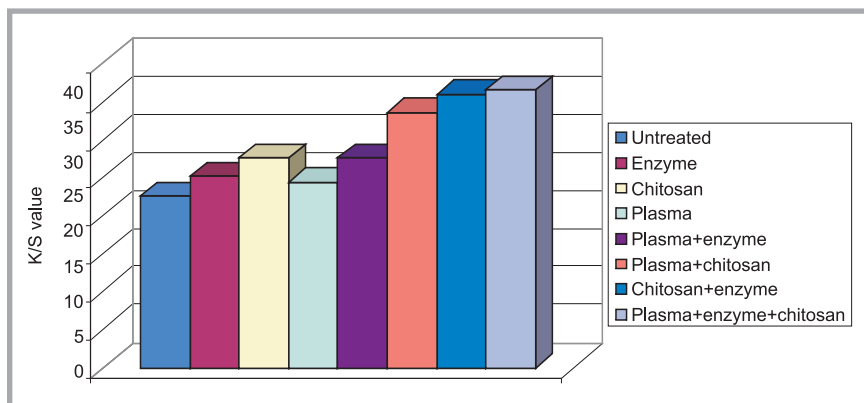


Figure 2. *K/S* Values of acid dyed fabrics.

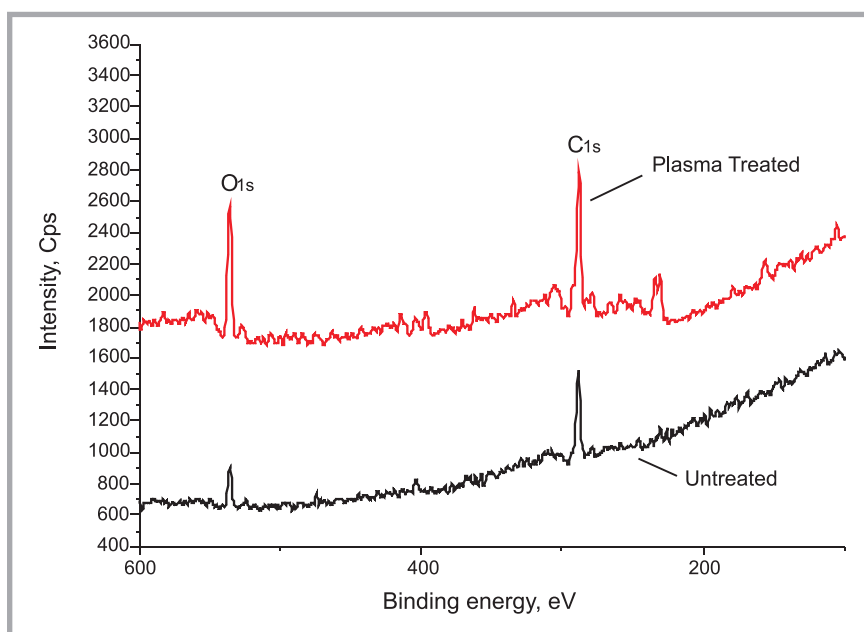


Figure 3. XPS peaks corresponding to untreated and plasma treated wool fabrics. The peak is between at 230.8 and 234.5 and is attributed to molybdenum caused by the sample holder.

dyeing efficiency than plasma+chitosan treatment.

### Fastness Properties

The colour fastness to washing and to dry and wet rubbing were studied in dyed samples. In the case of colour fastness to washing, all samples showed staining and colour change values of between four and five. When the colour fastness to dry and wet rubbing was studied, similar values of staining in dry and in wet (ranging between three and four) were observed among the treated fabrics. Since dye molecules were adsorbed on the surface and could not penetrate into the fiber as easily as treated samples, all treated fabrics show better values than untreated fabric to half a degree.

The values of fastness to light of the treated and untreated samples did not differ from each other within the range of 5.5-6. The results of light fastness show that plasma, enzyme, chitosan and the combined treatments have no detrimental effect on light fastness values.

