

# THE POSSIBILITY OF LOW-TEMPERATURE PLASMA TREATED WOOL FABRIC FOR INDUSTRIAL USE

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## Abstract

*In this paper, low-temperature plasma (LTP) treatment was applied to a wool fabric. The LTP-treated wool fabric was tested according to different international standard testing methods, and the results were compared with the industrial requirements (ASTM requirements). It was revealed that the LTP-treated wool fabric did meet industrial requirements. The results of the investigation are thoroughly discussed in this paper.*

## Key words:

*low temperature plasma (LTP); wool fabric; industrial requirements; standard testing method; wool fibre; environmentally friendly*

## Introduction

The presence of scale structure on the wool fibre surface introduces a number of problems such as felting and the formation of a surface barrier to dyestuff. In the past, chemical methods [1-5] were the major treatment for those problems. However, the effluents generated from wool dyeing and finishing processes are seriously contaminated with different kinds of chemicals, e.g. chloro-organic compounds from the anti-felting process. With the increase in ecological and economical restrictions imposed on the textile industry, environmentally favourable alternatives are required in the wool treatment processes. The low-temperature plasma (LTP) method which has developed rapidly over the past decade has been introduced into many branches of the textile industry.

LTP is an ionic gas whose components and characteristics are different from normal gas. With the help of electrical discharges, various plasma of different ionisation extents can be produced. Either non-polymerising gases or polymerising gases can be used in LTP treatment, but the final results of LTP will depend largely on the nature of the gases used [6]. Since the temperature of plasma is relatively low, the activating species in plasma will easily lose their energy once they react with the polymer material. As a result, the penetration of the activating species in plasma into the polymer materials is so shallow that the interior of the material is only lightly affected. LTP treatment can thus be used as an effective technique for modifying the surface properties of wool fabric without much alteration to the interior of the fibre (penetration only to a depth of about 1000Å [7]). In this paper, the LTP-treated wool fabrics were tested with different international standard testing methods, and the results were compared with the industrial requirements (ASTM requirements).

## Experimental

### Wool fabric

All chemicals and reagents were of A.R. or Laboratory Grade. 2/1 twill wool fabric (41 ends/cm, 31 tex; 36 picks/cm, 36 tex; 180 g/m<sup>2</sup>) was scoured with dichloromethane for 4 hours using the Soxhlet extraction method. The solvent-scoured wool fabric was then rinsed twice with 98% ethanol and washed twice with deionised water. The cleaned fabric was dried in an oven at 50°C for 30 minutes and then air-dried. Finally, the fabric was conditioned according to ASTM D1776 [8] before use.

### **Low-temperature plasma (LTP) treatment**

A glow discharge generator (Showa Co. Ltd., Japan) was used for the low-temperature plasma treatment of the wool fabric with the use of oxygen gas. The discharge power and system pressure were adjusted to 80W and 10 Pa respectively, and the duration of treatment was 5 minutes. After the LTP treatment, the LTP-treated wool fabric was conditioned according to ASTM D1776 [8] prior to use.

### **Tensile strength and elongation**

The wool fabric's tensile properties and elongation at break were measured according to ASTM D5035 [9] using an Instron Tensile Tester (the one-inch ravelled strip test was conducted). Ten specimens were prepared for testing, five for warp direction and the other five for weft direction. All measurements were repeated for the five equally treated samples and averaged.

### **Tearing strength**

The tearing strength of wool fabric was measured by the Elmendorf Tearing Tester from the Thwing-Albert Instrument Co. in accordance with ASTM D1424 [10]. Ten specimens were prepared, five in warp direction (for tearing across the weft) and five in weft direction (for tearing across the warp). All measurements were repeated for the five equally treated samples and averaged.

