

## Enhanced Deep Coloring of Micro Polyester Fabric

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Color strength of polyethylene terephthalate (PET) fabrics, which were constructed by microfibers, was can be increased by various pre and after treatment and also changing the condition of dyeing procedure. Alkali and plasma pretreatment on micro polyester fabric leave an insignificant effect on spectral reflectance of the treated colored fabric. However, the liposome dyeing as well as increasing the dyestuff percentage, as a change in dyeing condition, has the highest effects on increasing color strength. The color strength of liposome dyed sample is 24.62 and that of the sample dyed with 3 % (owf) dyestuff is 24.08, both were measured at the maximum wavelengths. The sol-gel coating method has the highest effects on the reflectance spectra of micropolyester fabric which lead to highest Root Mean Square (RMS = 0.052). The reflectance values of SiO<sub>2</sub> coated fabric considerably increased at the middle wavelength of visible spectrum (510 nm up to 710 nm) and besides, it decreased at short and long wavelengths.

*Keywords:* micropolyester, surface modification, alkali pretreatment, sol-gel coating, plasma treatment.

### 1. INTRODUCTION

Polyester fiber (polyethylene terephthalate) becomes more popular due to its unique properties such as excellent mechanical characteristics, good resiliency, chemical inertness and heat resistance. The micro polyesters have been more focal point in research during the past decade because of its aesthetic and highly soft touch [1]. A microfiber is defined as a fiber or filament with liner density of approximately 1 decitex. While polyester microfibers provide excellent hand properties, drape, and comfort, but there are still many problems associated with poor color fastness and uneven dyeing. The methods, dyes and auxiliaries are used for micro-polyester dyeing are as same as conventional polyester fiber [2]. The higher surface reflectance of micro-fiber due to its grater surface area causes the appearance of dyed micro-fibers to be visually paler than conventional fiber. So, it is required more dyes for achieving same visual depth of shade [3].

Polyester fibers reflect a large amount of light from the surface due to their intrinsic large refractive index ( $n = 1.725$ ) and it is difficult to obtain full shaded color especially in black or other dark colors. Recently, many studies have been performed to obtain full color intensity on micropolyester fibers surfaces by alkali treatment [4] low pressure plasma [5], sputter etching [6–8], low refractive index chemical resin coating [9], and liposome dyeing, which improve color strength and reduce dyeing temperature [10]. An increase in color depth upon dyeing was obtained after treating PET fabrics with plasma. This may be easily related to optical effects connected to the plasma-induced increase of surface roughness, which contributes to the increase of  $K/S$  values of dyed PET specimens by decreasing the fraction of light reflected from the treated surfaces respect to more smooth surfaces [11]. Sol-gel process is a method of fabricating solid materials from a chemical solution, mainly inorganic

nonmetallic materials, for several purposes such as preparing low refractive index surface [12], fiber protection, improved water, oil and soil repellence, changing the light absorption and bioactive layer [13].

According to Fresnel's equation, polyester fibers have surface reflectance of 7.1 % at the interface of air and the polymer. It is required to introduce non-reflective coating of resin having low reflective index of about 1.313 that can minimize reflectance in visible wavelength region [8]. One way to achieve a lower index than that of SiO<sub>2</sub> is to introduce voids into the material [14]. If the silicon atom is substituted with four leaving groups, the hydrolysis-condensation reaction will eventually lead to materials with the general formula SiO<sub>2</sub>. This famous chemistry is known as the sol-gel reaction, and it is a method for producing thin films of glass. The present study was performed to reduce the surface reflectance of polyester microfibers by sol-gel treatment and liposome dyeing procedure. The present study was performed to obtain the effect of various pre and after treatments on micro-polyester fabric to reduce the surface reflectance.

### 2. MATERIAL AND EXPERIMENTAL

#### 2.1. Reagents and supplies

Plain weave polyester fabric, which is constructed with microfiber (171 dtex), was employed as a substrate in this study. The chemicals used including Ultravon CN (non-ionic surfactant) and Disperse Blue 56 (C.I. 63285, Ciba-Geigy, Iran contact), tetraethyl ortho silicate (TEOS) and nitric acid (HNO<sub>3</sub>) were purchased from Merck company (Frankfurt, Germany) and Lecithin (Lipoid® S 75, Ludwigshafen, Germany) was received from lipoid company.