The rubbing fastness values of the treated fabrics are presented in Table 1.

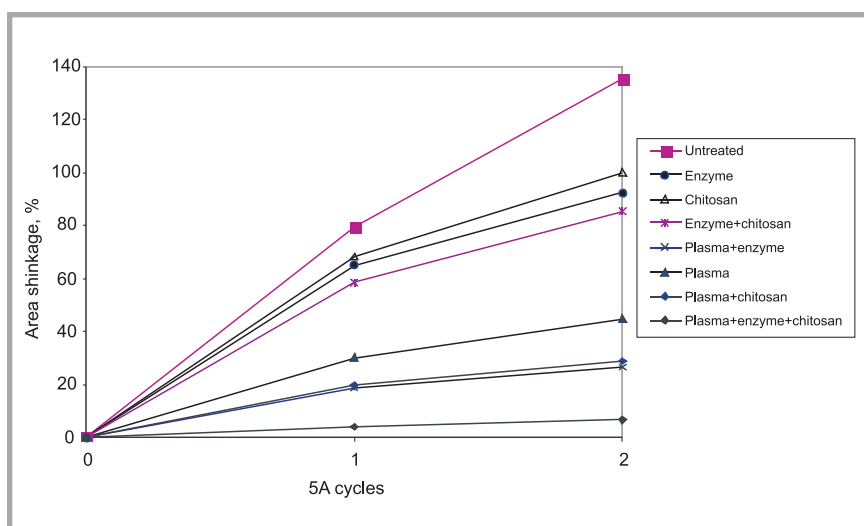
### Shrinkage properties

As is known, the etching effect of plasma reduces the differential friction coefficients of fibres and thus decreases the natural shrinkage tendency [9]. Also, proteases are used for the removal of scale cuticles or smoothing the edges [25]. Chitosan provides felting shrinkage to wool by covering the cuticle surface scales of wool. After combined plasma + enzyme + chitosan treatment, the area shrinkage of treated wool shows a significant reduction in contrast to untreated samples.

The area shrinkage of untreated and wool samples subjected to different treatments

**Table 1.** The rubbing fastness values of the treated fabrics.

Samples	Acid Dyes	
	Dry	Wet
Untreated	3-4	3
Enzyme	4	3-4
Chitosan	4	3-4
Enzyme+ Chitosan	4	3-4
Plasma+ Enzyme	4	3-4
Plasma	4	3-4
Plasma +Chitosan	4	3-4
Plasma +Enzyme+Chitosan	4	3-4



**Figure 4.** The area shrinkage of untreated and wool samples subjected to different treatments as a function of 5A Wascator shrinkage test cycles.

as a function of 5A Wascator shrinkage test cycles are presented in Figure 4.

After two 5A Wascator shrinkage cycles, the area shrinkage values are close to 8%. When the area shrinkage was lower than 8% after two 5A cycles, the wool was regarded as “machine washable” [8].

### Bursting strength

Bursting strength values of the wool fabrics are presented at Table 2.

It is known that enzymatic treatment has a negative effect on the strength of wool fabrics [24]. As can be seen from Table 2, plasma treatment has no significant effect on the bursting strength, unlike enzymatic treatment. During the enzymatic treatment with conventional proteases, damage to wool fibres was caused by enzyme penetration into the interior of fibres and a breaking down of the cell membrane complex. Protease enzymes are known to penetrate and degrade the internal structure of wool during processing, causing fibre damage [5]. Bursting strength data show that enzymatic treatment is affected by plasma treatment, by means of which enzymes can penetrate better. Chitosan has a coating effect on the fibre surface of the wool. The results reveal that chitosan treatment reduces the loss of bursting strength caused by the subsequent enzymatic treatment.