### **Fabric shrinkage**

The dimensional changes of the LTP-treated wool fabric was tested according to AATCC Test Method 99-1993 [11]. Due to the limited size of the plasma reaction chamber, the dimension of the fabric sample used was 20 x 20 cm, with a 15 cm x 15 cm square marked inside the fabric. The dimensional stability test was conducted in the following sequence: (i) relaxation, (ii) consolidation and (iii) felting, in which the decrease in dimension also followed such a sequence, that is, the shrinkage was smallest after the relaxation process, and largest after the felting process. The fabric was conditioned before measurement. The measurement was then conducted to assess the shrinkage in length of both warp and weft direction, and finally the area shrinkage was calculated. The degree of shrinkage (expressed in %) in length and area were calculated according to Equations (1) and (2) respectively.

$$\text{Length change} = \frac{l_f - l_o}{l_o} \times 100\% \quad (1)$$

where:

$l_f$  = final length after treatment (cm),  
 $l_o$  = original length before treatment (cm).

$$\text{Area change} = \frac{A - O}{O} \times 100\% \quad (2)$$

where:

A = final area after treatment (cm),  
O = original area before treatment (cm).

### **Dyeing of wool fabric**

Dyeing was conducted by placing the wool fabric in a dyebath containing 4% o.w.f. sulphuric acid and 5% o.w.f. Glauber's salt with a liquor ratio of 1:150. The dyebath was kept at a temperature of 70°C for 10 minutes after the addition of acid and salt. 1% o.w.f. Neolan Red GRE 200% (C.I. Acid Red 183) was then added to the dyebath and maintained at 70°C for a further 5 minutes before raising the temperature to 100°C at a heating rate of 1°C/minute. The dyeing was continued at 100°C for a further 180 minutes. After dyeing, the fabrics were rinsed with deionised water until no colour appeared in the rinse-off water. The fabrics were dried and finally conditioned before measurement.

### **Colour fastness test**

The colour fastness tests were assessed using an international standard, namely AATCC. Three colour fastness tests were conducted including washing, perspiration and rubbing.

### ***Washing fastness***

The AATCC 61-1994 1A test [12] was used for assessing the colour fastness to washing of the dyed wool fabric.

### ***Perspiration fastness***

The AATCC Test Method 15-1994 [13] was used for assessing the colour fastness to perspiration of the dyed wool fabric.

### ***Crocking fastness***

The AATCC Test Method 8-1989 [14] (AATCC crockmeter method) was used for assessing the colour fastness to crocking of the dyed wool fabric.

## **Results and Discussion**

### **Tensile strength and elongation**

The results of breaking load and elongation at break are shown in Tables 1 and 2 respectively.

**Table 1.** The breaking load of the wool fabrics in both warp and weft directions

Wool fabric	Untreated	LTP-treated
Breaking load in warp direction (kg)	26.25	32.70
Breaking load in weft direction (kg)	15.80	18.50

**Table 2.** The percentage of elongation at break of the wool fabrics in both warp and weft directions

Wool fabric	Untreated	LTP-treated
Percentage of elongation at break in warp direction (%)	53.7	54.2
Percentage of elongation at break in weft direction (%)	15.8	31.9

The breaking loads and the elongation at break of LTP-treated fabric were comparatively larger than those for the untreated fabric. In the tensile strength test, a load was applied to cause fabric breakage. In general, fabric breakage depends not only on the nature of the fibre but also on the fabric's construction. When considering the fabric's construction, the inter-yarn and inter-fibre friction plays an important role in the tensile strength properties of the fabric. With the application of LTP treatment, it was believed that such treatment increased the inter-yarn and inter-fibre frictional force, as confirmed by the roughening effect on the textile surface [17]. Hence, more forces are required in order to overcome the inter-yarn and inter-fibre friction before the occurrence of fabric breakage, resulting in a higher breaking load.

The modified elongation of LTP-treated fabric was probably due to the cleavage of the disulphide linkage present on the fibre surface [15]. This cleavage could soften the wool scales, thus making the fibres more elastic. In addition, the increase in inter-yarn and inter-fibre frictional force could further enhance the modification. The improved breaking load and percentage of elongation implied that the LTP-treated fabric could be extended even more when compared with the untreated fabric. This confirmed that LTP treatment could impart good extensibility to the fabric.

