# Effects of Low Temperature Plasma on Wool and Wool/nylon Blend Dyed Fabrics

Cristina Canal, Ricardo Molina<sup>1</sup>, Enric Bertran<sup>1</sup>, Antonio Navarro<sup>2</sup>, and Pilar Erra\*

 Surfactant Technology Department, Institute of Chemical and Environmental Research of Barcelona (IIQAB), Consejo Superior de Investigaciones Científicas (CSIC), c/Jordi Girona 18-26, 08034 Barcelona, Spain
 <sup>1</sup>FEMAN, Applied Physics and Optics Department, Universidad de Barcelona, Avda. Diagonal 647, 08034 Barcelona, Spain
 <sup>2</sup>INQUISUP, Chemical Engineering Department, Universidad Politécnica de Catalunya, c/Colom 1, 08222 Terrassa, Spain (Received October 22, 2007; Revised February 19, 2008; Accepted February 21, 2008)

**Abstract:** Knitted wool and wool/nylon blend dyed fabrics were treated with low temperature plasma (LTP) to achieve optimum shrink-resistance without impairing surface topography, colour or fastness to washing of the fabrics. As LTP tends to impair handle of the fabrics, both wool and wool/nylon blend fabrics were submitted to industrial softening and/or biopolymer treatments after LTP treatment, leading to hydrophilic wool and wool/nylon blend fabrics with improved shrink-resistance without any colour changes and good fastness to washing. The results obtained were compared with those obtained by an industrial shrink-resist treatment.

Keywords: Wool, Wool/nylon, Low temperature plasma, Dyed fabrics, Shrink-resistance

#### Introduction

The surface morphology of wool plays an important role in wool processing [1], being the scales of the wool fibre responsible for the directional frictional effect that is believed to be the main cause of felting shrinkage [2]. Traditional shrinkresist treatments imply the consumption of chemical products and often produce polluted wastewaters. With the increasing ecological and economical restrictions imposed on the textile industry, the companies were required to find environmentally favourable alternatives in wool treatment processes [3]. It is essential that any alternative shrink-proofing process imparts a high level of shrink resistance in order to be commercially acceptable [4]. A number of investigations concerning the effect of low-temperature plasma (LTP) treatment on the wool fibre have shown that the treatment can improve the processing and performance characteristics of the wool fibre, such as reducing the tendency to felting [5].

The effect of LTP on wool fabrics has usually been studied focusing on the detection of shrink-resistance effects and on the increase of dyeability. However, up to now there have been no studies reflecting how plasma could be inserted in the complex finishing process of an existing company, and very few [6] have studied the effects of LTP on previously dyed fabrics. In this paper we have investigated the optimum conditions for treating dyed fabrics with LTP and, after this stage we have studied the scientific viability of inserting LTP technology in the industrial finishing process of wool and wool/nylon blend knitted fabrics by comparing their properties with those of industrially treated fabrics.

Previous studies revealed that after LTP, the handle of wool fabrics became harsher and was unacceptable for commercial use [7]. Therefore, it is of great interest to know whether the fabrics can be industrially softened after a LTP treatment, keeping the LTP enhanced properties of wool unchanged. In addition, chitosan (CHT), a polycationic biopolymer has been used with the aim of enhancing the shrink-resist and pilling properties achieved by LTP.

