

USAGE OF COMMERCIAL CELLULASES IN BIOPOLISHING OF VISCOSE FABRICS

VİSKON KUMAŞLARIN BİO-PARLATILMASINDA TİCARİ SELÜLAZ ENZİMLERİNİN KULLANIMI

M. İbrahim BAHTİYARİ
Erciyes University
Textile Engineering Department
e-mail:ibahtiyari@yahoo.com

Kerim DURAN
Ege University
Textile Engineering Department

Ayşegül EKMEKÇİ KÖRLÜ
Ege University
Textile Engineering Department

Seher PERİNCEK
Ege University
Emel Akın Vocation School

ÖZET

Uzun süreden beri tekstil terbiyesinde kullanılan selülozlar üzerine yoğun araştırmalar mevcuttur. Selülozların pamuk, jüt, keten rami ve diğer selüloz esaslı lifleri hidrolize ettiği ve temiz parlak tekstil yüzeylerinin elde edilmesini sağladıkları iyi bilinmektedir. Selüloz enzimi ayrıca denim giysilerde taşlanmış ve giyinmiş efekti için de kullanılmaktadır. Selülozların kullanım alanlarını arttırmak mümkündür. Fakat selülozların, viskon kumaşların bio-parlatılmasında kullanılmasında sorunlar mevcuttur. Bio-parlatma sonrası viskon kumaşlarda mukavemet kaybı gözlemlenirken kumaş yüzeyindeki pilling ve tüylülük yeterli miktarda azalmamaktadır. Bu ikilem nedeniyle farklı ticari enzimlerle farklı süre ve konsantrasyonlarda istatistiksel bir araştırma yapılmıştır. En iyi sonucun Sellobiohidrolaz (CBH) esaslı selüloz enzimi ile elde edildiği tespit edilmiştir.

Anahtar Kelimeler: Viskon, Selüloz, Bio-parlatma, Pilling eğilimi, Yüzey modifikasyonu, FTIR.

ABSTRACT

Cellulases which have been in use for a long time in textile finishing were studied deeply. It is well known that cellulases can hydrolyze cotton, jute, linen, ramie and other cellulose based fiber and ensure a clean polished textile surfaces. Moreover cellulase enzyme is also used for worn and stone washed effect in denim cloths. It is possible to expand its usages. But there is a draw back in the use of cellulases for biopolishing of viscose based fabrics. Although strength loss during biopolishing of viscose fabrics was evident, the pilling and fuzz of surface did not decreased sufficiently. Because of this dilemma, a statistical research was carried out with the use of different commercial cellulase enzymes in different time and concentration. It was found that the best results were obtained from the use of CBH based Cellulase.

Key Words: Viscose, Cellulase, Biopolishing, Pilling tendency, Surface modification, FTIR.

Received: 04.12.2008

Accepted: 16.10.2009

1. INTRODUCTION

Viscose fibers are regenerated fibers, a sub group of man-made fibers in which to guarantee the desired product properties each stage of processing and spinning requires close attention. The viscose process is a demanding process that requires continuous, year-long operation to prevent gelling of the system and to yield high quality products (1). The fibrillar structure of viscose fibers does not develop very well. Fibers from regenerated cellulose (cellulose II) have a semi-crystalline structure and, therefore, are composed of crystallites together with more or less disordered ("amorphous") regions.

The fibers are of low crystallinity but highly accessible to different media due to their mainly amorphous molecular arrangement and an extensive inner surface. These morphological characteristics enable a stronger swelling effect when compared to the other regenerated cellulose fibers (2, 3). Beside good properties in use, viscose fibers have some problems like fibrillation and pilling. This problem could be solved with the use of commercial cellulases in new generation regenerated cellulose fibers.

Cellulases have been widely used in the textile industry for modification of the surface and properties of cellulosic

fibers and fabrics in order to achieve a desired hand or surface effect. Cellulases are used to remove the fuzz or pills on the fiber or fabric surface which will decrease the pilling propensity of the fabric. There is a lot of study on the use of cellulases and some other parameters effected the bio-polishing (4-15).

Cellulases are multicomponent enzymes. There are three major types of cellulases secreted by *Trichoderma reesei*: Endoglucanases, 1,4-β-D-glucan 4-glucanohydrolases; Cellobio-hydrolases, 1,4-β-D-glucan cellobiohydrolases; Cellobiases, β-D-glucosidases

(16). *Trichoderma reesei* has at least six endoglucanases, two cellobiohydrolases, and two β -D-glucosidases (17, 18). These multicomponents act synergistically for the degradation of cellulose. They act specifically on 1,4- β -glycosidic bonds of the cellulose. Cellulase has two domains. One of them is the catalytic domain and the other is the cellulose binding domain. These two domains linked by a short linker peptide forms the intact bimodular enzyme (19, 20).

Important thermophilic microorganisms capable of degrading cellulose are *Clostridium thermocellum*, *Thermomonospora fusca*, *Thermoascus aurantiacus*, *Sporotrichum thermophile*, *Humicola insolens* and *Chaetomium thermophile*. *Clostridium thermocellum* differs from other cellulolytic microorganisms since it secretes all its cellulolytic enzymes in a protein complex called cellulosomes (21). But most extensive research about the cellulases has been done on aerobic fungi such as *Trichoderma koningii* and *T. reesei* (22-25).

