Low-temperature plasma treatment of wool fabric for its industrial use

C W Kan, K Chan & C W M Yuen
Institute of Textiles and Clothing, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong SAR, China
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Low-temperature plasma (LTP) treatment using oxygen gas has been applied to wool fabric. The LTP-treated fabric has been tested for its properties, such as tensile strength, elongation, tearing strength, shrinkage and colour fastness, using different international standard testing methods and the results compared with the industrial requirements. It is observed that the LTP-treated wool fabrics meet the industrial requirements. LTP treatment is found to increase extensibility and decrease tearing strength and shrinkage tendency of the fabric with overall improved fastness properties.

Keywords: Low-temperature plasma treatment, Wool fabric
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1 Introduction
The presence of scale structure on the wool fibre surface introduces a number of problems, such as felting and surface barrier to dyestuff, in wool industry. In the past, chemical methods\(^5\) were mainly used for eliminating these problems. However, the effluents generated from wool dyeing and finishing processes were found to be seriously contaminated with different kinds of chemicals, e.g. chloro-organic compounds from anti-felting process. With the increase in ecological and economical restrictions imposed on the textile industry, environmentally-favourable alternatives were required in wool treatment processes. Low-temperature plasma (LTP) method developed rapidly in the past decade has been introduced to the textile industry.

LTP is an ionic gas whose components and characteristics are different from the normal gas. With the help of electrical discharge, 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 depend largely on the nature of gases used\(^6\). Since the temperature of plasma is relatively low, the activating species in plasma easily lose their energy once reacting with the polymeric material. As a result, the penetration of activating species in plasma into the polymeric 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 part of the fibre (penetrate only to a depth of about 1000Å)\(^7\). In the present investigation, the LTP with the use of oxygen gas has been employed in treating the wool fabric. The LTP-treated wool fabric has been tested for its properties with different international standard testing methods and the results are compared with the industrial requirements so as to evaluate the serviceability of the oxygen plasma-treated wool fabric.

2 Materials and Methods

2.1 Materials
All chemicals and reagents were of 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 h 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°C for 30 min and then air dried. Finally, the fabrics were conditioned before use\(^8\).

2.2 Methods

2.2.1 Low-temperature Plasma 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 80W and 10 Pa respectively, and the duration of treatment was...
5 min. After the LTP treatment, all the treated wool fabrics were conditioned according to ASTM D1776 prior to use.

2.2.2 Tensile Strength and Elongation

The tensile properties and elongation-at-break of wool fabric were measured according to ASTM D5035 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 the average was taken.

2.2.3 Tearing Strength

The tearing strength of wool fabric was measured by Elmendorf tearing tester (Thwing-Albert Instrument Co.) in accordance with ASTM D1424 (ref. 10). Ten specimens were prepared, considering 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 the average was taken.

2.2.4 Fabric Shrinkage

The dimensional changes of the LTP-treated fabrics were tested according to AATCC test method 99-2000 (ref. 11). Due to the limited size of the plasma reaction chamber, the dimension of the fabric sample used was 20 cm × 20 cm, with a marked 15 cm × 15 cm 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 the fabrics were conditioned before measurement. The measurement was conducted to assess the shrinkage in length in both warp and weft directions and finally the area shrinkage was calculated. The degree of shrinkage (expressed in %) in length and area was calculated according to the following equations:

Length change, % = \( \frac{\left(l_f - l_0\right)}{l_0} \times 100 \)

where \( l_f \) is the final length after treatment (cm); and \( l_0 \), the original length before treatment (cm).

Area change, % = \( \frac{\left(A - O\right)}{O} \times 100 \)

where \( A \) is the final area after treatment (cm²); and \( O \), the original area before treatment (cm²).

2.2.5 Dyeing of Wool Fabric

The dyeing was conducted by placing the wool fabrics in a dyebath containing 4% (owf) sulphuric acid and 5% (owf) Glauber’s salt with a material-to-liquor ratio of 1:150. The dyebath was kept at 70°C for 10 min after the addition of acid and salt. 1% (owf) Neolan Red GRE 200% (C.I. Acid Red 183) was then added to the dyebath and the temperature was maintained at 70°C for further 5 min and then raised to 100°C at a heating rate of 1 °C/min. The dyeing was continued at boil for a further 180 min. 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.