In order to enhance the bonding efficiency of chitosan polymer with wool and to increase the uniformity of its distribution on surfaces, it is good practice to increase the surface energy and anionic character of a wool surface. We can assume that the

increase in bursting strength after plasma and chitosan treatment is dependant on the increase in the reactivity of the wool surface.

As can be seen from the results, fabric bursting strength is negatively affected by the blank enzymatic treatment, and the etching effect of plasma treatment makes enzymatic treatment much more effective, which causes a reduction in strength. On the other hand, during the post-treatment of chitosan, a clear protective effect on the bursting strength can be observed. In other words, the loss of bursting strength after plasma and enzymatic treatment are recovered, to a certain degree, by chitosan treatment, which is within an acceptable range. According to Höcker, the loss of strength should be between 15 - 25% after enzymatic treatments [23].

### SEM analysis

In order to observe topographical changes, SEM observations were made. Figure 5 shows surface appearances of the treated and untreated wool samples.

**Table 2.** Bursting strength values of the wool fabrics.

Treatment	Bursting strength, kPa
Untreated	531.7
Enzyme	449.0
Enzyme+chitosan	461.6
Chitosan	548.3
Plasma	527.5
Plasma+chitosan	542.9
Plasma+enzyme	438.3
Plasma+enzyme+chitosan	450.5



As seen from the images, both atmospheric pressure plasma and enzymatic treatments cause degradation on the surface of the fiber in different ways. The dominant effects of the two processes are different. In the plasma process, only 1000 Å of the surface is affected [3, 16, 25, 26], etching is dominant and causes partial degradation, such as rounding scales, microcracks, recesses and tiny grooves. On the other hand, enzymatic treatment has a hydrolytic effect causing bond-cleavages, which makes the effect more degradative. As regards chitosan, it covers the scaly surface of the fibre and a smoother appearance of wool surfaces can be obtained without any damage. When the dual combinations (plasma + enzyme, plasma + chitosan, enzyme + chitosan) are evaluated, it can be seen that they are more effective than one process. Any combination which contains enzymatic treatment produces a smoother surface. These three processes enhance the smoothness of the surface through a combination of etching, hydrolysing or covering effects. Finally, a plasma + enzyme + chitosan combination causes the smoothest surface. This result is inconsistent with the shrinkage results.