Commercial cellulases currently used consist of multiple enzyme systems (cellobiohydrolases, endoglucanases, glucosidases) which hydrolyse cellulose in a synergistic way but, pilling problem is still in progress in viscose based textile surfaces. The predicted reason of this different effect of cellulases on different regenerated cellulosic fabrics resulted from different physical properties and macromolecular structure of them (26-28).

For explanations of the hydrolysis mechanism of cellulases during biopolishing of viscose fabrics there is some studies can be summarized like this: Kumar et al. use several man-made cellulosic fabrics-lyocell (Tencel), rayon (viscose) and cellulose acetate-were treated with cellulase. The treated fabrics and untreated controls were tested for surface fuzz removal, softening, pilling, weight and strength. The effect of cellulase on these different celluloses varied. On both rayon and lyocell, cellulase altered the handle and drapeability and removed surface fuzz. Cellulase also reduced the tendency of rayon to pill and reduced fibrillation of lyocell.

Cellulose acetate was minimally affected by cellulase under the selected test conditions. Cellulase treatment performed best on lyocell followed closely by rayon. The differences in the efficacy of cellulase on various fibers can be explained in part by the amount of non-cellulosic wood pulp-derived matter, the degree of polymerization, the extent and type of chemical modifications and the type and degree of crystalline fiber structure (29).

Carrillo et al. evaluated differences in enzymatic hydrolysis behaviour between lyocell and viscose type regenerated celluloses (viscose and modal) and found that the morphology and structure (crystallinity and orientation) of the different regenerated fibers studied lead to different rates of cellulosic degradation suitable for an industrial process of cellulose conversion the fibers proceeded. The maximum adsorption value was observed for a viscose. Comparing regenerated celluloses, the viscose fibers showed maximal hydrolysis with great differences mainly at extended hydrolysis times, where the adsorption step was less important (30).

A further investigation related to the issue was managed by Ciechańska et al., studied on the finishing of viscose-woven fabrics. They used a commercial enzyme of cellulase type Econase CE (Röhm Enzyme Finland Oy) experimental cellulases such as endoglucanase II (EGII), cellobiohydrolase I (CBHI) and cellulase enriched with EGII (Cell. F) from the *Trichoderma reesei* strain. Based on the results obtained, it was found that no significant changes in the molecular structure of modified viscose fabrics were caused after enzymatic treatment with either commercial or experimental cellulases. Enzymatic treatment

carried out in presence of cellulase type Econase CE allows smoothing of the surface of viscose fabric and the removal of impurities and individual loose fiber ends which protrude from the surface of the untreated fabric (31).

Since biopolishing of viscose fabrics with commercial cellulases is insufficient, the effect of commercial cellulases and optimization of biopolishing of viscose fabrics with different commercial cellulases were studied in this paper. Time of processes, enzyme concentrations and mechanical effects on viscose fabrics were investigated. Then the effect of different commercial enzymes was determined with evaluating the pilling tendency and bursting strength of fabrics.

2. MATERIALS AND METHODS

30/1 single jersey 100% viscose knitted fabric with 135 g/m² weight was used, the pretreatment processes of this fabric was realized on overflow of 5 kg (Doğuş trade mark) and tensionless dryer (Santex), then biopolishing was carried out in laboratory type dyeing machine Lab Dye HT 10. Pretreatment and bio-polishing of viscose fabrics were carried out in accordance with the test plan (figure 1) and the recipes (table 1).

Bio-polishing of viscose fabrics was performed at 55°C with different commercial cellulases notified in Table 2. pH of the bath differed according to the optimum application conditions of the enzyme used. For termination of bio-polishing, process temperature was raised to 95°C for 10 min.

Afterwards, fabrics were dyed with 1 % Cibacron B Blue FN G (Cl: Rea. Blue 204) and Cibacron Blue H GN (Cl: Rea. Blue 266) in a laboratory type

Table 1. Pretreatment and Bio-polishing recipes used for the trials

| Bleaching | Bleach Clean Up | Rinsing | Bio-polishing |
|---|------------------------------|---------------|-----------------------|
| 3.0 g/l Na ₂ CO ₃ | 0.5 g/l catalase | | Cellulase Enzyme |
| 2.0 g/l H ₂ O ₂ | 0.3 g/l CH ₃ COOH | | pH:5 with Buffer acid |
| 0.5 g/l Stabilizer | | | pH:7 without buffer |
| 90 °C, 60 min | 55 °C, 30 min | 60 °C, 30 min | 55 °C, 60 min |
| LR 1/20 | LR 1/20 | LR 1/20 | LR 1/15 |

*Liquor ratio (L.R.) is a term used in textile processes and means the ratio of fabric weight to the liquor weight of the bath

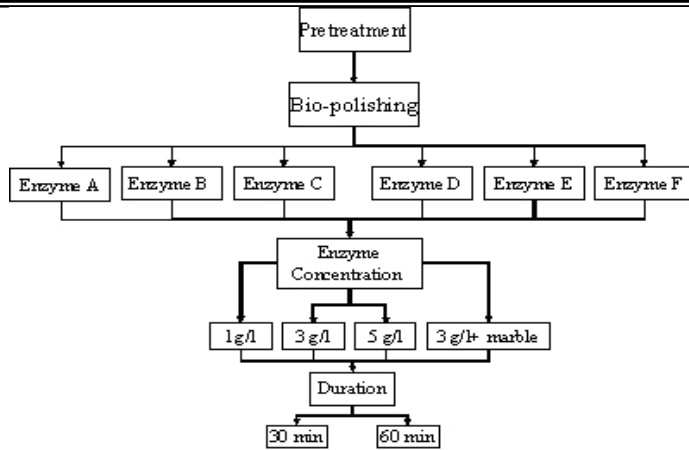


Figure 1. Test plan used for the pretreatment and printing of viscose fabrics.