#### **Experimental**

## Materials

A selection of industrially dyed knitted wool and wool/ nylon blend fabrics (Canet Punt S.A., Spain) was used. Wool fabrics studied (with a cover factor of  $1.34 \text{ tex}^{1/2}/\text{mm}$ , 316.6 g/m<sup>2</sup>, 6 wales/cm, 9 courses/cm) had been dyed industrially (in bobbin) dark red, grev and raw. Wool/nvlon blend fabrics (Wool/polyamide 60/40) (with a cover factor of 1.26 tex $^{1/2}$ / mm,  $331.19 \text{ g/m}^2$ , 5 wales/cm, 8 courses/cm) had been dyed industrially in flock brown, grey and navy blue colours. No more details on the dveing process could be disclosed by the company providing the fabrics. Before treatments, fabrics were washed for 10 min at 25 °C using an aqueous bath with 2 g/l of a non-ionic surfactant (Cadetram 9M, CADES) at a fabric to bath ratio (B/R) of 1/30. Samples were then rinsed with water for 3 min. A second washing was carried out with a 1.5 g/l surfactant solution at B/R 1/30 for 5 min at 25 °C. After rinsing for 5 min with water, samples were finally thoroughly rinsed with deionised water and dried at room temperature.

# Treatments

#### LTP Treatments

A radio-frequency reactor operating at 13.56 MHz was employed [8] using water vapour or oxygen as plasma gases. The distance between the electrodes was 8.5 cm, and the

<sup>\*</sup>Corresponding author: pesqst@iiqab.csic.es

sample was hung equidistant between the electrodes. The samples were placed in the vacuum chamber, which was evacuated to a pressure of approx. 10 Pa before introducing the plasma gas. During treatments, both the treatment time and the incident power were varied with the pressure constant at 100 Pa. The power was uniformly distributed on a 400 cm<sup>2</sup> cathode surface.

Optimisation of the LTP conditions was carried out with the red washed knitted wool fabric and the navy blue washed knitted wool/nylon blend fabric, as a way to ensure highest fastness, because they were the most intense and less solid colours among those studied. Treatment times of 40 s, 120 s and 300 s were studied at 50 W, 100 W and 150 W, using water vapour as plasma gas. When oxygen was used as plasma gas 120 s and 300 s treatment times were studied at an incident power of 100 W.

#### **CHT Treatment**

Fabrics were post-treated with CHT, supplied by Vanson (Raymond, WA, USA), with degree of deacetylation and viscosity of 79.8 % and 845 cps respectively, was used without further purification. Freshly prepared solutions of CHT were applied by exhaustion at 0.1 % o.f.w. (dissolved in 0.4 % acetic acid) for 15 min at 25 °C for 15 min at 25 °C at a B/R of 1/20. After the treatment, the fabrics were padded at 3 m/min and 3 bar, and dried at room temperature.

#### Softening Treatment

After plasma or after the combined treatment of plasma and CHT, the samples were subjected to industrial softening with 8 % o.f.w. Onyxan HSB (Cia. Suministros CADES, S.A.) at pH=6, B/R=1/30, 30 °C for 10 min in a oval winch, and then rinsed for 6 min. The fabrics were then dried in an industrial tumbler. The chemical structure of the cationic softener of a quaternary ammonium salt (2-heptadecanil hydroxyetyl imidazoline) is represented in Figure 1.

#### Industrial Shrinkproof

Industrial shrinkproof treatment (IT) was carried out in a company at 30 °C, with the product Fixolan (Albus), followed by Neutralitzan (Albus) and rinsed twice with water. Fabrics were further treated with Acetic acid at 40 % and softened with a cationic softener as described previously. No more details of the process could be provided, as they belong to the know-how of the company.

# CH<sub>3</sub> OH

# Methods

# Wettability

The hydrophilicity of the fabrics has been evaluated by studying the wettability in seconds using the drop test, according to AATCC Test Method 39-1980.

# K/S Value and Whiteness Degree

To evaluate K/S of the dyed samples and the degree of whiteness (CIE Ganz 85) of raw wool, the colorimetric measurement by reflectance on a Color-Eye 3000 spectrophotometer was carried out. Illuminant D<sub>65</sub> and 10° observer were employed during measurements, and in each case the K/S values shown in the study corresponded to the  $\lambda_{max}$  of each dyeing:  $\lambda_{max}^{\text{Dark Red}}$ =540 nm,  $\lambda_{max}^{\text{Grey}}$ =360 nm,  $\lambda_{max}^{\text{Brown}}$ =360 nm and  $\lambda_{max}^{\text{Navy Blue}}$ =580 nm.