### **Tearing strength**

**Table 3.** The tearing strength of the wool fabric in both warp and weft directions

Wool fabric	Untreated	LTP-treated
Tearing strength in warp direction (gram)	2328	1596
Tearing strength in weft direction (gram)	1032	920

The tearing strength of each sample is shown in Table 3. Under the influence of LTP treatment, the tearing strengths of the wool fabric were reduced in both warp and weft directions. The mechanism of tearing was explained by the appearance of a del [16] in the cut slit of the test fabric during the tearing strength testing. The formation of this del was probably due to the relative sliding of yarns during the tearing period. The del yarn broke consecutively as the load was applied, resulting in tearing of the fabric. When this del was large, more yarns experienced the same load, leading to an increase of sliding between yarns during the tearing period and causing the tearing strength to increase. However, when the inter-yarn frictional force between warp and weft yarns was too large, the sliding action of the yarns was relatively reduced, making the del become smaller, and consequently the tearing strength was decreased. In the previous study [17], it was found that the inter-yarn frictional force increased after LTP treatment. Therefore, it is postulated that the inter-yarn friction will restrict the sliding action of yarns during tearing, thereby reducing the tearing strength values.

### **Fabric shrinkage**

**Table 4.** The results of dimensional changes (lengthwise) of the samples

Wool fabric	Relaxation dimensional change (%)		Consolidation dimensional change (%)		Felting dimensional change (%)	
	Warp	weft	Warp	weft	Warp	weft
Untreated	5.0	2.0	6.8	2.6	9.6	3.6
LTP-treated	0.6	0.2	0.8	0.3	1.1	0.4

Three types of dimensional changes were measured and tabulated, as shown in Table 4. The values in Table 4 show the percentage shrinkage in warp and weft directions. It was clearly observed that the dimensional change in the warp direction was greater than in the weft direction.

The relaxation dimensional change occurred when the fabric was immersed in water without agitation, so that the strains and stresses imparted during fabric formation could be released. The fabric was then dried and reconditioned to the relative humidity of 65% at which it was originally measured. Of all the LTP treatment, it was found that the LTP-treated fabric underwent only a slight change in dimension after the relaxation process, 0.6% and 0.2% in the warp and weft directions respectively. The component of relaxation shrinkage produced by mild agitation in water may be referred to as consolidation shrinkage.

In consolidation shrinkage, the untreated wool fabric also showed the greatest change in both warp and weft directions when compared with the LTP-treated fabric. Generally speaking, the variation of dimensional change between the different LTP-treated wool fabrics was very small in the weft direction, and the variation of consolidation shrinkage in the warp direction was greater than in the weft direction.

The felting dimensional change is an irreversible dimensional change which occurs in a wool fabric when it is subjected to agitation in laundering. The felting dimensional changes were greatest in both warp and weft directions among other dimensional changes. The maximum value of the felting dimensional changes in the untreated wool fabric was 9.6%, which was only a moderate change for the untreated fabric. However, when this value was compared with the LTP-treated fabric, it demonstrated that the LTP treatment could impart significant shrink-resistant and anti-felting effects to the wool fabric.

For a detailed study of how the LTP affects the overall fabric shrinkage, the area shrinkage was calculated, and the results are shown in Table 5.

**Table 5.** The results of area shrinkage of samples

Wool fabric	Relaxation dimensional change in area shrinkage (%)	Consolidation dimensional change in area shrinkage (%)	Felting dimensional change in area shrinkage (%)
Untreated	6.90	9.22	12.28
LTP-treated	0.80	1.07	1.46

From Table 5, it may be seen that the area shrinkage significantly decreased after the subsequent LTP treatment. Clearly, the area shrinkage increased as the processing changed from relaxation shrinkage to felting shrinkage.