Table 2. Enzymes used in Bio-polishing

| Enzyme | Structure/Type | Optimum Application |
|----------|--|----------------------------|
| Enzyme A | CBH Enriched Modified Acidic Cellulase | pH 4.5 – 5.5 50 – 60 °C |
| Enzyme B | Conventional Acidic Cellulase | pH 5 50-55 °C |
| Enzyme C | Endo Enriched Acidic Cellulase | pH 4.5 – 5.5 55 – 60 °C |
| Enzyme D | Endo Enriched Acidic Cellulase | pH 5 50-55 °C |
| Enzyme E | Conventional Acidic Cellulase (concentrated) | pH 4.5 – 5.5 55 – 60 °C |
| Enzyme F | Neutral Cellulase | pH 7 55 °C |



Figure 2. Reactive Dyeing Method

exhausting machine according to the dyeing method in Figure 2. Washing-off was performed in a home-type washing machine with the procedure “rinsing (cold), acidic rinsing (60°C), washing with soap (90°C), rinsing (80°C), rinsing (60°C) rinsing (cold)”. During the treatment processes, “soft mill water (permutit-water)” was used.

In order to evaluate the results, pilling tests were performed in Martindale 2000 pilling machine and the pilling results after 2000 rpm were evaluated with the aid of EMPA Standard SN 198525 K3. The strength losses were measured by James J Heal Bursting

strength machine (with 7.5cm² area and 30.5 mm diaphragm diameter). In the paper CPV (Change in Pilling Value) and BSL (% Bursting Strength Loss) terms were used during evaluation as mentioned in equation 1 and 2.

$$\text{Change in Pilling Value (CPV)} = [(\text{PV of untreated fabric}) - (\text{PV of treated Fabric})] \text{ (equation 1)}$$

$$\% \text{ Bursting Strength Loss (BSL)} = 100 - \left[\frac{\text{BS of treated Fabric}}{\text{BS of untreated Fabric}} * 100 \right] \text{ (equation 2)}$$

Moreover K/S values of dyed fabrics were determined with a Minolta 3600d

Model Spectrophotometer and expressed as K/S-FNG (for **Cibacron B. Blue FN G**) and K/S-HGN (for **Cibacron Blue H GN**)

FT-IR analysis of fabrics was carried out with Perkin Elmer Spectrum 100. The results (expressed as means/standard deviation) of all assays were compared using MANOVA, followed by a post hoc test (Duncan’s test) for “Alpha = 0.05”. In all statistical analyses, the software package SPSS 10.0 (Statistical Analysis Program) was used.

3. RESULTS AND DISCUSSIONS

In order to investigate the effect of such parameters; enzyme, treatment time and enzyme concentration on the change in pilling tendencies, bursting strength losses and color efficiencies, a statistical research was carried out. MANOVA presented in table 3 for CPV, BSL, K/S-FNG and K/S-HGN among 96 sample indicated that there were significant impact of Enzyme and Enzyme Concentration on CPV and BSL. Moreover the interaction between Enzyme*Treatment time, Enzyme*Concentration, Treatment time*Concentration, Enzyme* Treatment time*Concentration has also been examined but only Enzyme*Treatment time interaction was found significant in terms of BSL.

Moreover color efficiencies of bio-polished viscose fabrics were investigated too. Unlike the parameter “Treatment time”, the enzyme type and concentration was found important if the fabric will be dyed with Cibacron Blue FNG. On the other hand for the fabric to be dyed with Cibacron Blue H GN none of the parameters was shown significance as seen from Table 3. The interaction of the parameters was also examined but no importance was found too (Table 3). Except this, it can be summarized as only the enzyme type and the concentration of enzyme is important statistically (Table 3).