#### Washing Fastness

The colour fastness to washing of the wool and wool/ nylon blend fabrics was determined according to the IWS Test Method 193. The colour change (in K/S or degree of whiteness) of the fabric sample was measured. The colour fastness against undyed adjacent cotton and wool fabrics was measured using a Grey Scale for Assessing Staining calibrated with the Color-Eye 3000 spectrophotometer under 10° observer and D65 illuminant.

#### **Determination of Fabric Pilling**

Pilling formation was determined in a Pilling-meter according to ASTM D3512-82 (by submitting the fabric to 2000 abrasion cycles). The degree of pilling was evaluated by comparison with the Verivide scale, ranging values from 1 to 5, being 5 the absence of pilling.

#### Determination of the Area Shrinkage

The shrink resistance test was performed in accordance with Woolmark test method n°31, in a Wascator washing machine model FOM 71 using programme 5A.

#### Scanning Electron Microscopy

Samples were gold coated with a Sputtering Polaron XC500 equipment previously to SEM observations, which were carried out in a HITACHI S-3500 Scanning Electron Microscope in secondary electron detection mode.

# **Results and Discussion**

#### **Effects of LTP Treatment**

In order to establish the optimum LTP conditions for the treatment of wool and wool/nylon blend samples, the changes in wettability, K/S, area shrinkage and fastness to washing have been studied at different plasma treatment time (40, 120 and 300 s) and power (50, 100, 150 W) conditions. Taking into account that during vacuum pumping of the reactor chamber, wool degases water vapour [9], the main plasma gas studied was water vapour, the main plasma gas studied was water vapour.

# Wettability

LTP treated samples are hydrophilic, with wetting times of 0-1 s in all conditions studied and independent of the sample

composition (wool or wool/nylon blend), while untreated wool and wool/nylon blend are highly hydrophobic, showing no wetting at all. However, the samples treated just for 40 s show irregular wetting along the fabrics, with areas with good wettability and others highly hydrophobic, so at such short times the plasma treatment is uneven. Previous studies [9] revealed a decrease in the advancing contact angle of the individual fibres from  $103^{\circ}$  of the untreated fibres to  $50^{\circ}$ and  $45^{\circ}$  for water vapour LTP for 120 and 300 s respectively, which reflected the fact that plasma renders the surface of keratin fibres hydrophilic. The fast wetting times obtained through the drop test confirm such surface changes and are a good and fast indication of the efficiency of the LTP.

The evolution of wetting time with time elapsed after LTP (Figure 2) reveals an ageing process of the surface. Both wool (Figure 2(a)) and wool/nylon blend (Figure 2(b)) fabrics show similar ageing behaviour, as wetting times increase with time elapsed after the treatment. However, fabrics



**Figure 2.** Evolution of the wetting time of (a) wool and (b) wool/ nylon blend fabrics as a function of the time elapsed after the LTP treatment (storage time).

treated for just 40 s show fast ageing, while the evolution of wool and wool/nylon blend samples treated for 120 s or 300 s is slower, confirming contact angle results shown by previous studies of our group [9] of similarly treated keratin

**Table 1.** K/S of water vapour or oxygen plasma treated red wool and navy blue wool/nylon blend fabrics at different LTP treatment times (t(s)) and powers (p(W))

	K/	S <sub><i>i</i>max</sub> We	ool	$K/S_{\lambda max}$ Wool/nylon blend			
P(W)/t(s)	40	120	300	40	120	300	
50	34.40	34.08	34.47	39.01	40.06	38.85	
100	34.85	33.61	33.54	39.56	39.33	39.17	
100, O <sub>2</sub>	_	35.40	34.52	_	37.10	39.66	
150	33.73	33.78	33.36	39.07	39.56	39.66	
UT		34.85			39.66		



**Figure 3.** SEM images of wool fibres (a) untreated (UT), and water vapour plasma treated (100 W) for (b) 40 s, (c) 120 s, and (d) 300 s.

fibres, as well as for nylon fibres [10]. Therefore, drop test provides a fast, easy and reliable way to follow the aging of LTP treated fabrics.