2.2.6 Colour Fastness Test

International standard testing method (AATCC) was used for colour fastness test. Three colour fastness testings were conducted including (i) washing (AATCC test method: 6-2001 1A)\(^{12}\), (ii) perspiration (AATCC test method: 15-2002)\(^{13}\) and (iii) crocking (AATCC test method 8-2001)\(^{14}\) for evaluating and assessing the colour fastness properties of the dyed LTP-treated wool fabric.

3 Results and Discussion

3.1 Tensile Strength and Elongation

Table 1 shows that the breaking load and the elongation-at-break of LTP-treated fabric are comparatively larger than those of the untreated fabric. In the tensile strength test, a load was applied to cause the fabric breakage. In general, the fabric breakage depends not only on the nature of fibre but also on the fabric construction. When considering the fabric construction, the inter-yarn and inter-fibre

<table>
<thead>
<tr>
<th>Sample</th>
<th>Breaking load, kg</th>
<th>Elongation-at-break, %</th>
<th>Tearing strength, g</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>15.80</td>
<td>53.7</td>
<td>1032</td>
</tr>
<tr>
<td>Untreated</td>
<td>15.80</td>
<td>53.7</td>
<td>1032</td>
</tr>
<tr>
<td>LTP-treated</td>
<td>18.50</td>
<td>31.9</td>
<td>920</td>
</tr>
</tbody>
</table>

Table 1—Properties of untreated and LTP-treated wool fabrics
frictions play an important role in the tensile strength properties of the fabric. With the application of oxygen LTP treatment, it is believed that the inter-yarn and inter-fibre frictions increase, as confirmed by the roughening effect on the textile surface. Hence, more forces must be required to overcome the inter-yarn and inter-fibre frictions before the occurrence of fabric breakage, resulting in higher breaking load.

The modified elongation of LTP-treated fabric is probably due to the cleavage of disulphide linkage present on the fibre surface. This cleavage softens the wool scales, making the fibres more elastic. In addition, the increase in inter-yarn and inter-fibre frictions further enhances the modification. The improved breaking load and percentage of elongation imply that the LTP-treated fabric can be extended even more when compared with the untreated fabric. This confirms that the LTP treatment imparts good extensibility to the fabric.

### 3.2 Tearing Strength

The tearing strength of each sample is shown in Table 1. Under the influence of LTP treatment, the tearing strengths of the wool fabric reduce in both warp and weft directions. The mechanism of tearing is explained by the appearance of del in the cut slit of the test fabric during the tearing strength testing. The formation of this del is probably due to the relative sliding of yarns during the tearing period. The del yarn breaks consecutively as the load is applied, resulting in tearing of fabric. When this del is large, more yarns will experience the same load, leading to the increase in sliding between yarns during the tearing period and causing the tearing strength to increase. However, when the inter-yarn friction is too large between warp and weft yarns, the sliding action of yarns relatively reduces, making the del to become smaller, and consequently the tearing strength decreases. In the previous study, it has been found that the inter-yarn friction increases after LTP treatment. Therefore, it is postulated that the inter-yarn friction restricts the sliding action of yarns during tearing, thereby reducing the tearing strength.

### 3.3 Fabric Shrinkage

Three types of dimensional changes (percentage shrinkage) in both warp and weft directions are shown in Table 2. It is observed that the dimensional change in the warp direction is greater than that in the weft direction. Therefore, the dimensional changes in the two directions are discussed separately.

The relaxation dimensional change occurs when the fabric is immersed in water without agitation so that the strain and stress imparted during fabric formation are released. The fabric is then dried and reconditioned under the relative humidity of 65% at which it was originally measured. Under the influence of oxygen LTP treatment, it is found that the LTP-treated fabric has 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 shows the greatest change in both warp and weft directions when compared with the LTP-treated fabric. 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 are the greatest in both warp and weft directions among other dimensional changes. The maximum value of felting dimensional changes in the untreated wool fabric is 9.6%, which is only a moderate change for the untreated fabric. However, when these values are compared with the LTP-treated fabric, it is observed that the LTP treatment imparts significant shrink-resistant and anti-felting effects on the wool fabric. For a detailed study on how the LTP affects the overall fabric shrinkage, the area shrinkage has been calculated and the results are shown in Table 3.

