

## DEVELOPMENT OF POLYESTER-WOOL FABRICS DYE ABILITY USING PLASMA SPUTTERING

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**Abstract:** In this paper, the dye ability of worsted (polyester/wool) fabrics after plasma sputtering treatment was investigated. The polyester/wool fabrics were sputtered by copper particles. The natural dye (madder and weld), metal complex and disperse dyes were used for dyeing at the boil point. The Scanning Electron Microscope (SEM) was applied for morphological study and the elemental analysis was followed using the EDX. Results were monitored by using, reflectance spectrophotometer analyses. The results show that, the dye ability of the polyester/wool fabrics after plasma sputtering is improved and this effect is more pronounced for natural dyes. This study may offer a way to achieve better dye ability of polyester/wool blend fabrics.

**Keywords:** Wool-polyester, plasma sputtering, dyeing, natural dye, dye ability.

### 1-Introduction

Polyester/wool is a very popular blend for woven apparel because Wool is an elastic, hydrophilic, and biodegradable protein fiber which can be dyed readily with various dyes, while it is susceptible to alkaline degradation and shows high felting shrinkage during laundering and polyester has outstanding mechanical and physical properties, but its strong hydrophobicity results in low water wet ability and limited dye ability to ionic dyes. Polyester/wool blend fabrics can show the complementary properties compared to pure polyester or wool fibers in terms of crease recovery, mechanical strength, abrasion resistance, fast drying, and dimensional stability[1]. The challenge in dyeing this blend is to achieve satisfactory dyeing of the polyester component. Polyester is normally dyed at 130-135°C, at which temperatures wool is severely damaged and discolored. Lower dyeing temperatures must therefore be adopted and dyes for the polyester component selected very carefully to enable the desired fastness properties to be achieved at the reduced temperature. One solution is to add a “carrier” (a chemical to increase the uptake of dye on polyester) may be added to the dye bath. Another solution is to dye at slightly lower temperature than for 100% polyester, e.g. 120°C, and include in the bath chemicals to protect the wool fibre from excessive damage. Often a combination of the two approaches is used [2]. Therefore disperse dyes of low-or intermediate-energy classes are selected to dye the polyester at the temperature range of 95-102 °C. Neutral-dyeing of 1:2 metal-complex and milling acid dyes are preferred for the wool component. Most milling acid dyes show satisfactory exhaustion and leveling. Chrome dyes are given an oxidative after-treatment that can damage the wool and change the hue of the dyed material [1]. Wang et al investigated the relationship between dyeing conditions and wool damage during one-bath dyeing of polyester/wool blends

and their attempt focus on protecting the wool from damage [3]. Many researchers have also studied on synthesis of the new dyes to dye polyester/wool blend fabrics[4]. Chao et al. synthesized several series of dyes containing carboxyl groups such as anthraquinone acid dyes and mono-azo disperse dyes with which the dye ability difference of the two fibers for the dyeing of polyester/wool blends can be minimized [5]. Dong et al was investigated union dyeing of the photo grafted fabrics using three reactive dyes of  $\alpha$ -bromoacrylamide reactive group [6].

The low temperature plasma (LTP) technique is used widely to modify textile materials. It is regarded as an environmentally friendly process because no chemicals are used [7-14]. Plasma treatment, on the other hand, is a dry and eco-friendly technology, which offers an attractive alternative to add new functionalities such as water repellence, long-term hydrophilicity, mechanical, electrical and antibacterial properties as well as biocompatibility due to the nano-scaled modification on textiles and fiber. Moreover, the bulk properties as well as the touch of the textiles remain unaffected [15-19]. The present work aims to investigate and compare dyeing process of worsted fabric (polyester/wool blend fabric) with untreated worsted fabrics and different times copper sputtering low temperature plasma treated worsted fabrics. Also dyeing of wool with natural dyeing and metal complex dyeing simultaneous were studied. For comprising these methods some technical tests as reflective spectrophotometer, Scanning electron microscope and EDX were used.

