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Research Article
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Investigation of the Effect of Different Plasma Treatment Condition on the Properties of Wool Fabrics

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Abstract

In this study, oxygen and nitrogen plasma treatment was carried out on wool fabrics during 5, 10 and 15 minutes at low (LF) (40 kHz) and radio (RF) (13.59 MHz) frequency. Then the effect of plasma treatment on tear strength, contact angle, whiteness and yellowness index of wool fabrics was investigated. In addition, the hydrophility of plasma treated samples was analyzed after the washing. The physical and chemical properties of wool fabrics treated with oxygen and nitrogen plasma were characterized by scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDX). According to the results, nitrogen plasma treatment improved the tear strength of samples. Furthermore, plasma treatment increased the yellowness of samples while decreasing the whiteness index of samples. The hydrophility of samples decreases with plasma treatment and with the increase in plasma treatment time. Moreover, SEM results showed that the plasma treatment caused to occur deformations on the surfaces. Lastly, EDX results showed that the amount of oxygen and nitrogen increased in the surfaces after oxygen and nitrogen plasma treatment.

Keywords: Oxygen plasma; Nitrogen plasma; Wool; Tear strength; Hydrophility; Whiteness; Yellowness index

Introduction

In textile industry, wool materials have been used for a long time because of their high thermal insulation, comfort and eco-friendliness, while having problems such as felting, pilling, luster and shrinkage [1-5]. In order to solve these problems, chemical and physical treatments have been recently used. Some chemical treatments used are ozone, enzyme and oxidation treatments while plasma treatment, electron beam irradiation, ion implantation and ultrasonic irradiation are among physical treatments commonly used [6-9].

Plasma technology has been applied in textile industry in order to produce a variety of surface modifications of textile materials. It also improves a wide range of textile properties such as hydrophobicity, dye exhaustion, adhesion etc. [10]. Furthermore, the use of plasma treatment provides more antibacterial, flame retardant, hydrophobic, hydrophilic, anti-pilling, electric conductivity, anti-static, scouring, anti-felting, ultraviolet protective textile materials [11-15]. Besides, plasma technology, characterized by low consumption of water, energy and chemicals, is a clean, ecologic and dry technique [6,16]. In addition to these advantages, plasma treatment does not influence textile material bulk properties [17,18].

A variety of gases have been used in plasma treatment such as oxygen, hydrogen, nitrogen and argon [16]. Each gas gives different features to textiles in relation to their chemical groups [19-21].

The aim of this study is to investigate the effects of plasma treatment and gas type on wool fabrics without any pre-treatment. In pre-treatment, wool fabrics were cleaned by Soxhlet extraction with dichloromethane rinsed with ethanol and deionized water before the plasma treatment [8]. This study differs from the others in that wool samples were not scoured. In this study, LF and RF oxygen and nitrogen plasma treatments were performed on wool fabrics for 5, 10 and 15 minutes. After the plasma treatment, the effects of plasma gas, time and frequency of treatment on the wool properties such as tear strength, hydrophility, whiteness index and yellowness were investigated. Moreover, the hydrophility of plasma treated samples was tested to analyze the durability of their hydrophobic properties after washing. The surface of wool fabrics was analyzed by SEM. The results demonstrated that nitrogen plasma treatments improved the tear strength of wool fabrics. Moreover, after the plasma treatment yellowness of samples increased and the whiteness index of samples decreased with the increase in treatment time. The hydrophility of samples decreased with the plasma treatment. The results did not change after the washing, as plasma treated samples were hydrophobic.

Material and Methods

Fabrics

In this study, 100% wool plain weave fabric was used to investigate the different effects of plasma treatment on the properties of fabric. The fabric specimens were 1:1 plain fabrics (24 ends cm^{-1}, 40 Nm; 22 picks cm^{-1}, 48 Nm; 40 g/m^2). Besides the wool fabrics were not cleaned by Soxhlet extraction with dichloromethane rinsed with ethanol and deionized water before the plasma treatment.