# Colour Evaluation and Surface Topography

Table 1 summarizes K/S values for dark red wool and navy blue wool/nylon blend fabrics subjected to the different plasma treatments.

The K/S values of both dyed wool and wool/nylon blend fabrics treated with LTP in all conditions studied are very similar to those of the untreated fabrics, and no visual differences could be appreciated among them. Other studies carried out with more aggressive plasma treatments [6] showed that the crater formation played an important role in the increase of the colour depth. As it can be seen from the SEM micrographs (Figure 3), the treatments carried out in this case do not cause relevant topographical changes on the surface. Only in Figure 3(d) the formation of microcraters  $(<10^{-2} \mu m)$  after 300 s of water vapour plasma treatment can be observed, although they are not wide enough so as to lead to visible colour changes. Such results confirm the observations made by Hirano, who indicated that microcraters, from 0.1 to 1  $\mu$ m in width, which correspond to the wavelength of the visible ray, are effective for increasing the colour depth of polyester fibre [11].

# Colour Fastness to Washing

It is also of interest to evaluate whether LTP affects the fastness to washing of the dyed fabrics, both wool and wool/ nylon blend (Table 2). Treatment of wool and wool/nylon blend fabrics with water vapour and oxygen plasma does not produce significant changes in the colour fastness to washing. The degree of staining of the adjacent cotton and wool fabrics in most cases even shows a slight improvement.

#### Shrink-resistance

The results shown in Figure 4 reveal that all LTP treatments reduce the area shrinkage of wool knitted fabrics considerably (ranging from 20 % to 9 % in the 2nd washing cycle) with respect to untreated wool (54 %). The plasma treatments that achieve lower shrinkage values of the wool fabrics in the

**Table 2.** Colour fastness to washing of LTP-treated red wool fabrics and navy blue wool/nylon blend fabrics at different treatment times (in seconds). Change in colour in K/S and degree of staining of two undyed cotton (CO) and wool (WO) fabrics are represented

	Colour fastness to washing									
Fabrics	W	/ool fabri	cs	Wool/nylon blend						
-	K/S	СО	WO	K/S	CO	WO				
UT	34.03	4	4	36.45	5	4-5				
40 s	32.78	4-5	4-5	33.78	5	5				
120 s	32.06	3-4	4-5	32.01	5	5				
120 s, O <sub>2</sub>	32.06	4-5	4-5	37.10	5	5				
300 s	30.70	4	4-5	35.16	5	5				

\*All treatments carried out at 100 W.



**Figure 4.** Area shrinkage of LTP treated wool fabrics at different time and power conditions as a function of the washing cycles (5A) in a Wascator washing machine.



**Figure 5.** Area shrinkage of LTP treated wool/nylon fabrics at different time and power conditions as a function of the washing cycles (5A) in a Wascator washing machine.

2nd washing cycle which then remain fairly stable in the 3rd washing cycle are those of water vapour plasma for 120 s at 100 W or 150 W, and either water vapour or oxygen plasma for 300 s at 100 W.

Untreated wool/nylon blend fabrics show much lower shrinkage values (18%) than wool (Figure 5), due to the presence of PA6 in the yarn. Nevertheless, all plasma treatments reduce shrinkage of the wool/nylon blend fabrics (ranging from 13% to 3%), and most of the conditions investigated would produce machine washable fabrics.

The shrink-resistance effect induced by the LTP treatment on wool is attributed to changes in the wool surface, such as the 1) formation of new hydrophilic groups by oxidation of the fatty acid monolayer, as confirmed by XPS studies [9], 2) progressive removal of fatty acids covalently bonded to the outermost surface of the fibre (Fatty-layer) with increasing treatment times, and 3) the etching effect [12,13]. Whereas the first two kinds of modification contribute to an increase in the wettability properties, removal of fatty acids and etching could result in a reduction in the differential friction effect of the fibres and, hence, in a decrease of the natural tendency of wool to shrinking [2].

