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## Session Fur & Wool

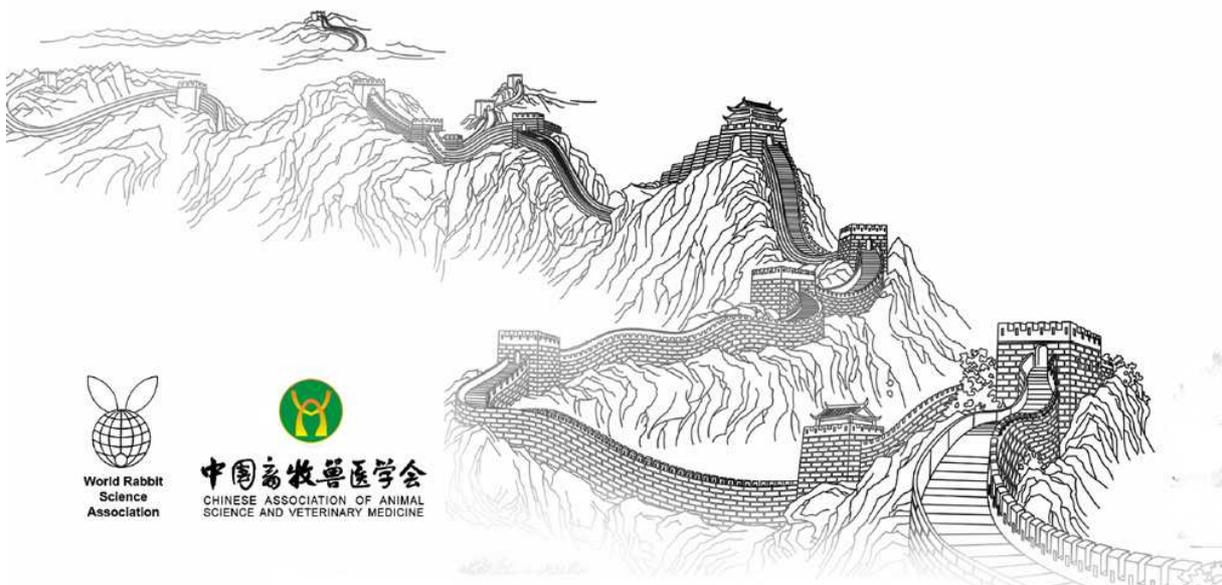
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ON DYEING OF REX FIBERS.

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## THE INFLUENCE OF LOW TEMPERATURE PLASMA ON DYEING OF REX FIBERS

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### ABSTRACT

In this work, the rex fur was treated by low-air temperature plasma and the parameters including the treating time and discharge power were further optimized. Then, the surface morphology and chemical composition were characterized by SEM, XPS. To explore the effects of air low temperature plasma on dyeing properties, the samples, which were treated by air low temperature plasma, were dyed. The results showed the low-air temperature plasma treatment can introduce more active groups such as hydroxyl (–OH), carboxyl (–COOH) in the surface of the rex rabbit fur and improved dyeing rate.

**Keywords:** Rex fibers, Low temperature plasma, XPS, SEM, Dyeing rate.

### INTRODUCTION

At present, low temperature plasma technique (LTP) has already been widely used to modify polymer and textile materials and is usually considered environmentally friendly processes since neither water nor chemicals are used (Ceria and Rombaldoni, 2010). Exposure to a suitable plasma can produce a more reactive surface and desired properties without affecting the basic properties of the bulk material (Karahan and Özdoğan, 2008). Chemical functionalization may also occur consequently to the incorporation of polar groups (hydroxyl –OH, carbonyl –C=O, ester –COOR, carboxyl –COOH, and amino –NH<sub>2</sub>) during the treatment in case of chemically reactive plasma (Shahidi and Ghoranneviss, 2007). The rex fur possesses of good characteristics of shortness, fineness, density, smoothness and beauty. However, there are some technical problems which limited the applications of the fibers in garments and other industries. For example, the surface of rex fibers is hydrophobic in nature which is due to the presence of the hard cuticle on the surface, and this hydrophobicity may give rise to many problems in the dyeing and finishing processes (Demir, 2010).