Table 3. MANOVA Tests of Between-Subjects Effects

| Source | Dependent Variable | Type III Sum of Squares | df | Mean Square | F | Sig. |
|---|--------------------|-------------------------|----|-------------|--------|-------|
| Treatment time | CPV | 2.296E-02 | 1 | 2.296E-02 | 0.367 | 0.547 |
| | BSL | 0.653 | 1 | 0.653 | 0.102 | 0.751 |
| | K/S-FNG | 1.972E-02 | 1 | 1.972E-02 | 0.081 | 0.777 |
| | K/S-HGN | 1.062E-02 | 1 | 1.062E-02 | 0.047 | 0.829 |
| Concentration | CPV | 0.913 | 3 | 0.304 | 4.870 | 0.005 |
| | BSL | 88.335 | 3 | 29.445 | 4.582 | 0.007 |
| | K/S-FNG | 4.921 | 3 | 1.640 | 6.766 | 0.001 |
| | K/S-HGN | 0.942 | 3 | 0.314 | 1.396 | 0.256 |
| Enzyme | CPV | 0.807 | 5 | 0.161 | 2.582 | 0.038 |
| | BSL | 446.644 | 5 | 89.329 | 13.902 | 0.000 |
| | K/S-FNG | 21.257 | 5 | 4.251 | 17.537 | 0.000 |
| | K/S-HGN | 2.455 | 5 | 0.491 | 2.183 | 0.072 |
| Treatment time * Concentration | CPV | 4.299E-02 | 3 | 1.433E-02 | 0.229 | 0.876 |
| | BSL | 8.031 | 3 | 2.677 | 0.417 | 0.742 |
| | K/S-FNG | 0.262 | 3 | 8.719E-02 | 0.360 | 0.782 |
| | K/S-HGN | 5.248E-02 | 3 | 1.749E-02 | 0.078 | 0.972 |
| Treatment time * Enzyme | CPV | 0.180 | 5 | 3.594E-02 | 0.575 | 0.719 |
| | BSL | 84.417 | 5 | 16.883 | 2.628 | 0.036 |
| | K/S-FNG | 0.584 | 5 | 0.117 | 0.481 | 0.788 |
| | K/S-HGN | 0.899 | 5 | 0.180 | 0.800 | 0.555 |
| Concentration * Enzyme | CPV | 0.439 | 15 | 2.928E-02 | 0.468 | 0.945 |
| | BSL | 105.583 | 15 | 7.039 | 1.095 | 0.386 |
| | K/S-FNG | 4.002 | 15 | 0.267 | 1.100 | 0.382 |
| | K/S-HGN | 0.589 | 15 | 3.929E-02 | 0.175 | 1.000 |
| Treatment time * Concentration * Enzyme | CPV | 0.939 | 15 | 6.263E-02 | 1.002 | 0.469 |
| | BSL | 85.416 | 15 | 5.694 | 0.886 | 0.583 |
| | K/S-FNG | 2.735 | 15 | 0.182 | 0.752 | 0.720 |
| | K/S-HGN | 0.450 | 15 | 3.003E-02 | 0.134 | 1.000 |

Abbreviations: Change in Pilling Value (CPV); Bursting Strength Loss (BSL)

Table 4. Duncan Post Hoc test of the Enzyme type in terms of CPV and BSL

| | Subset | | | | | |
|----------|--------|--------|---------|---------|---------|--|
| | CPV | | | BSL | | |
| | 1 | 2 | 1 | 2 | 3 | |
| Enzyme F | 0.2969 | | | 14.0000 | | |
| Enzyme B | 0.3750 | 0.3750 | | | 17.1875 | |
| Enzyme C | 0.4063 | 0.4063 | 10.4375 | | | |
| Enzyme D | 0.4833 | 0.4833 | | 13.8000 | | |
| Enzyme E | | 0.5469 | | | 16.1250 | |
| Enzyme A | | 0.5469 | | 15.4375 | 15.4375 | |
| Sig. | 0.060 | 0.090 | 1.000 | 0.092 | 0.072 | |

Abbreviations: Change in Pilling Value (CPV); Bursting Strength Loss (BSL)

Table 5. Duncan Post Hoc test of the Enzyme type in terms of K/S-FNG and K/S-HGN

| | Subset | | | | | |
|----------|---------|---------|---------|---------|---------|---------|
| | K/S-FNG | | | | K/S-HGN | |
| | 1 | 2 | 3 | 4 | 1 | 2 |
| Enzyme F | 12.4588 | | | | 9.7913 | |
| Enzyme C | 12.7569 | 12.7569 | | | 9.8713 | 9.8713 |
| Enzyme D | | 12.9233 | | | 9.8733 | 9.8733 |
| Enzyme A | | | 13.2806 | | 10.1375 | 10.1375 |
| Enzyme B | | | | 13.6756 | | 10.1712 |
| Enzyme E | | | | 13.7469 | | 10.1762 |
| Sig. | 0.095 | 0.346 | 1.000 | 0.686 | 0.065 | 0.113 |

Abbreviations: Change in Pilling Value (CPV); Bursting Strength Loss (BSL)

➤ Enzyme Type

As seen from the table 4 the enzyme type is of great importance in terms of CPV. So during bio-polishing the cellulase must be carefully chosen. It is obvious that use of neutral cellulases have got slight effect on decreasing the fuzzy of the fabrics. But with the use of enzyme E and A the pilling tendency reduced nearly 0.5 point in average.

On the other hand the bursting strength losses of fabrics showed that with the use of Endo Enriched modified enzymes which are Enzyme C and Enzyme D, the BSL was the lowest (nearly 10-14) (Table 4). Interestingly Enzyme F caused slight CPV but high BSL. This is related with why neutral cellulase is used in denim washing. They act aggressively and caused the loss of strength while the fuzzy of surface did not change.

One parameter that can affect the dyeability is performing a biopolishing process. In other words the enzyme type used in biopolishing can also change the dyeability of the fabrics. Hence in table 5 the changes in color efficiencies were analyzed with Duncan Post Hoc test.