Plasma treatment

Plasma treatment was performed on wool fabrics with Diener vacuum plasma with oxygen and nitrogen gas. In this study, the effects of different gases, frequency intensity and plasma treatment time on wool fabrics were investigated. Wool fabrics were treated with oxygen and nitrogen plasma for 5, 10 and 15 minutes. After a lot of

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pre-treatment were carried out by vacuum plasma device, optimum conditions were determined as 40 kHz frequency to LF and 13.59 MHz frequency to RF at the power of 100 W and at the pressure of 0.4 mbar. The properties of fabrics exposed to plasma treatment were given in Table 1.

Tear strength measurements

Tear strength of samples was analyzed by using SDL ATLAS M008E Digital Elmandorf with ballistic pendulum method according to ISO 13937-1:2000 Standard [22] and 64000 mN load was attached to pendulum. The measurements of tear strength were iterated three times.

Contact angle measurements

Contact angle of samples was measured with an optical contact angle measurement instrument such as Attension by Ksv Instrument. The velocity of digital camera was adjusted so as to take 80 images per second and the volume of drop was 4-6 cm^3. Distilled water was used to analyze the contact angle. Contact angle of samples was measured by the images. The measurements of contact angle were iterated four times.

Washing

The washing of plasma treated samples was carried out according to ISO105:C06 at 40°C for 30 minutes [23].

Whiteness index and yellowness measurements

Whiteness index of samples was determined by using Konica Minolta Spectrophotometer CM-3600d according to Stendsby Method and whiteness values were calculated using illuminant D65 and 10° standard observer values. Yellowness of samples was measured with Konica Minolta Spectrophotometer CM-3600d according to ASTM D 1925 [24] and yellowness values were calculated using C-10° standard observer values.

Physical and chemical properties of plasma treated wool fabrics

Physical and chemical structures of samples were analyzed with SEM and EDX by using ZEISS/EVO 40 Electron Microscope.

Results and Discussion

Results of tear strength measurements

The results of the measurements of tear strength of samples treated

Sample code	Properties of fabric
O_2 5′ LF	Oxygen plasma treatment at low frequency for 5 minutes
O_2 10′ LF	Oxygen plasma treatment at low frequency for 10 minutes
O_2 15′ LF	Oxygen plasma treatment at low frequency for 15 minutes
O_2 5′ RF	Oxygen plasma treatment at radio frequency for 5 minutes
O_2 10′ RF	Oxygen plasma treatment at radio frequency for 10 minutes
O_2 15′ RF	Oxygen plasma treatment at radio frequency for 15 minutes
N 5′ LF	Nitrogen plasma treatment at low frequency for 5 minutes
N 10′ LF	Nitrogen plasma treatment at low frequency for 10 minutes
N 15′ LF	Nitrogen plasma treatment at low frequency for 15 minutes
N 5′ RF	Nitrogen plasma treatment at radio frequency for 5 minutes
N 10′ RF	Nitrogen plasma treatment at radio frequency for 10 minutes
N 15′ RF	Nitrogen plasma treatment at radio frequency for 15 minutes

Table 1: The properties of fabrics exposed to plasma treatment.