## **2-Experimental**

### **2-1- Materials**

Woven polyester/wool (55/45) blend fabrics of 100 g/m<sup>2</sup> obtained from Iran Merinos Co, Iran, were used in this study. For sample preparation, size residue and contamination on the fabrics were removed by conventional scouring processes, which the fabrics were washed with 0.5 g/l<sup>-1</sup> sodium carbonate and 0.5 g/l<sup>-1</sup> anionic detergent solution (dilution ratio to water =1:10) at 80° C for 80 min and then washing was conducted twice with distilled water at 80° C for 20 min and once at ambient temperature for 10 min. Madder (*Rubia tinctorum*, Ronas in persian) and weld (*Reseda Luteola*, Sparak in Persian) were prepared from Khorasan, a providence of Iran, and used in this work as natural dyes, while copper sulfate, acetic acid and citric acid were purchased from Merck. For synthetic dye ability investigation Dianix Rubine CC (C.I.Disperse Red) as a disperse dye and Isolan Red 2S-BR (CI Acid Red 414) as a metal complex dye from Dystar Co were used. Anionic Carrier (Sera Gal PDCR Dystar Co. Germany) was used for disperse dyeing.

### **2-2- Plasma Sputtering Treatment**

Deposition of copper on the surface of polyester/wool fabric samples was performed in the handmade DC magnetron sputtering of Plasma Physics Research Center (Tehran, Iran). Schematic of the system is presented in Figure1. The samples were placed on the anode, and exposed to argon plasma in a cylindrical glass tube. The chamber was evacuated to a pressure of  $2 \times 10^{-5}$  Torr, using rotary and diffusion pumps, and then argon gas was introduced into the

chamber up to a pressure of  $2 \times 10^{-2}$  Torr. Voltage was kept at 2000 V and the discharge current was about 220 mA. The duration of Cu deposition was 3, 5 minutes for different samples. By this treatment, one part of polyester/wool fabric samples was coated with copper.

The untreated and sputtered samples were dyed with both natural and synthetic dyestuffs which are described below.

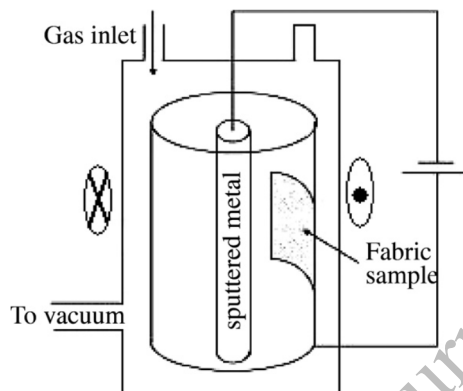


Figure.1. Schematic view of the DC magnetron sputtering setup

### 2-3-Natural dyeing

For natural dyeing, the sputtered samples directly were dyed by dyestuffs but it is necessary for untreated one to be pre-mordanted before natural dyeing.

The untreated samples were dyed as a two-path dyeing with natural dye which were steeped in the mordant bath prepared by 5% (o.w.f.) copper sulfate (pH = 4.5–5.5 adjusted by acetic acid). The bath ratio was 1:30 (1 g of fabric in 30mL of solution). Mordanting of samples was started at room temperature and the temperature was increased with the rate of 2 °C/min to the boil point and maintained for 60 min. Samples were then rinsed with tap water and dried at room temperature. Then the mordanted and copper sputtered and untreated samples were dyed with natural dyes. Natural dyeing was performed in a dye bath with liquor to good ratio of 1:30 that was prepared by 50% (o.w.f.) of extracted natural dye at pH= 4.5–5.5 (adjusted by acetic acid). The madder and weld were used as natural dyes. Dyeing of these samples was started at room temperature and the temperature was increased with the rate of 2 °C /min to boil and heated for 60 min then the samples were rinsed with tap water.