Taking into account all results discussed, and knowing that there is usually water vapour contamination in the reaction chamber, the best conditions for the treatment of both wool and wool/nylon blend would be to use water vapour as plasma gas for 120 s of time (it is the shortest time necessary to achieve a uniform treatment) at 100 W of incident power. Consequently, all results discussed from now on will correspond to such plasma conditions.

# Comparative Study between a Conventional Industrial Shrinkproof Treatment and LTP of Dyed Wool and Wool/nylon Blend Knitted Fabrics

The wool and wool/nylon fabrics used in this part of the work are the same as those used in the first part of this paper, but a wider range of colours is studied, and conventional industrial treatments are applied.

# Wettability

Drop test results summarized in Table 3 reveal a clear difference between the industrially finished fabrics (untreated (UT), softened, industrial shrinkproof (IT), IT+softened), which are highly hydrophobic (with wetting times which can be considered as infinite) and all plasma treated fabrics (LTP, LTP+CHT, LTP+softening, LTP+CHT+softening), which are hydrophilic. As expected, the colour of the sample does not remarkably influence the wettability of the fabrics. It is remarkable; however, that wool/nylon blend samples treated with LTP and CHT or LTP and CHT and softened, exhibit longer wetting times than wool fabrics with the same treatment. Such behaviour could be attributed to the presence of polyamide on the fabrics, which after plasma treatment adsorbs more CHT on the surface, as we have ascertained by means of contact angle measurements [14].

**Table 3.** Drop test results of wool and wool/nylon blend dyed fabrics untreated (UT), industrial shrinkproof (IT) and water vapour LTP treated (120 s, 100 W) and submitted to different finishing treatments

	Dr	op test ( Wool	(s)	Drop test (s) Wool/nylon blend			
	Dark red	Grey	Raw	Brown	Grey	Navy blue	
UT	$\infty$	$\infty$	$\infty$	$\infty$	$\infty$	$\infty$	
UT+softening	$\infty$	$\infty$	$\infty$	$\infty$	$\infty$	$\infty$	
IT	$\infty$	$\infty$	$\infty$	$\infty$			
IT+softening	$\infty$	$\infty$	$\infty$	$\infty$	$\infty$	$\infty$	
LTP	0	0	0	0	0	0	
LTP+CHT	25±2	13±4	1±1	503±763	981±999	835±191	
LTP+softening	30±13	97±15	15±9	78±25	15±4	66±26	
LTP+CHT+soft.	19±26	21±9	$3\pm1$	102±59	$17\pm8$	70±16	

\*Results obtained are the average of 5-8 droplets.

**Table 4.** K/S of water vapour plasma treated wool and wool/nylon blend dyed fabrics untreated (UT), industrial shrinkproof (IT) and water vapour LTP treated (120 s, 100 W) and submitted to different finishing treatments

		K/S <sub>\lambdamax</sub> Wool		K/S <sub>∂max</sub> Wool/nylon blend			
	Dark Red	Grey	Raw*	Brown	Grey	Navy Blue	
UT	34.09	2.04	3.23	2.85	2.39	41.56	
UT+softened	38.85	2.15	1.17	2.83	2.48	43.16	
IT	35.64	1.88	0.40	_	_	_	
IT+softening	36.46	1.04	17.96	2.81	2.37	42.47	
LTP	34.03	2.08	1.63	2.78	2.47	39.81	
LTP+CHT	37.03	2.11	7.14	2.81	2.39	39.74	
LTP+softening	41.29	2.33	6.86	3.17	2.68	42.86	
LTP+CHT+soft.	38.52	2.38	9.35	3.14	2.57	42.38	

\*Degree of whiteness CIE Ganz 85.