The dyeability of cellulosic fibers is related with the morphological structure of the fiber, the amorphous structure of the fiber is of great importance on the dye uptake. Since cellulase is more effective in the amorphous cellulose of viscose depending on the enzyme component, color efficiencies after dyeing were effected by the enzyme type used in biopolishing. Interestingly conventional acidic cellulases (Enzyme B and E) were especially improved the dye ability of viscose among the tested cellulase enzymes. This change due to the biopolishing is especially evident after dyeing with FNG type reactive dyes which have high reactivity. However during the dyeing with low reactive dyestuffs, the medium temperature must be high (as recommended in CIBA catalog). As a result of this high temperature, swelling of the fabrics finally the dye take up increases. So the effect of biopolishing before dyeing does not

Table 6. Duncan Post Hoc test of the Enzyme Concentration in terms of CPV and BSL

| | Subset | | | |
|--------------------|--------|--------|---------|---------|
| | CPV | | BSL | |
| | 1 | 2 | 1 | 2 |
| 3 g/l + metal ball | 0.2917 | | 14.2083 | 14.2083 |
| 1 g/l | 0.4271 | 0.4271 | 13.0417 | |
| 3 g/l | | 0.5000 | | 15.4783 |
| 5 g/l | | 0.5521 | | 15.3333 |
| Sig. | 0.068 | 0.110 | 0.120 | 0.109 |

Abbreviations: Change in Pilling Value (CPV); Bursting Strength Loss (BSL)

Table 7. Duncan Post Hoc test of the Enzyme Concentration in terms of K/S-FNG and K/S-HGN

| | Subset | | | |
|--------------------|---------|---------|---------|---------|
| | K/S-FNG | | K/S-HGN | |
| | 1 | 2 | 3 | 1 |
| 1 g/l | 12.8054 | | | 9.8792 |
| 3 g/l | | 13.1291 | | 9.9739 |
| 3 g/l + metal ball | | 13.1963 | 13.1963 | 10.0108 |
| 5 g/l | | | 13.4392 | 10.1542 |
| Sig. | 1.000 | 0.641 | 0.096 | 0.073 |

Abbreviations: Change in Pilling Value (CPV); Bursting Strength Loss (BSL)

Table 8. Characteristic absorption bands of regenerated cellulose fibers.

| Wavenumber (cm ⁻¹) | Assignment |
|--------------------------------|--------------------------------------|
| 3326 | -OH stretching |
| 2890 | -CH stretching |
| 1650 | -OH of water absorbed from cellulose |
| 1420 | CH ₂ symmetric bending |
| 1365 | CH bending |
| 1200 | OH in plane bending |
| 1155 | C-O-C asymmetric stretching |
| 894 | Group C ₁ frequency |

provide a significant increase in color efficiencies.

➤ Enzyme Concentration

During the experiment all enzymes were used in 3 different concentrations and with one of these concentrations metal balls were used for mechanical effect. Hence it was tried to simulate a bio-polishing in textile treatment machine used in textile plants (overflow-jet). Moreover the enzyme concentration was combined with different treatment times (30-60 min).

With this experimental design, it was aimed to investigate the common belief on the use of enzyme concentration and efficiency of bio-polishing. It was found that with the increase in enzyme concentration the fuzzy of surface has reduced but this decrease in the fuzzy of the surface with the increasing concentration of cellulase enzyme was detected not so important. In other words, the statistical test found the result nearly similar in a group shown in table 6. However use of marbles for mechanical effect result less increase

in pilling values when compared the one treated with only enzymes. The reason of this is thought as with the use of marbles the efficiencies of enzymes hope to be increased however this mechanical effect cause some new fibrils and fibers come out from the yarn structure too. This caused improve in pilling tendency limited.

When the enzyme concentration was examined in terms of BSL it was found that the effect of 3 g/l and 5 g/l enzyme concentration has shown nearly same effect but 1 g/l enzyme caused less BSL. The metal balls found in the bio-polishing bath has shown higher BSL than treatment with 1 g/l enzyme but the addition of marbles decreased the loss of strength with the use of same enzyme concentration.

Interestingly, the increase in the enzyme concentration during biopolishing improved the dyeability of fabric unlike CPV and BSL. But as told before, this increase was found inconsiderable while dyeing was performed with HGN.

➤ FTIR

The infrared spectra of greige and treated viscose fibers were analyzed to examine the changes in crystalline and amorphous region content caused by enzymes. The infrared spectra of greige and treated viscose fibers are shown in Figure 3. Table 8 shows the most significant bands that were studied and analyzed, and their corresponding assignment (32,33).

In the FTIR spectral region studied, significant differences in intensity and shape could not be observed between the greige and treated samples. Table 9 shows the infrared crystallinity index results obtained for greige and treated viscose fibers. These indexes have been calculated by the infrared ratios proposed by Nelson and O'Connor, a_{1376}/a_{2902} (total crystalline index (TCI)) and a_{1420}/a_{893} (lateral order index (LOI)), and the doublet a_{1278}/a_{1263} proposed by Carrillo-Colom (30,31).