### 2-4-Synthetic dyeing of samples

As it was mentioned before, the untreated and sputtered samples were dyed with synthetic dyes. Disperse dyeing was done with 2 %(o.w.f) disperse dye and 1.5 g/l carrier in pH=5-6 in liquor to good ratio of 1:30 for 60 min to the boil point. The rest of both untreated and sputtered samples were dyed with Metal complex. Metal complex dyeing also was done in pH=6-7 and liquor to good ratio of 1:30 for 60 min to the boil point. The other parts of dyeing were performed in two bath dyeing which were first dyed with metal-complex dye then were dyed with disperse dye in pH=5-6.

## 2-5-Characterization methods

The morphology of the fabrics was observed using a Scanning Electron Microscope (SEM). All of the samples were gold coated before conducting the SEM examination. An EDX unit (Energy Dispersive X-Ray) connected to a SEM microscope was used to determine the percentage of atomic contents of elements present in the treated fabrics and comparing the amount of copper deposited on the surface of treated and untreated sample. Color intensities of dyed fabrics were measured using a UV–VIS–NIR Reflective Spectrophotometer (Varian, Carry 500), over the range of 400–700 nm, and the reflection factor (R) and K/S were obtained. The relationship between reflectance R(%) and color strength was shown in equation (1).

$$\frac{K}{S} = \frac{(1 - R)^2}{2R} \quad (\text{Eq.1})$$

Where K is the absorption coefficient and S is the scattering coefficient.

## 3-Results and Discussion

In this research work as it was mentioned, polyester/wool samples were treated by plasma sputtering technique for different period of times or mordanted using copper sulfate for the samples that dyed with natural dyestuff. After these pretreatments, the prepared samples were dyed using different classes of natural and synthetic dyestuffs and the amount of dye absorption were compared by reflective factor and K/S.

The morphology of the plasma treated, mordanted and untreated samples were analyzed using Scanning Electron Microscopy. The SEM images are shown in Figure 2. As it can be seen, the surface of some of fibers is completely smooth. These smooth fibers are polyester and the other fibers with scaly surface are wool. As it is seen, by increasing the time of plasma sputtering to 5 min, the surface of scales of wool is covered by copper. However this effect about CuSO<sub>4</sub> treated one is not pronounced. The SEM images of dyed polyester/wool fabric with different classes of dyestuffs are shown in Figure 3. Some new small particles can be seen on the surface of fibers that are related to dyestuffs. The morphological changes of dyed plasma treated samples are shown in Figure 4 and 5. As it is seen more amounts of dye particles can be seen on the surface of copper sputtered fabrics.

The results of Reflective Spectroscopy confirm these images achieved by SEM. As it is seen in Figure 6 that is related to madder, by increasing the time of plasma sputtering, the reflectance of the sample after dyeing is decreased. It concludes that the relative color strength (K/S) for 5 min sputtered sample is more than the others. The maximum absorption wavelength for madder dye is around 500 nm, so this area was chosen for the investigation.

The spectrophotometry results related to dyed samples with weld as a natural dye are shown in figure 7. It can be concluded that, plasma sputtering is more effective on natural dye absorption in comparison with mordant treatment. So by plasma sputtering more amounts of natural dyes can be absorbed on the surface of fabrics.

The results of Reflectance and K/S factor for the synthetic dyed samples are shown in Figure 8-10. As it is seen similar results were achieved for synthetic dyed samples and sputtering treatment has very good effect on increasing the absorption of dyestuffs.

It should be mentioned that as it was expected, the average K/S for the natural dyed samples is less than synthetic one. For metal complex dyed samples, it is seen that the average K/S is less as



Figure 2: The SEM images of mordanted and plasma untreated samples with different classes of dyestuffs

Figure 3: The SEM images of dyed treated samples.