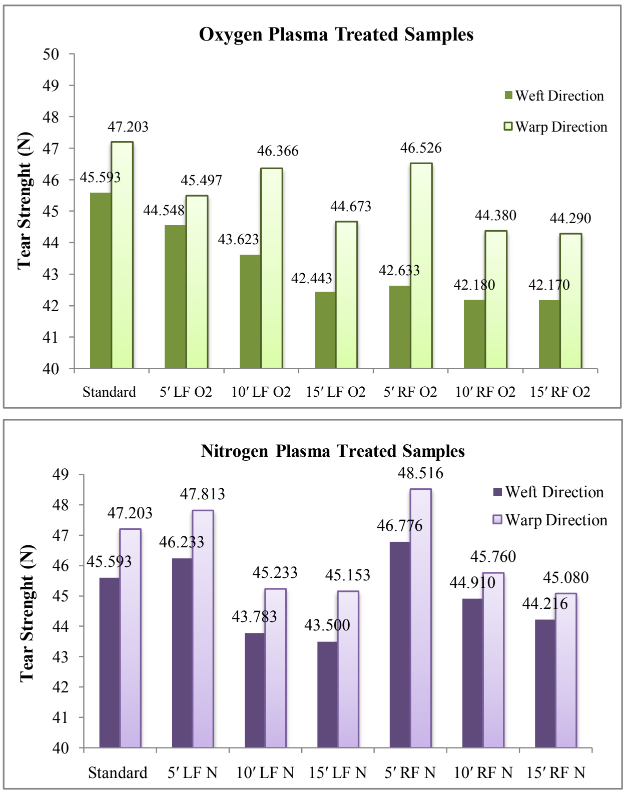


Figure 1: Tear strength of samples.

with oxygen and nitrogen plasma were given in Figure 1.

According to Figure 1, the results showed that the tear strength of warp direction of all samples was higher than that of weft direction of all samples as the warp density of all samples was higher than their weft density. The increase in the number of yarns led to the increase in the strength density of warp direction of all samples. Furthermore, the results demonstrated that oxygen plasma treatment resulted in the decrease in the tear strength of samples. After oxygen plasma treatment, deformation on surface increased with the increase in oxygen treatment time.

The comparison of tear strengths of samples in terms of plasma gas demonstrated that tear strength of samples treated with nitrogen plasma was higher than those treated with oxygen plasma. According to these results, compared to nitrogen plasma, oxygen plasma treatment was considered to cause greater damage on the wool fibers since oxygen has higher electronegativity than nitrogen, therefore, causing more damage than nitrogen.

According to results, the tear strength of samples that were applied LF plasma was lower than those with RF plasma treatment. This is because LF plasma treatment was considered to remove the wax layer slower than RF plasma treatment. Furthermore, it was considered that RF plasma treatment removed less wax layer than LF plasma treatment. Such removal arose from the collusion and scattering of the ionized electrons in different ways due to their high frequency. As the RF collusion occurred more rapid than LF collusion, less ionized electrons were deemed to interact with the samples.

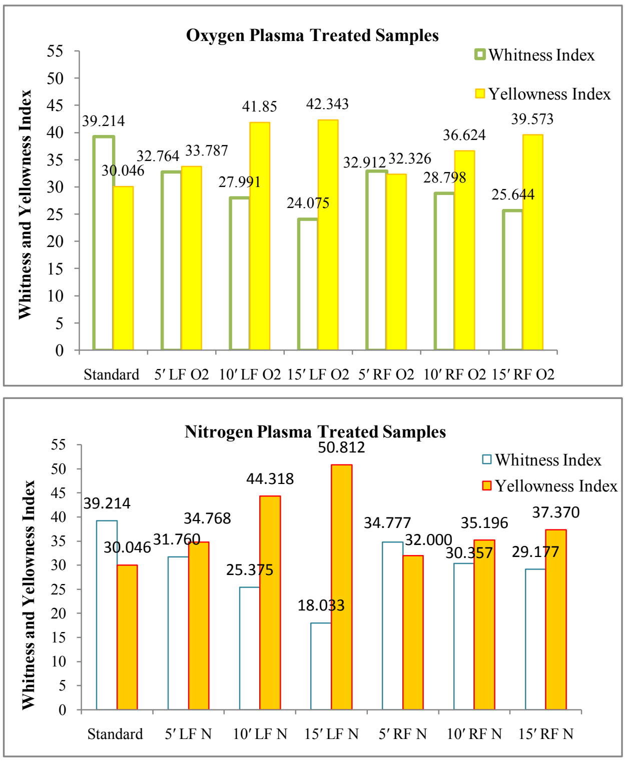


Figure 2: Whiteness and yellowness index of samples.

Results of whiteness and yellowness measurement

Figure 2 shows the measurements of whiteness and yellowness results of the samples treated with oxygen and nitrogen plasma.