# **Colour Change**

Table 4 shows the effect of the different treatments applied on wool and wool/nylon blend dyed fabrics on the colour of the samples. As can be seen, neither grey wool, nor grey or brown wool/nylon blend show any relevant colour differences with any of the treatments applied (less than 1 point of K/S difference with respect to the UT sample).

However, dark red wool fabrics reveal higher colour sensibility to the different treatments, mainly after the softening treatments.

LTP does not produce any relevant changes on the whiteness degree of raw wool fabrics. However, IT+softening and CHT application on LTP fabrics increase significantly the whiteness degree of the fabrics.

# Colour Fastness to Washing

Dark red wool fabrics submitted to industrial treatments (Table 5) are those showing lowest colour fastness to washing and worst degree of staining both on the adjacent wool and cotton undyed fabrics. On the contrary, LTP and further post-treatments tend to improve the degree of staining of dark red wool fabrics. White and grey wool show good degree of staining values in all cases, independently of the treatment applied.

All wool/nylon blend fabrics, independently of the colour and treatment applied show good colour fastness to washing, and the degree of staining remains mainly unaltered after the different industrial and LTP treatments and post-treatments.

# **Pilling**

Table 6 reflects that while softening and IT treatments tend to increase pilling formation, LTP treatments or LTP+CHT tend to reduce pilling formation. Previous studies [15] observed some interfibre CHT bonding, which was assumed to prevent movement of fibres and reducing shrinkage. Such deposition of CHT on the wool fibre surface and interfibrillar bonding could prevent fibres escape the fabric structure, therefore

Table 5. Colour fastness to washing of dyed wool and wool/nylon blend dyed fabrics with different finishing treatments. C	Change in colour in
K/S and degree of staining of two undyed cotton (CO) and wool (WO) fabrics are represented	
Colour fastness to washing	

				Coloui	idotilebb to v	vaoning				
	Wool fabrics									
	Dark Red				Grey			Raw		
	K/S	СО	WO	K/S	СО	WO	CIE Ganz85	СО	WO	
UT	34.03	4	4	2.02	4-5	4-5	10.48	5	5	
UT+softening	33.42	3-4	3	2.14	5	4-5	11.29	4-5	5	
IT	28.28	2-3	3	2.15	4-5	4-5	11.14	4-5	4-5	
IT+softening	31.85	3	3	1.94	5	5	18.07	4-5	5	
LTP	32.68	3-4	2-3	2.45	5	3	1.63	5	3-4	
LTP+CHT	30.15	4	4	2.41	5	4-5	7.13	5	5	
LTP+softening	32.95	4	4-5	2.28	5	4-5	11.64	4-5	4-5	
LTP+CHT+soft.	32.17	4	4	2.42	5	4-5	11.56	5	4-5	
	Wool/nylon blend fabrics									
		Brown Grey				Navy Blue				
	K/S	СО	WO	K/S	СО	WO	K/S	СО	WO	
UT	2.86	4	4-5	2.52	5	4-5	36.45	5	4-5	
UT+softening	2.94	5	4-5	2.47	4-5	4-5	39.17	4-5	4	
IT+softening	3.09	4-5	4-5	2.50	5	4-5	36.88	5	4-5	
LTP	2.94	5	4-5	2.51	5	4-5	33.14	5	4-5	
LTP+CHT	3.98	5	4-5	2.52	5	4-5	32.85	5	4-5	
LTP+softening	2.93	5	4-5	2.46	5	4-5	35.16	5	4-5	
LTP+CHT+soft.	2.91	5	4-5	2.63	5	4-5	36.38	5	4-5	