Figure 4: The SEM images of plasma treated samples for 3 min after dyeing with different classes of dyestuffs

Figure 5: The SEM images of plasma treated samples for 5 min after dyeing with different classes of dyestuffs

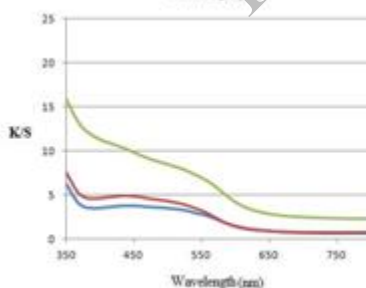
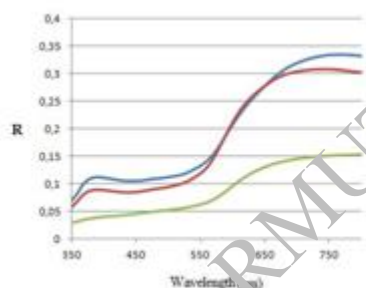


Figure 6- reflective spectra for dyed samples with madder.

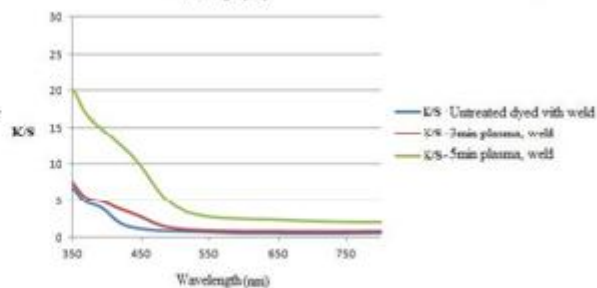
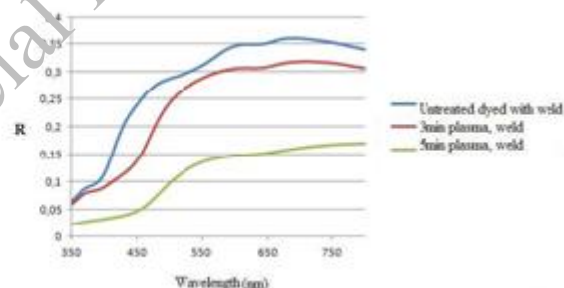


Figure 7: the Reflectance factor and relative color strength for the dyed samples with weld.



compared with disperse dyed samples. It is because of the composition of the used polyester/wool fabric, that, it contains 55% polyester and 45% wool yarns. For the two-bath-dyeing with both disperse and metal complex dyes, it is seen that, the relative color strength for the dyed fabrics is increased. However increasing the time of exposure to 5 min causes more increase in K/S factor.

In the other point of view, the hue of the 5 min plasma treated fabric after dyeing was changed and the maximum absorption wavelength were shifted to lower wavelengths. It is because of more amounts of copper on the surface after 5 min treatment.

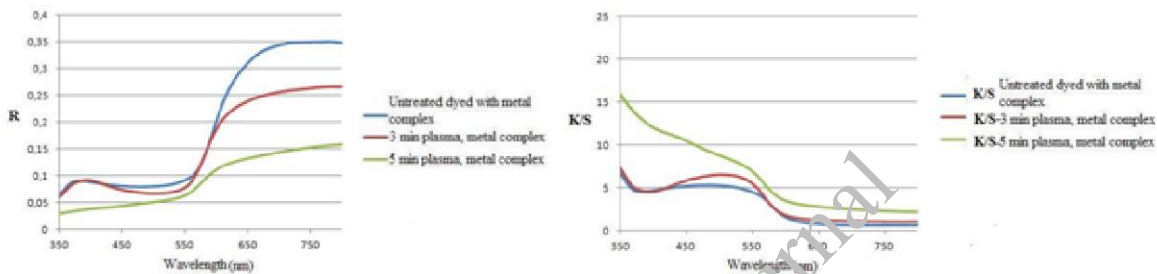


Figure 8: the Reflectance factor and relative color strength for the dyed samples with metal complex dye

The results related to EDX analyze is shown in Figure 11, as it is seen just the amount of copper on the surface of 5 min plasma sputtered samples before and after dyeing is shown. It should be mentioned that, EDX results for all classes of dyestuffs are similar. It is concluded that, the fastness of the copper particles is very good. After dyeing, the amount of copper is decreased but it is not noticeable.