According to the measurements, oxygen and nitrogen plasma treatment decreased whiteness index of samples while increasing their yellowness. Whiteness measurements led to the consideration that the plasma treatment of oxygen and nitrogen gas caused deformation on surface. After the plasma treatment, the roughness of surface increased. As is known, reflections of light from rough surfaces are less than smooth ones [25]. Furthermore, increasing treatment time of plasma application resulted in the decrease in whiteness index of samples and the increase in their yellowness values. Such result arises from the consideration that the etching effect of plasma treatment on wax layer of wool fabrics increased due to the increase in plasma treatment time. For this reason, the yellowness of samples increased with the plasma treatment [26]. Besides, comparison of the results in terms of types of plasma gas demonstrated that, at low frequency, nitrogen plasma treatment reduced whiteness index of wool samples while increasing yellowness index. However, at radio frequency, oxygen plasma treatment yielded the same results at a better rate. Such results led to the consideration that each type of gas affected the structure of wool differently [25].

Results of contact angle measurements

The results of contact angle measurements of samples treated with oxygen and nitrogen plasma were given in Table 2.

The results of Table 2 departed from other results in that oxygen and nitrogen plasma treatment led to the increase in hydrophobicity of wool samples [27]. Such result was based on the consideration that samples used in this study were not treated with pre-treatment. These results were considered that the wax layer of surfaces of wool fabric could not be removed, and therefore wax layer was penetrated the wool fabric surfaces with plasma treatment. Furthermore, when the results

Sample	Contact Angle
Standard	111.69
5′ LF O_2	131.90
10′ LF O_2	130.60
15′ LF O_2	129.33
5′ RF O_2	131.14
10′ RF O_2	128.83
15′ RF O_2	125.00
5′ LF N	136.840
10′ LF N	126.570
15′ LF N	116.24
5′ RF N	122.773
10′ RF N	118.110
15′ RF N	117.320

Table 2: Contact angle measurements of samples.

Sample	Contact Angle
Standard	111.69
15′ LF O_2	111.783
15′ RF O_2	125.703
15′ LF N	116.19
15′ RF N	112.297

Table 3: Contact angle measurements of samples.

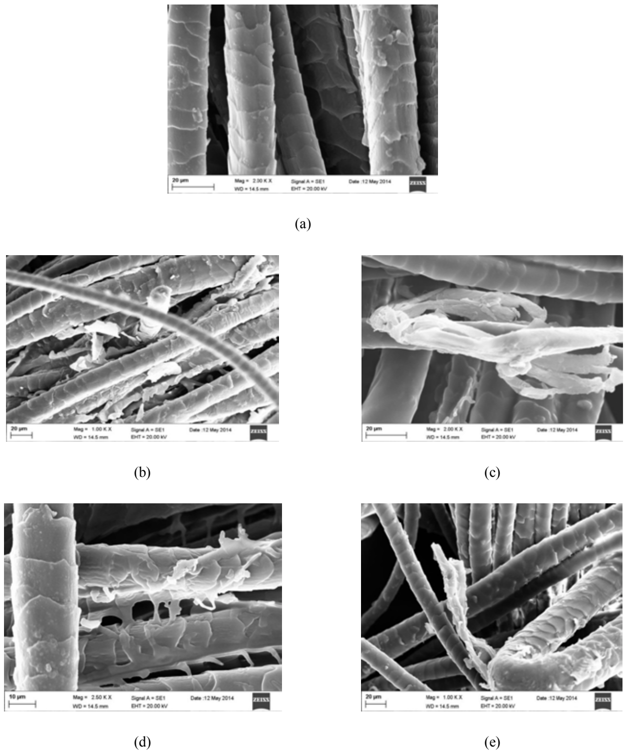


Figure 3: The SEM images of samples ((a) The SEM images of raw wool, (b) The SEM images of sample treated with LF oxygen plasma for 15 min, (c) The SEM images of sample treated with RF oxygen plasma for 15 min, (d) The SEM images of sample treated with LF nitrogen plasma for 15 min, (e) The SEM images of sample treated with RF nitrogen plasma for 15 min).

were compared in terms of the plasma gas, the results demonstrated that the contact angle of samples treated with oxygen plasma was higher than that treated with nitrogen plasma. The reason of this result considered that oxygen gas caused to penetrate more oil on the surface than nitrogen gas because of being more electronegative gas.