**Table 6.** Pilling of dyed wool and wool/nylon blend fabrics

 submitted to different finishing treatments

	Pilling	of Woo	l blend	Pilling of Wool/nylon			
	Dark Red	Grey	Raw	Brown	Grey	Navy Blue	
UT	3/4	3/4	3	4	4	3/4	
UT+softening	2	2	2	3	3/4	3	
IT	2	3	2	3/4	4/5	4	
IT+softening	3	3	3/2				
LTP	4/5	4/5	4/5	4/5	5	5	
LTP+chitosan	5	5	4/5	4	4	4	
LTP+softening	3/4	3/4	3	2/3	3/4	4/5	
LTP+chit.+soft.	4/5	4/5	4	3	2	3/4	

exerting a protective function and preventing pilling formation. It is remarkable that while LTP and CHT tend to decrease pilling formation, softening of both wool and wool/nylon blend fabrics tends to worsen, as observed in [16]. That could be attributed to the inverse effect of softeners, which may favour fibre displacements and, consequently, pilling.

# Shrink-resistance

The analysis of the results shown in Figure 6 reveals a progressive increase of the area shrinkage with the washing cycles for all samples, but to different extents depending on the treatment.

It has to be remarked that softened and IT+softened wool

fabrics behave like untreated wool, with area shrinkage values over 43 % in the 2nd washing cycle. Although the industrial shrink-resist treatment achieves effective reduction of the area shrinkage, after the application of the softener, wool shrinks again as though it were UT.

In all three wool colours, the samples showing lower shrinkage values are those treated with LTP and LTP+CHT, and can be considered as shrink-resistant (machine washable). The softening process applied, improves handle of LTP treated wool but also increases considerably the shrinkage of wool fabrics.

Combined application of LTP, CHT and softening treatments produces wool fabrics with good shrink-resistance properties. That could be attributed to the combined effect of LTP and CHT. On the one hand plasma reduces shrinkage and enhances adhesion on the surface of wool fibres, which produces enhanced adhesion of CHT on the wool fibres [12]. On the other hand, as we already mentioned, CHT deposition on the surface forms a thin film which may reduce friction coefficient and interfibre bonding which prevents fibre movement [12]. That is probably the main reason that justifies that shrinkage remains within good values (below 15 %) after the softening process, conversely as in the former cases without CHT. Unfortunately, handle of the fabrics treated with LTP+ CHT+softening is not good enough, as CHT produces a stiffness of the fabrics that the softening process itself is not able to compensate.



W////// UT Softening 80 Dark Red IT IT 70 IT + Softening LTP 60 ITP+CHT LTP+Softening 1777 50 LTP+CHT+Soft % Area shrinkage 40 30 20 10 0 1st cycle 2nd cycle 3rd cycle -10 -Washing cycles //// UT 80 Grey Softening Softening IT 5 70 -IT+Softening LTP 60 LTP+CHT 50 LTP+Softening % Area shrinkage 40 30 20 10 0 2nd cycle 1st cycle 3rd cycle -10 -Washing cycles Raw 777777 UT 80 -Softening IT 📖 70 · IT+Softening 60 LTP LTP+CHT 50 Z LTP+Softening % Area shrinkage LTP+CHT +Soft 40 30 20 10 0 1st cycle 2nd cycle 3rd cycle -10 -Washing cycles

**Figure 6.** Area shrinkage of wool fabrics with different finishing treatments.

As seen before, UT wool/nylon blend fabrics (Figure 7) show area shrinkage values (in the second washing cycle) above 20 % for brown and grey fabrics, and below 10 % for navy blue. Such differences could be explained by small differences in machine tension during knitting. As with wool, softening even increases the area shrinkage of wool/nylon blend fabrics. As shown by drop test and in other studies [7], such phenomenon could be attributed to the increase of hydrophobicity of wool after the softening process.

LTP treatment not only reduces shrinkage, but even provokes the enlargement of wool/nylon blend fabrics with the successive washing cycles. That could be attributed to the fact that now both wool and PA6 fibres are hydrophilic and during washing they could be surrounded by a water film which eases the relative displacement of fibres leading to fabric relaxation.