For the fabric shrinkage study, generally speaking, the wool fabric shrinkage is correlated with the frictional coefficient of the constituent wool fibres, and it is common knowledge that LTP treatment increases the dry and wet frictional coefficient in the scale and anti-scale direction [25]. However, the effect of the LTP process is attributed to several changes in the wool surface, such as the formation of new hydrophilic groups, the partial removal of covalently-bonded fatty acids belonging to the outermost surface of the fibre, and the etching effect [26, 27]. The first two changes contribute mainly to the increased wettability properties, while the last basically reduces the differential friction coefficients of the fibres, and thus decreases the natural shrinkage tendency [23].

## **Colour fastness**

### ***Colour fastness to washing***

The AATCC standard was used to assess the colour fastness of the LTP-treated wool fabric and the results are shown in Table 6.

**Table 6.** Colour fastness (washing) of wool fabric using the AATCC standard

Wool fabric	Staining						Colour change
	Wool	Acrylic	Polyester	Nylon	Cotton	Acetate	
Untreated	3-4	3-4	3-4	3-4	3	3-4	3-4
LTP-treated	4	4	4	4	3-4	4	4

The principle of the AATCC washing fastness test is to test the samples under appropriate conditions of temperature, detergent solution, bleaching and abrasive action so that the colour change is similar to that occurring after five hand, domestic or commercial launderings. The colour change can be obtained in a conveniently short time. The abrasive action is due to the result of the frictional effects of fabric against a container, the low liquor ratio and the impact of steel balls on the fabric.

After LTP treatment, it was found that all the dyed LTP-treated fabrics had a slightly improved colour fastness to washing for both staining and colour change assessment. The results of all the LTP-treated fabric was the same (a ½-step increase in each case).

### ***Colour fastness to perspiration***

**Table 7.** Colour fastness (perspiration) of wool fabric using the AATCC standard

Wool fabric	Staining						Colour change
	Wool	Acrylic	Polyester	Nylon	Cotton	Acetate	
Untreated	3	4	4	2	2-3	4	2-3
LTP-treated	3-4	4-5	4-5	2-3	3	4-5	3-4

Table 7 shows the results of the colour fastness to perspiration under the AATCC standard. Obviously, the LTP-treated fabric has a similar fastness rating, which was better than those achieved using the untreated wool fabric. In the colour change rating, the LTP treatment gives a positive improvement of the rating, implying that the LTP-treated wool fabric has become faster than the untreated wool fabric.

### ***Colour fastness to crocking***

The AATCC standard was used for assessing the colour fastness of the LTP-treated wool fabric, and the results are shown in Table 8. In the dry crocking condition, the colour fastness of the LTP-treated wool fabric was slightly improved, whereas the colour fastness in wet crocking showed no significant improvement.

**Table 8.** Colour fastness (crocking) of wool fabric using the AATCC standard

Wool fabric	Croaking fastness (wet)	Croaking fastness (dry)
Untreated	4	4
LTP-treated	4	4-5

After the LTP treatment, the wool fibre surface was modified [18], and the extent of the surface modification was previously examined by transmission electron microscopy [19]. Investigation showed that the LTP treatment only modified the A-layer of the cuticle to varying degrees, which resulted in parts of the A-layer being sputtered off, thus leading to the formation of grooves [19] in this layer. The affinity of the fibre for dyes is significantly increased due to the partial degradation of the A-layer, which represents a barrier to the diffusion of dyes into the wool fibre due to the high number of crosslinks, and also due to the hydrophilisation of the fibre surface. The dye can therefore accumulate to a greater extent in this layer, and thus diffuse into the fibre faster and more homogeneously. The facilitated dye absorption is probably also caused by a modification of the endocuticle and the neighbouring cell membrane complex, and thus by a modification on the intercellular path of diffusion. As a result, the colour fastness of the LTP-treated wool fabric was improved.