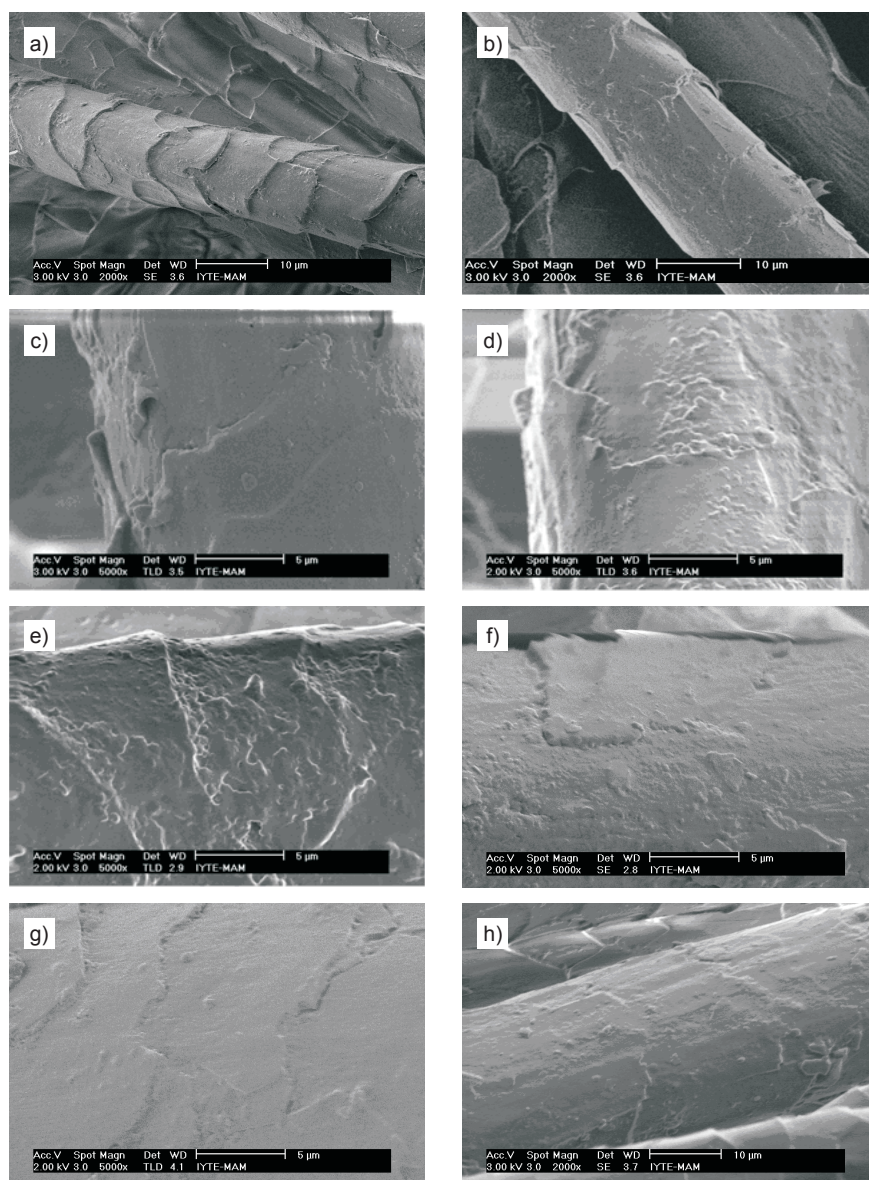
## Summary and conclusions

The main aim of this research was to treat wool fabrics with a combination of plasma, enzyme and chitosan. The following conclusions were made from the results of our experiments.

Plasma, enzyme and chitosan modify the wool surface in different manners. While plasma has etching effect, enzymatic treatment has hydrolytic effect. On the other hand chitosan, as an additive material, covers the surface without any degradation.

Plasma, enzyme and chitosan and combinations of these treatments increase the dyeing efficiency of wool fabrics. The plasma + enzyme + chitosan combination gave the best *K/S* values. Since plasma pretreatment modifies the surface, enzymatic treatment can remove the hydrophobic layer of wool more efficiently, which makes dye migration higher after the application of chitosan.

Significant changes were not observed in then fastness properties of the treated fabrics.



**Figure 5.** SEM images of untreated (a) and treated: enzyme (b), enzyme+chitosan (c), chitosan (d), plasma (e), plasma+chitosan (f), plasma+enzyme (g), and plasma+enzyme+chitosan (h) wool samples.

The plasma + enzyme + chitosan combination showed the highest resistance to shrinkage, as stated in the SEM analysis. The surface became so smooth that the shrinkage ratio decreased considerably.

When we evaluate the bursting strength of modified surfaces we came to the conclusion that chitosan treated ones have the highest value because of the covering effect of chitosan. Plasma treatment has no significant effect on bursting strength, unlike enzymatic treatment. Fabric bursting strength is negatively affected by blank enzymatic treatment. The etching effect of plasma treatment makes enzymatic treatment much more effective, which causes a reduction in strength. On the other hand, during the post-treatment

of chitosan, a clear protective effect on the bursting strength can be observed. In other words, the loss of bursting strength after plasma and enzymatic treatment is recovered, to a certain degree, by chitosan treatment.

A new ecological wool process is proposed applying enzyme and biopolymer chitosan to wool previously subjected to atmospheric plasma treatment. This process is attractive since there is no serious environmental issue involved (contamination of wastewater with absorbable organic chlorides) as in the Chlorine-Hercosett process, for example, which is often used today. These processes can be used in any combination according to desired effects.



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