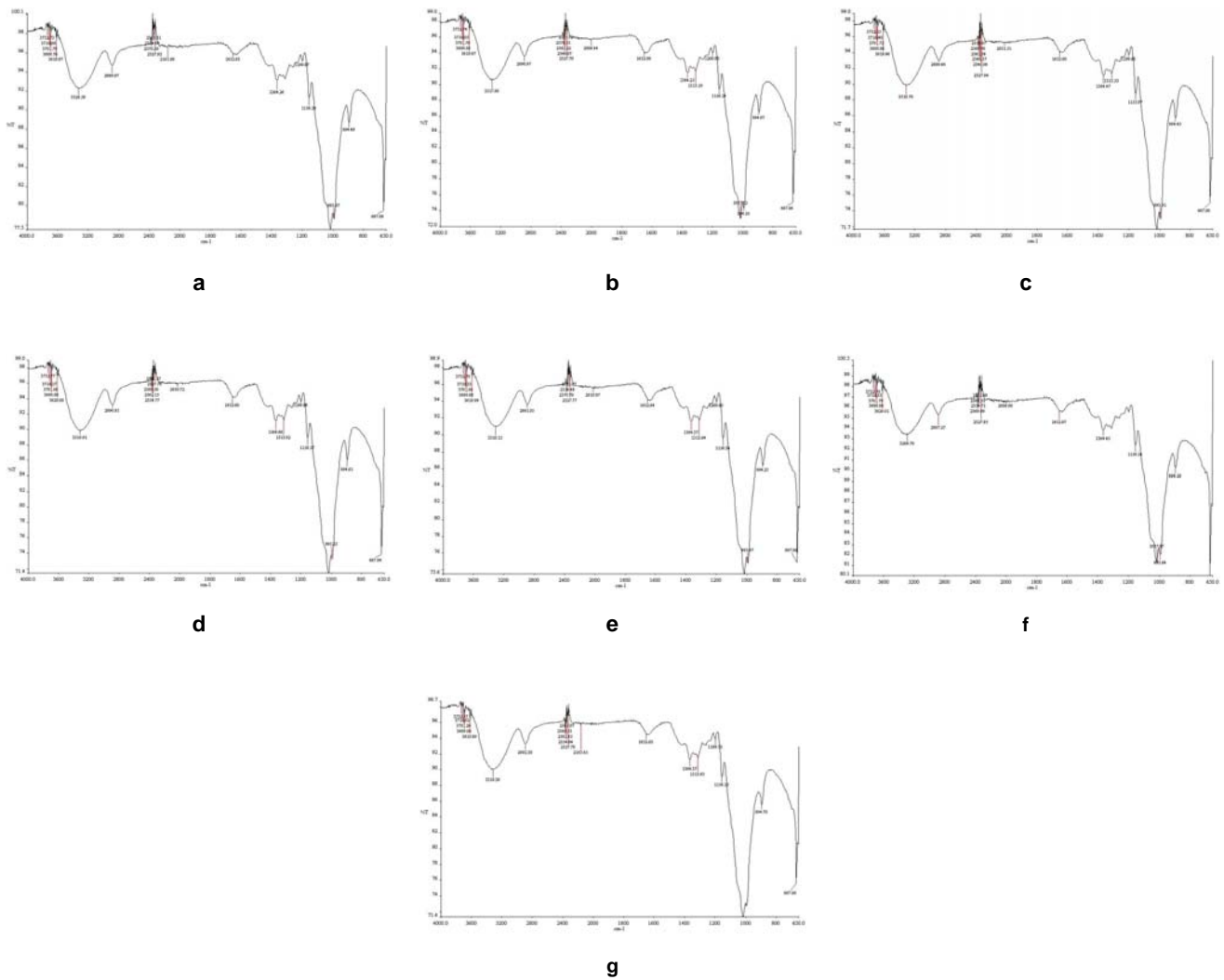


Figure 3. Infrared spectra of greige and treated viscose fibers (a. untreated, b. 5g/l Enzyme E, c. 5g/l Enzyme C, d. 5g/l Enzyme A, e. 5g/l Enzyme B, f. 5g/l Enzyme D, g. 5g/l Enzyme F)

Table 9. Infrared crystallinity indexes data on viscose after enzymatic treatments

| Enzyme Type | TCI (1376/2902) | LOI (1420/893) | CCI (1278/1263) |
|-------------|-----------------|----------------|-----------------|
| Enzyme A | 1.00 | 0.92 | 0.996 |
| Enzyme B | 1.02 | 0.93 | 0.995 |
| Enzyme C | 1.02 | 0.92 | 0.998 |
| Enzyme D | 1.00 | 0.95 | 0.998 |
| Enzyme E | 1.02 | 0.92 | 0.998 |
| Enzyme F | 1.02 | 0.92 | 0.997 |

Abbreviations: Total crystalline index (TCI), lateral order index (LOI)

Treated viscose fibers have a similar crystallinity. This is confirmed by the respective FTIR 1420/893 and 1278/1263 ratios. From the Table 9, it can be noted that the TCI, LOI and CCI shows no significant changes for any of the samples.