### Matching with performance specification requirements

Although the LTP treatment could improve or change the properties of fabrics to different extents, it was necessary for the LTP-treated wool fabric to fulfil the performance specification requirements. Two performance specifications were selected, namely (i) ASTM D3780-95: Standard performance specification for men's and boys' woven dress suit fabrics and woven sportswear jacket, slack, and trouser fabrics [20] and (ii) ASTM D4155-95: Standard performance specification for women's and girls' woven sportswear, shorts, slacks, and suiting fabrics [21]. Table 9 showed the performance specification of different fabric properties, which included breaking strength, tearing strength, dimensional change, colour fastness to washing, colour fastness to perspiration and colour fastness to crocking.

**Table 9.** Performance specification of different properties

Properties	ASTM D3780-95	ASTM D4155-95	Untreated	LTP-treated
Breaking strength	178N min.	155N min.	<b>262.5N (warp), 158N (weft)</b>	<b>327N (warp), 185N (weft)</b>
Tear strength	11N min.	8.9N min.	<b>23.3N (warp), 10.3N (weft)</b>	<b>16N (warp), 9.2N (weft)</b>
Dimensional change <sup>A</sup>	2% max. in each direction	3% max. in each direction	<b>9.6% (warp), 3.6% (weft)</b>	<b>1.1% (warp), 0.4% (weft)</b>
Colour fastness to washing (shade change)	Class 4 min.	Class 4 min.	Class 3-4	<b>Class 4</b>
Colour fastness to washing (staining) <sup>B</sup>	Class 3 min.	Class 3 min.	<b>Class 3</b>	<b>Class 4</b>
Colour fastness to perspiration (shade change)	Class 4 min.	Class 4 min.	Class 2-3	Class 3-4
Colour fastness to perspiration (staining) <sup>B</sup>	Class 3 min.	Class 3 min.	Class 2	Class 2-3
Colour fastness to crocking (dry)	Class 4 min.	Class 4 min.	<b>Class 4</b>	<b>Class 4-5</b>
Colour fastness to crocking (wet)	Class 3 min.	Class 3 min.	<b>Class 4</b>	<b>Class 4</b>

**A** – Felting dimensional change

**B** – the lowest staining rating among different multi-fibre components

The values in **BOLD and ITALIC** form shows the improved properties when compared with the performance specification

Table 9 demonstrated the performance test results of the LTP-treated fabric in comparison with the standard performance specifications. In the breaking strength values, all fabrics (including the

untreated fabric) met the standard requirements. Although the untreated fabric could itself have met the requirements, the breaking strength of the fabrics was further enhanced after the LTP treatment.

The tearing strengths of the fabrics in warp and weft directions fulfilled the performance specification requirements.

In assessing colour fastness to washing, the untreated wool fabric failed to meet the minimum requirement in shade (colour) change, and just fulfilled the minimum requirements after the LTP treatments. On the other hand, the staining colour fastness of all fabrics satisfied the standard requirements.

Regarding colour fastness to perspiration, neither untreated nor LTP-treated fabrics achieved the minimum specification requirements. The fastness ratings in both shade change and staining assessment were found to be slightly improved under the influence of LTP treatments.

All fabric samples fulfilled the specification requirements in both dry crocking and wet crocking tests of the colour fastness to crocking assessment. In the dry crocking test, the staining ratings of the LTP-treated fabrics were merely improved by a ½-step. However, all the staining ratings of the fabric samples in the wet crocking test were the same.

Although the hand feel of the LTP-treated wool fabrics was not assessed in this investigation, the hand feel of the LTP-treated wool fabrics became harsh [17, 28]. However, the LTP-treated wool fabric could enhance the polymer deposition [22] which could in turn improve the hand feel [23, 24].

## Conclusion

From the above experimental results, we concluded that the LTP treatment appears to be a method for modifying the wool fabric with quite a significant effect. The LTP-treated wool fabrics could also meet the performance specification requirements.

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