### MATERIALS AND METHODS

#### Scan Electron Microscope analysis test

The morphology of one untreated sample and one LTP treated sample (5min) was observed with a scanning electron microscope (SEM) using a JSM-7500F SEM (JEOL, Czech Republic, Europe Road Electronics Co., Ltd. Beijing, China) to study the effects of low temperature plasma treatment using a DT-O2S Low-Temperature Plasma Processing Apparatus (Plasma Science and Technology Co., Ltd. Suzhou City Hops, Suzhou, China) on the surface structure of the rex fibers. All the samples were coated with gold before SEM examination.

#### The strength of rex fibers measurement

According to GB/T 3916-1997, YG001A model Single Fiber Electronic Strength Tester (Laizhou Yuan-mao Instrument Co., Ltd. Laizhou, China) was used for measuring the strength of 100 rex fibers.

### XPS analysis test

The chemical compositions of the surface of the fibers were investigated by X-ray photoelectron spectroscopy (XPS) (Kratos, Britain). Measurements were made according the following test conditions: Al / Mg target, high voltage 14.0kV, power 250W.

### Dyeing Processes and measurement

The dyeing process with the acid dyestuffs (FUR RED-NT, anionic dyestuffs; Dye Chemical China Limited, Beijing, China), was carried out in an oscillating sampling machine. The dyeing time was 180 min and the temperature was 60°C. The dye concentrations in each exhaust dye bath were measured at  $\lambda_{\max}$  (506 nm) in a 10 mm quartz absorption cell using a UV1900 spectrophotometer. The percentage exhaustion (E) was calculated according to Equation  $E (\%) = (A_0 - A_t) / A_0 \times 100\%$ .

Where % E is the percentage exhaustion at time t,  $A_0$  is the initial absorbance of dye solution (at 0 minute), and  $A_t$  is the absorbance of dye solution measured at time t (Yang and Li, 2012).

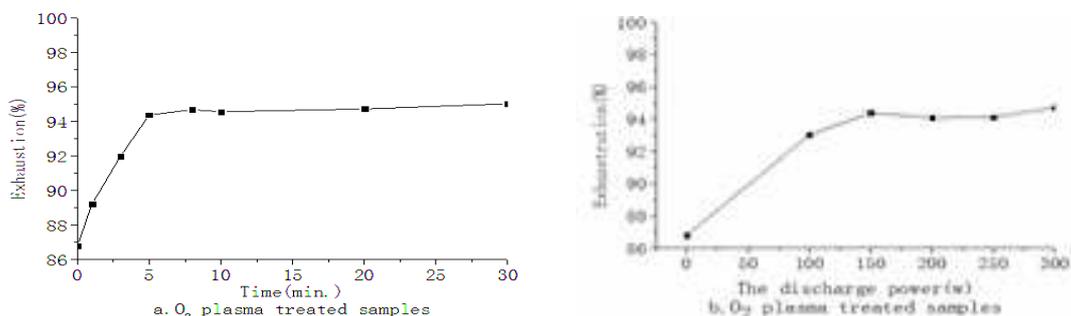
### Color tests

The reflectance of dyed rex fibers was measured on a Color-eye 7000A spectrophotometer using illuminant D65 and a 10° standard observer. Color strengths (K/S) of dyed samples were calculated using the kubelka-munk equation:  $K/S = (1 - R)^2 / 2R$ .

Where R is the observed reflectance, K is the absorption coefficient, and S is the light scattering coefficient.

## RESULTS AND DISCUSSION

### Optimization of low-temperature plasma treatment time and discharge power



**Fig 1:** Dye bath exhaustion changes under different treatment time and discharge power

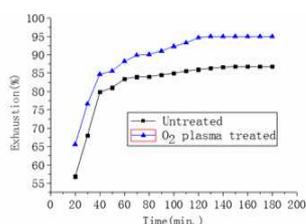
To select optimum treatment time, 8 groups of treatment times were considered as 0, 1, 3, 5, 8, 10, 20, 30 min, respectively. Then the specimens were dyed. Fig 1: a clearly shows that exhaustion rate increase considerably with increasing treatment time (before 10min), but as time continues (after 10min), the bath exhaustion changes slightly. Therefore, the most appropriate treatment time for O<sub>2</sub> plasma is about 5 min. O<sub>2</sub> plasma treatment time was 5 min.

