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A Study of the Oxygen Plasma Treatment on the Serviceability of a Wool Fabric

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Abstract: Low temperature plasma (LTP) treatment using oxygen gas was applied to a wool fabric. The LTP treated wool fabric was tested with several methods: ASTM D5035-1995, ASTM D1424-1996, AATCC Test Method 99-2000, AATCC Test Method 61-2001 1A, AATCC Test Method 15-2002 and AATCC Test Method 8-2001 and the results were compared with the industrial requirements (ASTM D3780-02 and ASTM D4155-01). The results revealed that the LTP treated wool fabric could fulfil the industrial requirements. The results of the investigation were discussed thoroughly in this paper.

Keywords: Low temperature plasma (LTP), Wool fabric, Industrial requirement, Standard testing method, Wool fibre, Environmentally friendly

Introduction

The scale structure on the wool fibre surface causes a number of problems such as felting and surface barrier to dyestuff in wool industry. In the past, chemical methods [1-5] were the major treatment for eliminating those problems. However, the effluents generated from wool dyeing and finishing processes may contaminate with different kinds of chemicals, e.g., chloro-organic compounds from antifelting process. With the increasing of ecological and economical restrictions imposed on the textile industry, environmentally favourable alternatives have been required in wool treatment processes. Low temperature plasma (LTP) technology developed rapidly in the past decade has been introduced to the textile industry for wool treatment process.

LTP is an ionic gas whose components and characteristics are different from the normal gas. With the help of electrical discharge, 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 react with the polymer material. As a result, the penetration of the activating species in plasma into polymer materials is so shallow that the interior of the material is slightly affected. LTP treatment can thus be used as an effective technique for modifying the surface properties of wool fabric without much alteration in the interior of the fibre (penetrate only to a depth of about 1000 Å [7]). In this investigation, the LTP using oxygen gas was employed in treating the wool fabric. The LTP treated wool fabric was tested with different standard testing methods and the results were compared with industrial requirements so as to evaluate the serviceability of the oxygen plasma treated wool.

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Experimental

Wool Fabric

All chemicals and reagents, unless otherwise stated, were in Analytical Reagent Grade. 2/1 twill wool fabrics (41 ends/cm, 31 tex; 36 picks/cm, 36 tex; 180 g/m^2) were scoured with dichloromethane for 4 hours using the Soxhlet extraction method. The solvent scoured wool fabrics were then rinsed twice with 98 % ethanol and washed twice with deionised water. The cleaned fabrics were dried in an oven at 50 ^oC for 30 minutes and the air dried. Finally, the fabrics were 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 wool fabrics with the use of oxygen gas. The discharge power and system pressure were 80 W and 10 Pa, respectively, and the duration of treatment was 5 minutes. After the LTP treatment, all treated wool fabrics were conditioned according to ASTM D1776 [8] prior to use.

Tensile Strength and Elongation

The tensile properties and elongation at break of wool fabric were measured according to ASTM D5035 [9] using Instron Tensile Tester. 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 Elmendorf Tearing Tester (Thwing-Albert Instrument Co.) in accordance with ASTM D1424 [10]. Ten specimens were prepared in which 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 fabrics were tested according to AATCC Test Method 99-2000 [11]. Due to the limited size of the plasma reaction chamber, the dimension of the fabric samples used was 20 cm × 20 cm, with a marked 15 cm × 15 cm square inside the fabrics. The dimensional stability tests were conducted in the following sequence: (i) relaxation, (ii) consolidation and (iii) felting, in which the decrease in dimension also followed such sequence, i.e., the shrinkage was the smallest after the relaxation process, and the largest after the felting process. All of the fabrics were conditioned before measurement. The measurement was conducted to assess length shrinkage in both warp and weft directions and finally, area shrinkage were calculated. The degree of shrinkage (expressed in %) in length and area were calculated according to equations (1) and (2), respectively.

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

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 fabrics 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, commercial product without further purification) was then added to the dyebath and maintained at 70 °C for 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 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

Standard testing method, namely AATCC, was used in the colour fastness test. Three colour fastness testing were conducted including (i) washing (AATCC Test Method: 6-2001 1A) [12], perspiration (AATCC Test Method: 15-2002) [13] and (iii) crocking (AATCC Test Method 8-2001 (AATCC crockmeter method)) [14] for evaluating and assessing the colour fastness properties of the dyed LTP treated wool fabric.