Similarly as observed for wool fabrics, softening of LTP



**Figure 7.** Area shrinkage of wool/nylon blend fabrics with different finishing treatments.

treated fabrics provokes an important increase of area shrinkage (except for blue wool/nylon blend, maybe due to inefficient softening treatment). CHT application on LTP treated wool/ nylon blend fabrics keeps very good area shrinkage values, corresponding to shrink-resistant fabrics. Again, fabrics treated LTP+CHT+softening are shrink-resistant.

# Conclusion

We have compared industrially treated wool and wool/

# Fibers and Polymers 2008, Vol.9, No.3 299

nylon blend dyed fabrics with LTP treated ones, and posttreated with the biopolymer CHT and/or industrially softened, showing the compatibility of such processes with the present industrial process.

The LTP based processes achieve the improvement of many properties of the dyed fabrics; all fabrics treated with LTP and post-treated are hydrophilic, do not cause any relevant colour alteration and show improved fastness to washing. In addition, LTP, LTP+CHT and LTP+CHT+ softening produce shrink-resistant wool and wool/nylon blend fabrics, although the handle is still not optimum for commercialisation and will have to be further improved in future studies.

#### Acknowledgements

This research has been carried out within the FEMAN and INQUISUP partner units. The authors acknowledge the financial support from by the MCYT (PETRI project n° 950655), as well as the collaboration of Ms. I Muñoz on the experimental work and JM Fortuño and M Escusa on the SEM images. We would also like to heartily acknowledge the cooperation of the company Canet Punt S.A. on this study.

## References

1. M. Feughelman in "Mechanical Properties and Structure of Alpha-keratin Fibres: Wool, Human Hair and Related Fibres" (M. Feughelman Ed.), p.5, University of New South Wales Press, Sydney, 1997.

- K. R. Makinson in "Surface Characteristics of Fibers and Textiles" (M. J. Schick Ed.), Vol. 1, p.109, Marcel Dekker, New York, 1975.
- C. W. Kan, K. Chan, C. W. M. Yuen, and M. H. Miao, J. Materials Proc. Tech., 83(1-3), 180 (1998).
- R. J. Denning, G. N. Freeland, G. B. Guise, and A. H. Hudson, *Text. Res. J.*, 64(7), 413 (1994).
- C. W. Kan, K. Chan, C. W. M. Yuen, and M. H. Miao, J. Materials Proc. Tech., 82, 122 (1998).
- J. Ryu, T. Wakida, and T. Takagishi, *Sen-I Gakkaishi*, 47(11), 612 (1991).
- C. Canal, R. Molina, E. Bertran, and P. Erra, *Macromol. Mater. Eng.*, **292**, 817 (2007).
- G. Viera, J. Costa, F. J. Compte, E. García-Sanz, J. L. Andujar, and E. Bertran, *Vacuum*, 53, 1 (1999).
- 9. R. Molina, P. Jovancic, D. Jocic, E. Bertran, and P. Erra, *Surf. Interface Anal.*, **35**, 128 (2003).
- C. Canal, R. Molina, E. Bertran, and P. Erra, J. Adh. Sci. Technol., 18(9), 1077 (2004).
- 11. Y. Hirano, Sen'I Kikai Gakkaishi, 37, 131 (1984).
- P. Erra, R. Molina, D. Jocic, M. R. Julia, A. Cuesta, and J. M. D. Tascon, *Text. Res. J.*, **69**, 811 (1999).
- I. M. Zuchairah, M. T. Pailthorpe, and S. K. David, *Text. Res. J.*, 67(1), 69 (1997).
- C. Canal, Ph.D. Thesis, Universitat Politecnica de Catalunya, 2005.
- S. Vilchez, P. Jovancic, A. M. Manich, M. R. Julià, and P. Erra, *J. Appl. Polym. Sci.*, **98**(5), 1938 (2005).
- M. Bona, E. Prina, and F. Ramazio, *Proc. 9th Int. Wool Text. Res. Conf.*, V, 230 (1995).