The discharge power of low temperature plasma was arranged for 100, 150, 200, 250 and 300 W (5 treatments), respectively. After dyeing, final bath exhaustion was determined. Fig 1: b illustrates that the exhaustion rate increases considerably with increasing treatment power (50,100,150 W), but thereafter, it changes slightly. Therefore, the most appropriate discharge power for O<sub>2</sub> plasma treatment is 150 W.

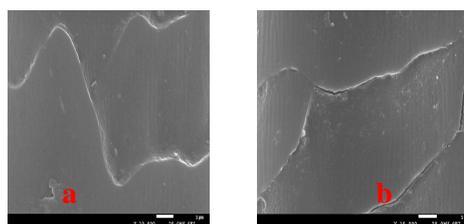
### Study test on dyeing behavior

It can be seen from fig. 2 that the dyeing rate of LTP treated samples is faster than that of untreated samples. This results could be due to the fact that samples treated with oxygen plasma improve etching which can be seen in scanning electron microscopic study.

### The effect of LTP on the surface of rex fibers morphology



**Fig 2:** Dye bath exhaustion of treated and untreated specimens



**Fig 3:** SEM image of sample's surface  
 a. untreated b. LTP treated

It can be seen from fig. 3 that the surface of untreated rex fibers is covered with compact and intact scale and it makes the surface of rex fibers difficult to get wet. However, after the low temperature plasma treatment some scale edges are lightly eroded and rounded.

**Table 1:** The strength of the fibers measurement.

Specimens	Untreated	O <sub>2</sub> plasma treated
Strength (cN)	3.080	2.933

As it can be observed from Table 1, the strength decreases slightly after plasma treatment. This is caused by the etching effect on fibers surface (SEM images) after LTP plasma treatment, but this slight change will not have much effect on the strength performance of the fiber.

### X-ray Photoelectron Spectroscopy (XPS) Measurement

XPS survey spectra enabled us to follow the surface chemical modifications through the atomic content variations. It can be seen from table 2 that the carbon content is significantly reduced after LTP plasma treatment. This reduction is probably due to the etching effect of LTP treatment on the rex fibers resulting in the removal of fiber surface materials. After the etching process, the inner surface of the rex fibers is exposed and also the chemical effect due to the plasma species introduces new functional groups. Both factors contribute together causing a change of the surface composition.

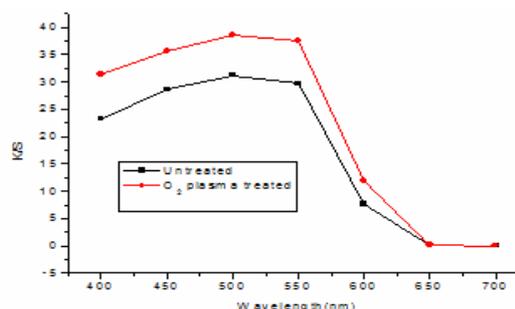
**Table 2:** Elemental analysis and atomic ratio of rex fibers with different plasma gases

sample	Elemental concentration (wt. %)				Atomic ratio	
	C <sub>1s</sub>	N <sub>1s</sub>	O <sub>1s</sub>	S <sub>2p</sub>	N/C	O/C
Untreated	77.74	2.57	18.91	0.78	0.03	0.24
O <sub>2</sub> plasma treated	71.78	5.16	22.85	0.21	0.07	0.33

### Color measurements

The dye-ability of the untreated and treated rex fibers were studied in this research work.

Fig 4 suggests that the K/S of the treated samples are much larger than that of untreated specimens. This may be due to O<sub>2</sub> plasma treatment which can produce more active groups (C=O, COOH and O-H) in the fiber surface, thus greatly improve the bonding ability between anionic dyestuffs and the fibers.



**Fig 4:** K/S values of untreated and LTP plasma treated samples the rex fibers

## CONCLUSIONS

The LTP treatment introduced oxygen and nitrogen functional groups such as –OH, –C=O, –COOH and other reactive groups in the surface of the rex fibers. The increased number of active groups in the surface of the rex fibers could enhance the reaction between dyestuffs and the rex fibers.

## ACKNOWLEDGEMENTS

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