Results and Discussion

Tensile Strength and Elongation

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

The breaking loads and elongation of LTP treated fabric were comparatively larger than those of the untreated fabric. In the tensile strength test, a load was applied until the fabric broke. In general, the fabric breakage depends not only on the nature of the fibre but also on the fabric construction. When considering the fabric construction, the inter-yarn and inter-fibre friction plays an important role in the tensile strength properties of the fabric. With the application of oxygen LTP treatment, it was believed that this technique increased the inter-yarn and inter-fibre friction as confirmed by the roughening effect on the textile surface [15,16]. Hence, more forces must be required to overcome the inter-yarn and inter-fibre friction before the occurrence of fabric breakage, resulting in higher breaking load [16].

The modified elongation of LTP treated fabric was probably due to the cleavage of the disulphide linkage on the fibre surface [17]. This cleavage could soften the wool scales 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 more than the untreated fabric. This confirmed that LTP treatment could impart good extensibility to the fabric [16].

Tearing Strength

The tearing strength of each sample is shown in Table 3.

Table 1. The breaking load of the wool fabrics in both warp and weft directions (with analytical tolerance limited to 5 %)

Sample	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 of the wool fabrics in both warp and weft directions (with analytical tolerance limited to 5 %)

Sample	Untreated	LTP treated
Percentage of elongation in warp direction (%)	53.7	54.2
Percentage of elongation in weft direction (%)	15.8	31.9

Table 3. The tearing strength of the wool fabric in both warp and weft directions (with analytical tolerance limited to 5 %)

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

Due to LTP treatment, the tearing strength of the wool fabric was reduced in both warp and weft directions. The mechanism of tearing was explained by the appearance of a del [18] 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 would experience 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 friction was too large between warp and weft yarns, the sliding action of yarns would be relatively reduced making the del smaller, and consequently the tearing strength was decreased. In previous studies [15,16], it was found that the inter-yarn friction was 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 values of tearing strength.

Fabric Shrinkage

Three types of dimensional changes were measured and tabulated as shown in Table 4.

Table 4 showed the percentage shrinkages in warp and weft directions. It was clearly observed that the dimensional change in the warp direction was greater than that in the weft direction. As a result, the dimensional changes in the two directions will be discussed separately.

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 % after which was originally measured. Under the influence of the oxygen LTP treatment, it was found that the LTP treated fabric had only a slight change in dimension, 0.6 % and 0.2 % in warp and weft directions respectively, after the relaxation process. The component of relaxation shrinkage produced by mild agitation in water may be referred to as consolidation shrinkage. In the consolidation shrinkage, the untreated wool fabric also showed significant change in both warp and weft directions when compared with the LTP treated fabric. The felting dimensional change is an irreversible dimensional change when occurs in wool fabric when it is subjected to agitation in laundering. The felting dimensional changes were the greatest in both warp and weft directions among other dimensional change. The maximum value of the felting dimensional changes in the untreated wool fabric was 9.6 %, which can be regarded moderate change for the untreated fabric. However, when these values were compared with the LTP treated fabric, it reflected that the LTP treatment could impart significant shrink-resistant and anti-felting effects on the wool fabric.

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

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.

The wool fabric shrinkage is correlated with the frictional coefficient of the constituent wool fibres, and it is a common knowledge that LTP treatment increases the dry and wet frictional coefficient in the scale and anti-scale direction [19]. However, the effect of the LTP process is attributed to several changes in the wool surface, such as the formation of new hydrophilic group, partial removal of covalently bonded fatty acids belonging to the outermost surface of the fibre, and the etching effect [20,21]. Whereas the first two changes contribute mainly to increased wettability while the last basically reduces the differential friction coefficients of the fibres and thus decreases the natural shrinkage tendency [22].

Colour Fastness

Colour Fastness to Washing

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

Table 4. The results of dimensional changes (lengthwise) of the samples (with analytical tolerance limited to 5 %)

Sample	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

Table 5. The results of area shrinkage of wool fabrics (with analytical tolerance limited to 5 %)

Sample	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

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

Sample	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

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

Sample	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

The principle of AATCC washing fastness test is to test the samples under appropriate conditions of temperature, detergent solution, bleaching and abrasive action such that the colour change is similar to that occurring in five hand, home 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 container, low liquor ratio and the impact of the steel balls on the fabric. After LTP treatment, it was found that the dyed LTP treated fabrics had a slightly improved colour fastness to washing in both staining and colour change assessment.

Colour Fastness to Perspiration

The AATCC standard was used for assessing the colour fastness of the LTP treated wool fabric and the results were shown in Table 7.

Table 7 shows the results of the colour fastness to perspiration under AATCC standard. Obviously, the LTP treated fabrics have a similar fastness rating which were better than those achieved using the untreated fabric. In the colour change rating, the LTP treatment makes a positive improvement implying that the LTP treated fabric become more fast 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.