According to the results, contact angle of the samples treated with RF plasma was higher than that of the samples treated with LF plasma. The ground for this result was considered that the collusion of RF plasma is higher than the collusion of LF plasma on the surface of wool fabric due to higher frequency of RF plasma. For this reason, it was deemed that the penetrated wax on the samples increased.

Contact angle measurements after the plasma treatment

The hydrophility of plasma treated samples was achieved after the washing to analyze durability of hydrophobic properties of plasma treated samples. The contact angle measurements of plasma treated samples were carried out after washing. The results of contact angle measurements of samples were given in Table 3.

According to the results, the contact angle of plasma treated samples was higher than standard sample after the washing. The hydrophobic properties of plasma treated samples did not significantly decrease after washing when compared to plasma treated samples. Owing to the results, the penetrated wax layer on the wool surface after the plasma treatment was not removed with washing.

Scanning Electron Microscope images

Surface characterization of samples was analyzed with SEM. The SEM images of samples treated oxygen and nitrogen plasma for 15 minutes were shown in the Figure 3.

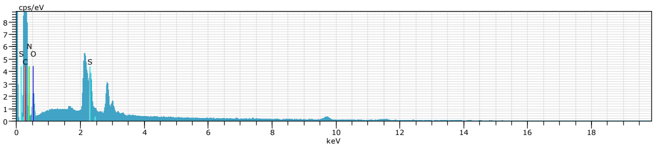
SEM results led to the conclusion that oxygen and nitrogen plasma treatment gave rise to micro cracks in the surface of samples. In addition, the increase in the duration of treatment of plasma application increased micro cracks in the surface.

When the results compared with each other in terms of plasma gas, oxygen plasma treatment caused more damage than nitrogen plasma treatment, which was attributed to higher electronegativity of oxygen compared to nitrogen.

Results of energy dispersive X-Ray spectroscopy

The EDX results of samples treated with oxygen and nitrogen plasma for 15 minutes were given in Figure 4.

According to the results, the amount of carbon decreased with plasma application. The amount of carbon in the sample treated with oxygen plasma is lower than that with nitrogen plasma. Furthermore, the oxygen and nitrogen plasma treatment led to the increase in the amount of oxygen, nitrogen and sulfide in the samples as each type of gas and treatment frequency is deemed to affect the structure of wool differently [27].



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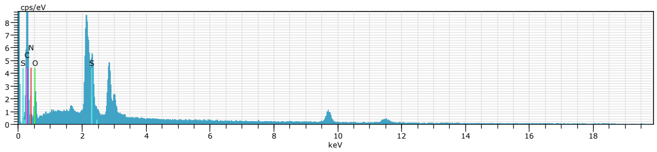
El AN Series unn. C norm. CAtom. C Error
[wt.%] [wt.%] [at.%] [%]

C 6 K-series 61.59 61.59 67.77 18.9
O 8 K-series 23.72 23.72 19.60 7.7
N 7 K-series 12.39 12.39 11.69 4.4
S 16 K-series 2.29 2.29 0.94 0.1

Total: 100.00 100.00 100.00
(a)



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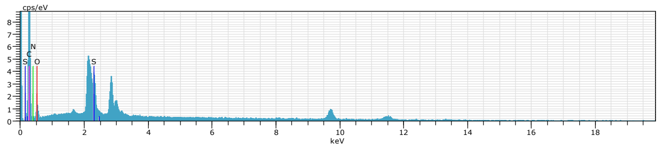
El AN Series unn. C norm. CAtom. C Error
[wt.%] [wt.%] [at.%] [%]