From Table 3, it may be observed that the area shrinkage significantly decreases after the subsequent LTP treatment. The area shrinkage increases as the processing changes from relaxation shrinkage to felting shrinkage. Generally, 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 coefficients in the scale and anti-scale directions. However, the effect of LTP treatment is attributed to several changes in the wool surface, such as formation of new hydrophilic group, partial removal of covalently bonded fatty acids belonging to the outermost surface of the fibre, and the etching effect. The first two changes contribute mainly to the increased wettability while the last basically reduces the differential friction coefficients of the fibres and thus decreases the natural shrinkage tendency.

3.4 Colour Fastness

The AATCC standards were used for assessing the colour fastness of LTP-treated wool fabric and the results are shown in Table 4. It is observed that all the dyed LTP-treated fabrics have a slightly improved colour fastness to washing for both staining and colour change assessment.

Table 4 shows that the LTP-treated fabrics have a similar perspiration fastness rating which is better than that achieved using the control fabric. In the colour change rating, the LTP treatment makes a positive improvement, implying that the LTP-treated fabric becomes faster than the untreated wool fabric.

In the dry crocking condition, the colour fastness of LTP-treated wool fabric is slightly improved whereas wet crocking shows no significant improvement in colour fastness.

After the LTP treatment, the wool fibre surface is modified and the extent of surface modification has been examined by transmission electron microscopy previously. It is observed that the LTP treatment only modifies the A-layer of cuticle to varying degrees as part of the A-layer have been sputtered off leading to the formation of grooves in this layer. Due to the partial degradation of A-layer, which represents a barrier to the diffusion of dyes and other chemicals into the wool fibre due to the high number of crosslinks, and hydrophilization of the fibre surface, the affinity of the fibre for dyes is significantly increased. Therefore, the dye can accumulate more in this layer and thus diffuses into the fibre faster and more homogeneously. The facilitated dye absorption is probably caused by the modification of endocuticle and neighbouring cell membrane complex, causing a modification of intercellular path of diffusion. As a result, the colour fastness of the LTP-treated wool fabric improves.

3.5 Performance Specification Requirement

Although the LTP treatment improves or changes the properties of fabrics to different extent, it is necessary that the LTP-treated wool fabric should meet 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, and (ii) ASTM D4155-01: standard performance specification for women’s and girls’ woven sportswear, shorts, slacks and suitings fabrics. Table 5 shows the performance specifications of different fabric properties which include breaking strength, tearing strength, dimensional change, and colour fastness to washing, perspiration and crocking. The performance test results of the untreated and LTP-treated fabrics have been compared with the standard performance specifications. In the breaking strength values, both the fabrics meet the standard requirements. Although the untreated fabric itself could meet the
requirements, the breaking strength of the fabrics has been further enhanced after the LTP treatment. The tearing strength of the fabrics in warp and weft directions also meets the performance specification requirements.

In assessing the colour fastness to washing, the untreated wool fabric fails to meet the minimum requirement in shade (colour) change but the fabric meets the minimum requirements after the LTP treatments. On the other hand, the staining colour fastness of all the fabrics is found to be satisfactory with the standard requirements.

In the case of colour fastness to perspiration, both untreated and LTP-treated fabrics do not achieve the minimum specification requirements. The fastness ratings in both shade change and staining assessment are found to be slightly improved under the influence of LTP treatments.

All the fabrics meet the specification requirements in both dry and wet crocking tests. In the dry crocking test, the staining ratings of the LTP-treated fabrics are merely improved by half a step. However, all the staining ratings of the fabric samples in the wet crocking test are found to be the same.

Although the hand feel of the LTP-treated wool fabric is not assessed in this investigation, it is reported to be harsh. However, the LTP treatment enhances the polymer deposition on wool fibre surface which improves the hand feel of the fabrics.

4 Conclusions

The LTP treatment with the use of oxygen gas helps in modifying the wool fabric and the effect is quite significant. The LTP treatment is found to increase the extensibility and decrease the tearing strength and shrinkage tendency of the fabric with overall improved fastness properties. The oxygen plasma treated wool fabrics, on the other hand, meet the performance specification requirements with international standards.

References