The crocking fastness ratings in wet and dry conditions are summarised 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

Sample	Crocking fastness (wet)	Crocking fastness (dry)
Untreated	4	4
LTP treated	4	4-5

After the LTP treatment, the wool fibre surface [23] and the extent of the surface modification were examined by transmission electron microscopy [24]. The surface investigation showed that the LTP treatment only modified the A-layer of the cuticle to varying degrees and parts of the A-layer were sputtered off leading to the formation of grooves [24] in the A-layer. Due to the partial degradation of the A-layer which represents a barrier to the diffusion of dyes into the wool fibre, the high number of crosslinks, and the hydrophilization of the fibre surface, the affinity of the fibre for dyes significantly increased. Therefore, the dye can accumulate more in this layer and thus diffuse into the fibre fast and more homogeneously. The facilitated dye absorption may also be caused due to a modification of the endocuticle and the neighbouring cell membrane complex as well as 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 extent, it was necessary that the LTP treated wool fabric should fulfil the performance specification requirements. Two performance specifications were selected: (i) ASTM D3780-02: Standard performance specification for men's and boys' woven dress suit fabrics and woven sportswear jacket, slack, and trouser fabrics [25] and (ii) ASTM D4155-01: Standard performance specification for women's and girls' woven sportswear, shorts, slacks, and suiting fabrics [26]. Table 9 showed the performance specification of different fabric properties which include breaking strength, tearing strength, dimensional change, colour fastness to washing, colour fastness to perspiration and colour fastness to crocking.

Table 9 demonstrated the performance test results of the LTP treated fabric compared with the standard performance specifications. In the breaking strength values, both fabrics fulfilled the standard requirements. Although the untreated

Table 9. Performance specification of different properties

Properties	ASTM D3780-02	ASTM D4155-01	Untreated	LTP treated
Breaking strength	178 N min.	155 N min.	***262.5 N (warp), 158 N (weft)***	***327 N (warp), 185 N (weft)***
Tear strength	11 N min.	8.9 N min.	***23.3 N (warp), 10.3 N (weft)***	***16 N (warp), 9.2 N (weft)***
Dimensional change[a)]	2 % max. in each direction	3% max. in each direction	***9.6 % (warp), 3.6 % (weft)***	***1.1 % (warp), 0.4 % (weft)***
Colour fastness to washing (Shade Change)	Class 4 min.	Class 4 min.	Class 3-4	***Class 4***
Colour fastness to washing (Staining)[b)]	Class 3 min.	Class 3 min.	***Class 3***	***Class 4***
Colour fastness to perspiration (Shade Change)	Class 4 min.	Class 4 min.	Class 2-3	Class 3-4
Colour fastness to perspiration (Staining)[b)]	Class 3 min.	Class 3 min.	Class 2	Class 2-3
Colour fastness to crocking (Dry)	Class 4 min.	Class 4 min.	***Class 4***	***Class 4-5***
Colour fastness to crocking (Wet)	Class 3 min.	Class 3 min.	***Class 4***	***Class 4***

[a)]Felting dimensional change, [b)]the lowest staining rating among different multifibre components, The values in ***bold and italic*** form shows the improved properties when compared with the performance specification.

fabric could meet the requirements, the breaking strength of the fabrics was further enhanced after the LTP treatment.

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

In assessing the 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 both fabrics satisfied the standard requirements.

In the case of the colour fastness to perspiration, both untreated and LTP treated fabrics did not achieve the minimum specification requirements. The fastness ratings in both shade change and staining assessment were found to be slightly improved by 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 1/2 step. However, all the staining ratings of the fabric samples in the wet crocking test were not changed.

Although the hand of the LTP treated wool fabrics were not assessed in this investigation, it was reported that the hand of the LTP treated wool fabrics become harsh [15,16]. However, the LTP treated wool fabrics could enhance the polymer deposition [27] which could hence improve the hand feel of the LTP treated wool fabrics [22,28].

Conclusion

The basic aim of this investigation was to investigate the influence of LTP treatment, with the use of oxygen gas, on the different properties of a wool fabric. From the experimental results, the LTP treatment using oxygen gas appeared to be a good and practicable method for modifying the wool fabric environmentally friendly, and its effect on fabric properties was significant. The oxygen plasma treated wool fabric, in addition, could also fulfil the performance specification requirements with international standards. On the other hand, this investigation provided helpful information in improving the wool fabric finishing process in an environmentally friendly way.

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