Study of different methods to improve wool fabrics properties

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1.0 Introduction The unshrinkable wool obtention is an important goal that allows the production of fabrics that apart of having the very appreciate wool properties, doesn't present the natural tendency to shrinkage by felting.

The different processes to obtain unshrinkable wool, are a good prove of the above mentioned goal interest. All the procedures to treat wool with energic oxidant agents, like clorine, perform an agressive attack to it, and are very risky for the people that handle them. The presence of these oxidant agents in the effluents become a serious problem to the environment.

Similar effects to those obtained on wool fibers subjected to an oxidative treatment (chlorination, hydrogen peroxide, etc.) are achieved when proteolytic enzymes are applied on wool fabrics to obtain good handle properties and improve dyeing yield (1) (2) (3). This enzymatic treatment partially reduces the felting propensity of wool during aqueous washing (4). The Bioengineering Department from the Faculty of Engineering in Uruguay produced an enzymatic extract (5), with the characteristics mentioned above. It was applied on wool fabrics and properties as breaking load, shrinkage in automatic laundering, colourfastness to washing, Cielab colour differences and final bath exhaustion were evaluated on treated and non treated fabrics.

In order to attain the required levels of IWS Test Method 31 (the shrinkage area at the second wash - cycle has to be less than 8 %), different treatments were carried out over

enzymatical and non enzymatical treated wool, including an oxidative process, and a chitosan treatment (6).

2.0 Materials and methods

2.1 Microorganism and enzymatic extract production

2.1.1 Microorganism and their maintenance

The trials were performed with an indigenous strain of *Bacillus* sp. selected in a screening program (7) (8) carried out by Bioengineering Department, Faculty of Engineering Universidad de la República, Uruguay. The strain was maintained on nutrient agar slant and subcultured every 3 months and stored at 4°C .

2.1.2 Growth medium and growth conditions

2.1.2.1 Medium

Inoculation medium: The following inoculation medium was used containing (gL^{-1}) : glucose, 5; nutrient broth, 8; yeast extract, 3; K_2HPO_4, 0.7; KH_2PO_4, 0.3; $MnCl_2.4H_2O$, 0.1; $CaCl_2.2H_2O$, 0.3; $MgSO_4.7H_2O$, 0.01.

Fermentation medium: The composition of the culture medium was (gL^{-1}): glucose, 13; nutrient broth, 8; yeast extract, 3; K_2HPO_4, 0.7; KH_2PO_4, 0.3; $CaCl_2.2H_2O$, 0.3; $MgSO_4.7H_2O$, 0.01. In all cases media were first adjusted to pH 7 by 0.1 N HCl or 0.1 N NaOH and then sterilized by autoclaving for 15 min at 121 °C.

2.1.2.2 Shake flask experiments

Fully slants of 12 h old *Bacillus* sp. incubated at 37 °C were scrapped off and suspended in 10 mL of sterile saline solution. The cell suspension was shaken thoroughly to break up the aggregates and used for building up of inoculum in 1000 mL Erlenmeyer flasks containing 100 mL of inoculation medium. The inoculated flasks were incubated on an orbital

shaker at 300 rpm and 37 °C for 12 hours. Inoculum was used to inoculate Erlenmeyers flasks of 2000 mL with 200 mL of fermentation medium. Culture temperature was 37 °C, shaking at 300 rpm. The concentration of cells at the beginning of fermentation was 0.2 gL^{-1}. Biomass was measured turbidimetrically at 650 nm..

2.1.3 Proteolytic activity measurement

Proteolytic activity was mesured as a coagulant activity by the Arima method (9). An activity of 400 Units (400 U) is the amount of enzyme which coagulates 5 mL of 10 % solution of skimmed milk powder in $CaCl_2$ 0.01 M, at 35 °C in 60 seconds.

2.1.4 Enzymatic extracts

The enzymatic extracts used were the ones identified as PK2- i and CP. The first one is the extract obtained with the fermentation media detailed in 2.1.2, used with three different proteolytic activities:350 U/mL (PK2-1), 436 U/mL (PK2-2) and 1422 U/mL (PK2-3). CP is a commercial product of common use in wool fabrics in Uruguay, to give them unshrinkable properties. The comercial complex CP, was used in 3%, (80 U/mL), this condition was recommended by the seller.

2.2 Textil process

2.2.1 Wool fabric and top

- Wool fabric with the following characteristics was used for the different tests:warp yarns (ends) in fabric 3200; linear density 1/21; tpm 340 S; 100 % wool; 23 μ
- filling yarns (picks) in fabric 1700; linear density 1/21; tpm 340 S; 100 % wool; 23 μ

- mass per unit area: 300 g/m^2; width: 150 cm; design: twill 2/1

2.2.2 Dyes

Drimalan F dyes: Red Brillant Drimalan FB, Blue Brillant Drimalan FB.