O 8 K-series 42.28 42.28 37.66 14.2
C 6 K-series 31.34 31.34 37.18 10.1
N 7 K-series 23.46 23.46 23.87 8.6
S 16 K-series 2.92 2.92 1.30 0.1

Total: 100.00 100.00 100.00
(c)



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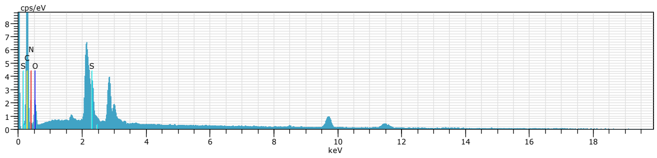
El AN Series unn. C norm. CAtom. C Error
[wt.%] [wt.%] [at.%] [%]

C 6 K-series 43.88 43.88 50.45 14.1
O 8 K-series 36.89 36.89 31.85 13.0
N 7 K-series 16.96 16.96 16.73 7.0
S 16 K-series 2.27 2.27 0.98 0.1

Total: 100.00 100.00 100.00
(b)

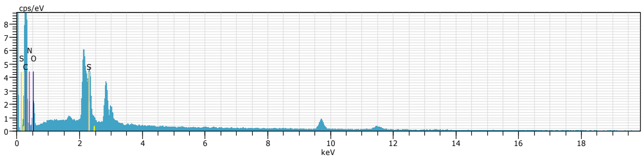


Objects Date:12/5/2014 12:26:00 PM HV:20.0kV Pulsth.:7.44kcps

El AN Series unn. C norm. CAtom. C Error
[wt.%] [wt.%] [at.%] [%]

O 8 K-series 41.60 41.60 36.63 13.7
C 6 K-series 35.74 35.74 41.92 11.3
N 7 K-series 20.30 20.30 20.42 7.3
S 16 K-series 2.36 2.36 1.04 0.1

Total: 100.00 100.00 100.00
(d)



Objects Date:12/5/2014 12:29:42 PM HV:20.0kV Puls th.:7.65kcps

El AN Series unn. C norm. C Atom. C Error
[wt.%] [wt.%] [at.%] [%]

C 6 K-series 41.02 41.02 47.49 13.0
O 8 K-series 36.77 36.77 31.96 12.4
N 7 K-series 19.54 19.54 19.40 7.3
S 16 K-series 2.66 2.66 1.16 0.1

Total: 100.00 100.00 100.00
(e)

Figure 4: The EDX analysis of samples ((a). The EDX analysis of raw wool fabric, (b). The EDX analysis of wool fabric treated with oxygen plasma at low frequency for 15 minutes, (c). The EDX analysis of wool fabric treated with oxygen plasma at radio frequency for 15 minutes, (d). The EDX analysis of wool fabric treated with nitrogen plasma at low frequency for 15 minutes, (e). The EDX analysis of wool fabric treated with nitrogen plasma at radio frequency for 15 minutes).

Conclusion

The use of plasma treatment in textile industry has long been discussed. Plasma treatment provides many advantages in terms of water, energy, chemical consumption and ecological production for textile industry. In this study, wool fabrics were treated with oxygen and nitrogen plasma for different duration of treatment. After plasma treatment, tear strength, contact angle, hydrophility, yellowness, whiteness index and surface properties of wool fabrics were investigated so as to determine the effect of plasma treatment. The results demonstrated that nitrogen plasma treatment led to the increase in the tear strength of wool fabrics whereas oxygen plasma treatment resulted in the decrease in the tear strength of wool fabrics. In addition, oxygen and nitrogen plasma treatment increased the hydrophobicity of wool fabrics, which, in turn, decreased with the increment in the duration of application of plasma treatment. Moreover, nitrogen and oxygen plasma treatment increased the yellowness of samples. The increase in the duration of plasma treatment gave rise to the decrease in the whiteness index of samples. Besides, SEM images demonstrated that nitrogen and oxygen plasma treatment created micro cracks in the surface of wool fabrics. As a result of this study, plasma treatment can be used in textile industry for the modification of textile materials.

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