4. CONCLUSION

It was found that in industrial applications of textile processes although cellulases have wide use commercially in different name and structure, they were not effective in bio-polishing of viscose fabrics. For the

study, 6 different commercial cellulases were obtained and with the use of them the decrease in pilling tendencies and bursting strength losses was determined. As a result of all experimental processes, pilling values did not increase more than 0.75

point while Bursting Strength Loss (BSL) has increased (the untreated fabrics's pilling value was 2). However, some interesting results were obtained with the use of CBH (cellobiohydrolase) based commercial cellulase; Pilling Value (PV) was significantly increased in terms of statistical analysis. The neutral cellulase was ineffective but aggressive as a result they ensured high BSL. Moreover it was found that Endo Enriched cellulase enzymes have attacked cellulose more recessive so the BSL of the fabrics treated with these enzymes has shown less increase. The common belief that with an increase in treatment time the effect of bio-polishing getting well is

found inadequate from MANOVA results of tested conditions. That is to say the 30 min of treatment time is sufficient; increasing the time to 60 min. did not ensure a significant change in the results. On the other hand the concentration of enzyme has of importance for the efficiency of bio-polishing. The increase in concentration is why the increase in BSL and PV. But the increase in PV is slightly less than the one in the BSL. In this study simulating the mechanical effect with use of metal balls did not ensure any difference of the efficiency of bio-polishing. It may be caused by insufficient mechanical simulation. In order to investigate the effect of additives found in commercial

cellulases, 2 same structured enzymes (Enzyme E and B) with different concentration and additives were used. From the results obtained it was found that the use of Enzyme B caused more BSL although it has less activity than enzyme E. Because of additives like wetting agent found enzyme B provides the hydrolysis of cellulose faster due to better penetration. However unlike BSL enzyme B ensured less Change in Pilling Value (CPV) than Enzyme E.

Moreover, the crystallinity change owing to the biopolishing was studied and no considerable change was observed.

REFERENCES

1. Kroschwitz, I. J., *Polymers; Fibers and Textiles, A Compendium* New York: Wiley Interscience Publication; 1990. p. 746-757. 1990
2. Müller M., Riekel C., Vuong R., 2000, "Skin/core micro-structure in viscose rayon fibres analysed by X-ray microbeam and electron diffraction mapping", *Polymer*, Vol. 41, pp. 2627-2632.
3. Smole, M. S., Persin, Z., Kreze, 2003, "X-ray Study of Pre-treated Regenerated Cellulose Fibers", *Materials Research Innovations*, Vol. 7, pp. 275-282.
4. Cavaco-Paulo, A., 1998, "Mechanism of cellulase action in textile processes", *Carbohydrate Polymers*, Vol. 37, pp. 273-277.
5. Cavaco-Paulo A., Almeida L., 1996, "Cellulase Activities And Finishing Effects", *Textile Chemist and Colorist*, Vol. 28, pp.28-32.
6. Cavaco-Paulo A., Almeida L., 1994, "Cellulase hydrolysis of cotton cellulose: the effects of mechanical action, enzyme concentration and dyed substrates", *Biocatalysis*, Vol. 10, pp. 353-360.
7. Heikinheimo L., Miettinen-Oinonen A., Cavaco-Paulo A., 2003, "Effect of Purified Trichoderma Reesei Cellulases on Formation of Cotton Powder from Cotton Fabric", *Journal of Applied Polymer Science*, Vol. 90, pp. 1917-1922.
8. Morgado, J. Cavaco-Paulo, A. Rousselle, M.-A, 2000, "Enzymatic Treatment of Lyocell-Clarification of Depilling Mechanisms", *Textile Research Journal*, Vol. 70, Issue 8, pp. 696-699.
9. Heikinheimo L., Buchert J., Miettinen-Oinonen A., 2000, "Treating Denim Fabrics with Trichoderma Reesei Cellulases", *Textile Research Journal*, Vol. 70, Issue 11, pp.969-973.
10. Cortez J.M., Ellis J., Bishop D.P., 2001, "Cellulase finishing of woven, cotton fabrics in jet and winch machines", *Journal of Biotechnology*, Vol. 89, pp.239-245.
11. Ibrahim N. A., Fahmy H. M., Hassan T. M., 2005, "Effect of cellulase treatment on the extent of post-finishing and dyeing of cotton fabrics", *Journal of Materials Processing Technology*, Vol. 160, pp.99-106.
12. Cavaco-Paulo A., Almeida L., Bishop D., 1998, "Hydrolysis of Cotton Cellulose by Engineered Cellulases from Trichoderma Reesei", *Textile Research Journal*, Vol. 68, Issue 4, pp. 273-80.
13. Körlü A. E., Duran K., Bahtiyari M. İ., 2008, "Selülaz Enziminin Selülozik Esaslı Kumaşlar Üzerine Etkisi", *Tekstil ve Konfeksiyon*, Yıl:18(1), s:35-40.
14. Duran K., Ayaz Ö., 2000, "Selülazların Rejenere Selüloz Liflerinde Kullanımıyla Alternatif Terbiye Prosesleri" *Tekstil ve Konfeksiyon*, Sayı: 1-2, s: 34-37
15. Duran K., Öneş M., 1994, "Tekstil Terbiyesinde Enzimler ve Kullanımı", *Tekstil ve Konfeksiyon*, Sayı: 4 s: 318-328.
16. Ng, T.B., 2004, Peptides and proteins from fungi, *Peptides*, Vol. 25, pp.1055-1073.
17. Bhat, M.K., Bhat S., 1997, "Cellulose degrading enzymes and their potential industrial applications", *Biotechnology Advances*, Vol. 15, pp. 583-620.
18. Heikinheimo L., 2002, "Trichoderma reesei cellulases in processing of cotton", VTT Biotechnology, Espoo., VTT Publications No: 483, pp.11-61.
19. Sandgren M., Stahlberg J. and Mitchinson C., 2005, "Structural and biochemical studies of GH family 12 cellulases: improved thermal stability, and ligand complexes", *Progress in Biophysics and Molecular Biology*, Vol. 89, pp.246-291.
20. Kleywegt, G.J., Zou J. Y., Divne C., Davies G. J., 1997, "The Crystal Structure of the Catalytic Core Domain of Endoglucanase I from Trichoderma reesei at 3.6 Å Resolution, and a Comparison with Related Enzymes", *Journal of Molecular Biology*, Vol. 272, pp.383-397.
21. Nemeth A., Kamondi S., Szilagyı A, 2002, "Increasing the thermal stability of cellulase C using rules learned from thermophilic proteins: a pilot study", *Biophysical Chemistry*, Vol.132, pp.229-241.