2.2.3 Dyeing of treated wool

Due to the modification of the fibre surface, shrink resistant and antifelt treated wool exhibits a change in the dyeing behaviuor with respect to the untreated wool. Since the dye begins in the lower temperature range and builds up more rapidly in the heating phase, the dyeing methods must be adapted to these conditions in order to avoid unlevelness.

Wool treated in this way also exhibits much poorer wetfastness properties and particularly in medium to dark shades the requiered fastness properties can no longer be met with acid levelling dyes. For this reason, a careful selection of dyes is essential (10). The method used was *Optilan* with a liquid ratio of 1/100:

a)starting bath temperature 40 °C

addition: 1 g/L sodium acetate, X % Drimalan F dyes, 1 % of Lyogeno FN liquid (amphoteric levelling agent, retards dyeing rate of Drimalan F dyes on wool treated), pH = 5 acetic acid.

b) increased temperature in 40 minutes to 85 °C

c) maintain 30 - 60 minutes at 85 °C The fabric was dyed in four different shades (2 % and 0.2 % Blue Brillant Drimalan FB, and 2 % and 0.2 % Red Brillant Drimalan FB).

2.2.4 Final exhaustion

The final bath exhaustion is determined at several wave lenghts, by using a spectrophotometer Unicam UV2.

2.2.5 Colour differences measurement

Test methods ITR TEX T023, Textiles Department (LATU)

Equipment : Elrepho 2000; Illuminat: day light D 65

Observer: 10 ° ; Spot: 12mm

The colour differences and tolerances between the references (fabric or top untreated) and the treated wool (fabric or top) was expressed as total difference (ΔE) using the CIE 1976 L*a*b* formula.

2.2.6 Standard test methods for breaking load (raveled strip)

Test methods: Protocol TEX 004 Textiles Department (LATU) (based on ASTM D 5035-95).

The expanded uncertainty of the methods is ± 2.82 kgf/25 mm.

2.2.7 Colourfastness to washing domestic and laundering

Test method: Protocol TEX 005 Textiles Department (LATU) (based on AATCC 61 1996) Multifiber: No. 10 Testfabric. Evaluate the colour change of test specimens using the Gray Scale for Staining ISO R105/1 Part 3. Uncertainty: one grade in the whole scale, uncertainty type B (interlaboratory).

2.2.8 Dimensional changes in automatic home laundering of woven and knit fabrics

Test method: ITR TEX E029 Textiles Department (LATU) based on AATCC 135

Equipment: automatic home laundering Electrolux.

Temperature : 40 °C; Laundering cycle : 3 (no wool cycle)

Drying conditions: screen dry; Pressing: Singer steam press at 148 °C.

2.2.9 Fiber identification

Test method: Protocol Tex 406 a Textiles Department (LATU) (based on AATCC 20 A - 1995). Equipment: Microscope of polarized light Leitz Laborlux 12 Pol (T13) with photografic equipment.

2.3 Improvement process

2.3.1 Enzymatic treatment

The enzymatic treatment was done in a jigger (John Jeffreys). It was treated 8 m of fabric, with 1.0 L bath (Liquid ratio = 1/3). The temperature was 60 °C, and the treatment last 60 minutes. It was used the enzymatic complex PK2-1,PK2-2, PK2-3 and CP.

After the enzymatic treatment, one rinse at 80 °C for 10 minutes was done.

The residual coagulant activity in the dye bath was under the limit of detection.

2.3.2 Oxidative treatment

33 % (w/v) H_2O_2 , 2 g/L $Na_4O_7P_2$, pH = 9.0, Liquid ratio = 1/30, Temperature = 70 'C

treatment time = 1 h

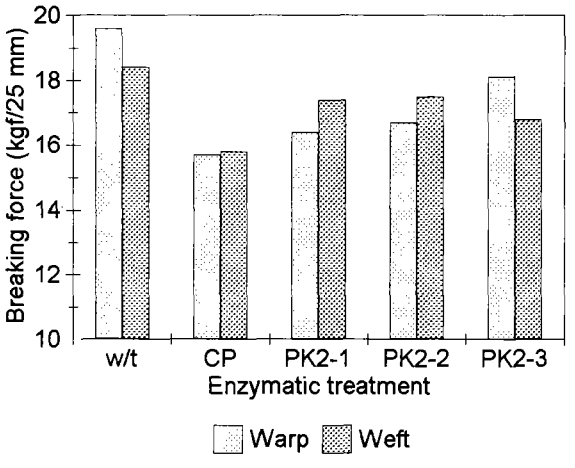
2.3.3 Chitosan treatment

0.3% (w/v) chitosan solution in acetic acid (4 g/L), Liquid ratio = 1/20, temperature = 25 'C

treatment time = 1 h

3.0 Results and discussion

3.1 Wool with enzymatic treatment only



3.1.1 Fiber tenacity

The tenacity was not reduced by the enzymatic treatments. The fiber tenacity was reduced only in 4% or less than the tenacity of wool fiber not treated.