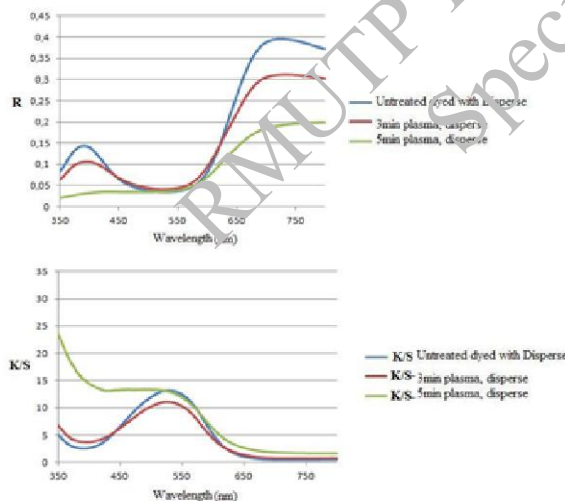


Figure 9: the Reflectance factor and relative color strength for the dyed samples with disperse dye

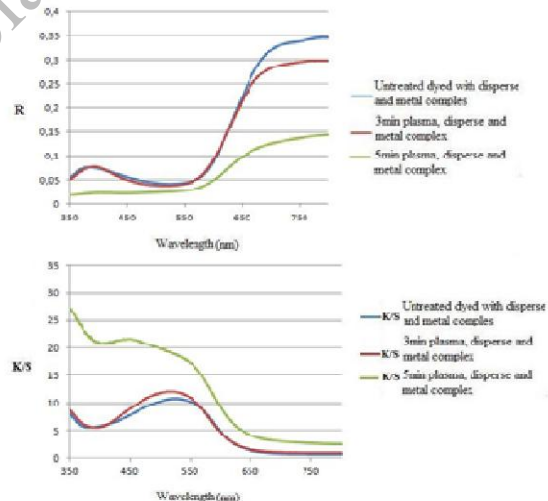


Figure 10: the Reflectance factor and relative color strength for the dyed samples with disperse and metal complex dyes

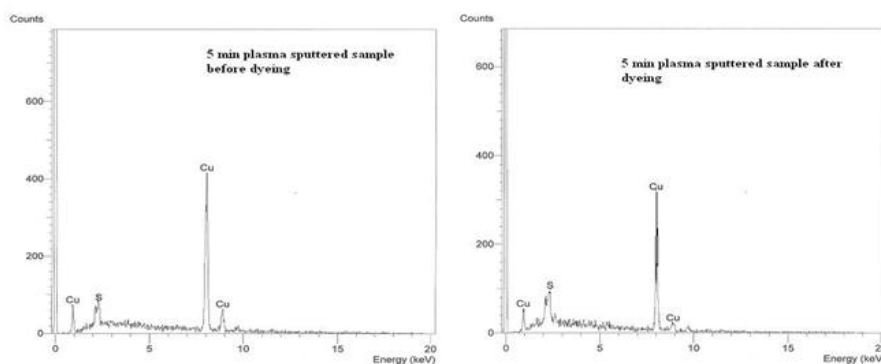


Figure 11: The EDX results for 5 min plasma treated sample before and after dyeing

#### 4-Conclusion

The plasma sputtering treatment was easily used on the polyester/wool fabrics by low temperature plasma. In this research, the dye ability of worsted (polyester/wool) fabrics after plasma sputtering treatment to natural and synthetic dyes was improved. The results that have been obtained from the reflectance spectrophotometer confirm the higher dye absorption of the polyester/wool blend fabric after plasma sputtering treatment and the best results are obtained from 5min plasma sputtering treatment. Also the Scanning Electron Microscope (SEM) was used for morphological study. The morphological changes of dyed plasma treated samples show more amounts of dye particles on the surface of copper sputtered fabrics. EDX results for all classes of dyestuffs are similar and it is concluded that, the fastness of the copper particles is very good. After dyeing, the amount of copper is decreased but it is not noticeable.

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