22. Halliwell, G. and Vincent, R., 1981, "The action of cellulose and its derivatives of a purified 1,4-β-glucanase from *Trichoderma koningii*", *Biochem. J.*, Vol. 199, pp.409-417.
23. Miettinen-Oinonen, A., Paloheimo, M., Lantto, R., Suominen, P., 2005, "Enhanced production of cellobiohydrolases in *Trichoderma reesei* and evaluation of the new preparations in biofinishing of cotton", *Journal of Biotechnology*, Vol. 116, pp. 305-317.
24. Liming, X., Xueliang, S., 2004, "High-yield cellulase production by *Trichoderma reesei* ZU-02 on corn cob residue", *Bioresource Technology*, Vol. 91, pp. 259-262.
25. Medve, J., Karlsson, J., Lee, D., 1998, "Hydrolysis of Microcrystalline Cellulose by Cellobiohydrolase I and Endoglucanase II from *Trichoderma reesei*: Adsorption, Sugar Production Pattern, and Synergism of the Enzymes", *Biotechnology and Bioengineering*, Vol. 59, Issue 5, pp. 621-634.
26. Bredereck K., 1998, "Lyocellfasern: Struktur, Eigenschaften und Veredlungsverhalten" Proceedings, *VIIIth International Izmir Textile and Apparel Symposium*, Izmir/Turkey,. pp. 408-416.
27. Bahtiyari, M. İ., 2005, "Preventing Pilling Problem with Different Type of Enzymes In Viscose Fabrics And Comparing The Obtained Effects", *MSc Thesis*, Ege University, Turkey.
28. Körlü A.E., Taralp A., Duran K., Sezerman U., Bayram G., Bahtiyari M.İ., 2005, "Effect of Crosslinked and Commercial Cellulases on Bio-polishing of Viscose Based Knitted Fabrics", *World Textile Conference 5th Autex Conference*, Portoroz Slovenia, pp.584-589.
29. Kumar A., Pintail C. and Lepola M., 1994, "Enzymatic treatment of man-made cellulosic fabrics", *Text Chem. Color.*, Vol. 26, Issue 10, pp. 25-28.
30. Carrillo F., Colom X., Lo'pez-Mesas M., 2003, "Cellulase Processing of Lyocell and Viscose Type Fibres: Kinetics Parameters", *Process Biochemistry*, Vol. 39, Issue 2, pp.257-261.
31. Ciechańska D., Struszczyk H., Miettinen-Oinonen A., 2002, "Enzymatic Treatment of Viscose Fibres Based Woven Fabric", *Fibres & Textiles in Eastern Europe*, Vol. 10 pp.60- 63.
32. Colom X., Carrillo F., 2002, "Crystallinity changes in lyocell and viscose-type fibres by caustic treatment", *European Polymer Journal*, Vol. 38, pp. 2225-2230.
33. Carrillo F., Colom X., Sunol J.J., 2004, "Structural FTIR analysis and thermal characterisation of lyocell and viscose-type fibres", *European Polymer Journal*, Vol. 40, pp. 2229-2234.

Bu araştırma, Bilim Kurulumuz tarafından incelendikten sonra, oylama ile saptanan iki hakemin görüşüne sunulmuştur. Her iki hakem yaptıkları incelemeler sonucunda araştırmanın bilimselliği ve sunumu olarak "**Hakem Onaylı Araştırma**" vasfıyla yayımlanabileceğine karar vermişlerdir.

ARAMİT NONWOVENLAR

Aramit nonwovenların sağladığı avantajlar; yüksek kimyasal dayanıklılık, yüksek uzun süreli sıcaklık dayanımı, tutuşmazlık özellikleri, yüksek çekme mukavemeti, yüksek tenasite, düşük uzamadır. Meta aramitler, Nomex ve Conex, para-aramitler Kevlar ve Twaron'dır.

Para-aramitler uzay ve havacılık uygulamalarında, özellikle zehirli gazlardan korunma, tutuşmazlık ve ısı dayanıklılık özelliklerinden dolayı kullanılır. Diğer kullanım amaçları çelik endüstrisinde koruyucu giyimde ve transformatör ve motorlarda elektrik yalıtımında kullanılır. Aramitler ayrıca spor malzemeleri, hatlar, frenler ve kavramalarda kullanılır.

Tutuşmaz özellikli nonwovenlar, endüstride ve döşeme sanayinde kullanılır. Ayrıca ağır gramajlı aramit nonwovenlar kurşun geçirmez yelekler de kullanılır. Sıcak gaz filtrasyonunda da meta-aramitler çok kullanılırlar. (İNTERNET)