3.1.2 Fabric breaking load (raveled strip)

The breaking load of the fabrics treated with the differents enzymatic procedures was determinated by the raveled strip method. The results (Figure 1) showed that the enzymatic treatment not modified the tensometric property. The lowering of the breaking force was of 4.0 kgf/25 mm, but the combined and expanded uncertainty of the method was 2.82 kgf/25 mm.

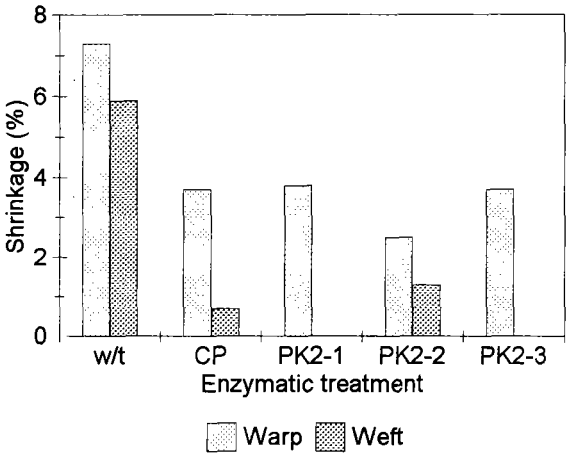


Figure 1 Study of the breaking force with PK2-1, PK2-2, PK2-3 and CP using the raveled strip method. (w/t - without enzymatic treatment)

3.1.3 Shrinkage in automatic laudering

The results are showed in Figure 2. The treatments were effective in reducing the shrinkage due to automatic home laundering (no wool cycle). This reduction was in the order of 65%. This improvement has no relation with the increased of the coagulant activity of the extract used.

Figure 2 Shrinkage of the fabric with the different enzymatic treatment PK2-1, PK2-2, PK2-3 and CP. (w/t - without enzymatic treatment)

3.1.4 Colourfastness to washing domestic and laundering

The fabric dyed in blue and in red, had colorfastness to staining 5 or 4-5 in all the fibers of the Multifiber No. 10, and for all the treatments.

3.1.5 Cielab Colour Differences The colour difference was higher in the pale shades (0.2 %). It was obtained DE 2.11 for pale blue with PK2-3 and 1.69 with CP. In some cases the yield with the comercial product was higher (DE 3.12 for red with CP and DE 2.81 with PK2-2). In other cases the yield improved when the coagulant activity increases (pale blue).

3.1.6 Dye bath exhaustion

The higher final exhaustion was obtained with the treated fabrics. Results are detailed in Table I.

Table I Final exhaustion of the dyeing bath

	istion (%)	
natic Extract		
w/t	75.2	93.2
CP	80.9	96.1
PK2-1	69.7	94.3
PK2-2	83.9	94.1
PK2-3	85.0	95.1

w/t: without treatment

3.2 Wool with enzymatic and chitosan treatment

3.2.1 Shrinkage on the fabrics treated by the enzymatic-chitosan process

The wool treated with the enzymatic product PK2-2 and followed an oxidative process and chitosan application is the only one which fulfill the IWS requirements for an unshrinkable wool (less than 8 % for the second wash with Wascator equipement). However, the handle obtained not fulfill the customers requirements. The results are shown in Table II.

Table II Shrinkage in Wascator

	ge (%)		
nt			
	ge (%)		
nt			

chit.			
chit.			
chit.			
	ge (%)		
nt			
. + chit.			
. + chit.			
oxid. + chit.			
oxid. + chit.			
oxid. + chit.			

Note: w/t: without enzymatic treatment **Enz.**: Enzymatic treatment
Enz. + Chitosan : Enzymatic treatment followed with chitosan treatment
Enz. + Oxid. + Chitosan: Enzymatic treatment followed with oxidative process followed with Chitosan treatment **1c, 2c, 3c** : Wascator cycles

4.0 Conclusions

- No one of the enzymatic treatments (CP and PK2) has negative effects on the breaking load, as all the values obtained are comprised in the uncertainty of the method.
- The shrinkage obtained in home laundering machine (no wool cycle), with samples treated with enzymes, was lowered up to 65 %.
- The visual observation of the dyed fabrics allows to establish that the dyeing

yield is improved up to 10 %.

- The final exhaustion of the dyeing baths was up to 13 % superior with respect to the untreated sample, for the red dyed fabric. But the exhaustion for the blue dyed fabric reached only 2 % more.
- The washfastness obtained were as desired : grade 5 no staining.
- The shrinkage results obtained when the enzymatic and the chitosan treatments are applied successively allows to affirm that this is a promissory procedure for obtaining articles that meet the IWTO requirements, in spite of the handle obtained was not the desired.

5.0 References

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