#### 2.2. Fabric Preparation

In order to clean the fabrics from the natural and synthetic impurities, they should be scoured. For this purpose, they were immersed in a solution containing

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Ultravon CN (2 g/l) for 20 min at 60 °C, then rinsed and dried. Since polyester fabric needed a surface treatment or modification for improved wetting behavior and adhesion for coating the alkali was carried out on fabric.

### 2.3. Fabric Alkali Treatment

Alkali-treatment followed by immersing the samples in a sodium hydroxide solution which its concentration is 40 % (w/w) at 50 : 1 liquor ratio. The initial temperature was 40 °C and the temperature was raised to boil in 30 min.

### 2.4. Sol Preparation

In order to prepare the sol solutions for dip-coating procedure, the tetraethyl ortho silicate is used as precursors. The suitable solutions were prepared by hydrolysis of TEOS in presence of nitric acid HNO<sub>3</sub> and water H<sub>2</sub>O, for deposition of porous SiO<sub>2</sub> film on polyester fabric. So, the sol was prepared by stirring a mixture of 20 ml TEOS, 85 ml ethanol, 20 ml H<sub>2</sub>O and 3 ml HNO<sub>3</sub> (65 %) for 16 hours and finally, aged for 4 hours at room temperature.

### 2.5. Sol-Gel Coating

Polyester fabric (20 cm × 10 cm) was dipped in the sol solution for 5 min and padded with a wet pickup of 50 %, dried at room temperature for 2 hours and finally, cured at 100 °C for 1 hour in an oven. The flow chart of the present study is denoted in Figure 1.

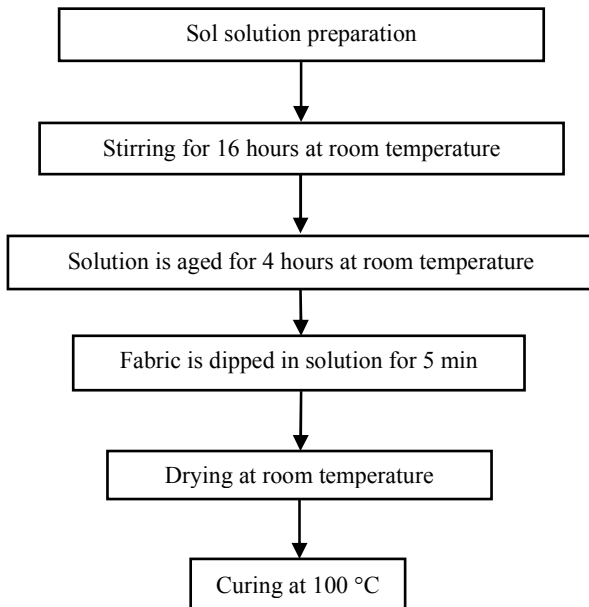


Fig. 1. The flow chart for sol-gel coating process

### 2.6. Plasma treatment

The oxygen gas was chosen for plasma treatment and the samples were treated at two conditions as mentioned in Table 1.

### 2.7. Dyeing procedure

All the dyeing procedures were carried out at pH 5.5. Polyester fabrics were dyed in a laboratory-scale dyeing machine. Dyeing liquor ratio (L : R) of 50 : 1 and the final

temperature was 120 °C. The liposome-dye solution was prepared by preparing the dye solution and then added to the lipid solution (0.5 w/w), and the resulting colored solution was stirred for 30 min.

Table 1. The plasma treatment conditions

Sample	Power, W	Gas flow, cm <sup>3</sup> /s	Pressure, Pa	Time, s
Low Intensity	100	1.67	10	60
High Intensity	150	1.67	20	60

### 2.8. Color Measurement

The reflectance spectra of the samples were measured by Color-Eye spectrophotometer from Gretag Macbeth in the visible region.

The CIE terms namely, L\*, a\*, b\* and C\* color-coordinates under illuminant D<sub>65</sub> and 10° standard observer were measured for evaluating the color of samples.

Color strength as (K/S) values calculated from the reflectance factor at the wavelength of maximum adsorption ( $\lambda_{\max} = 600$  nm). Changes in surface reflectance of treated sample comparing with the untreated one were expressed as the Root Mean Square (RMS) calculated by the following equation:

$$R_{RMS} = \sqrt{\frac{1}{N} \sum_{i=1}^N (R_{i,p} - R_{i,t})^2}, \quad (1)$$

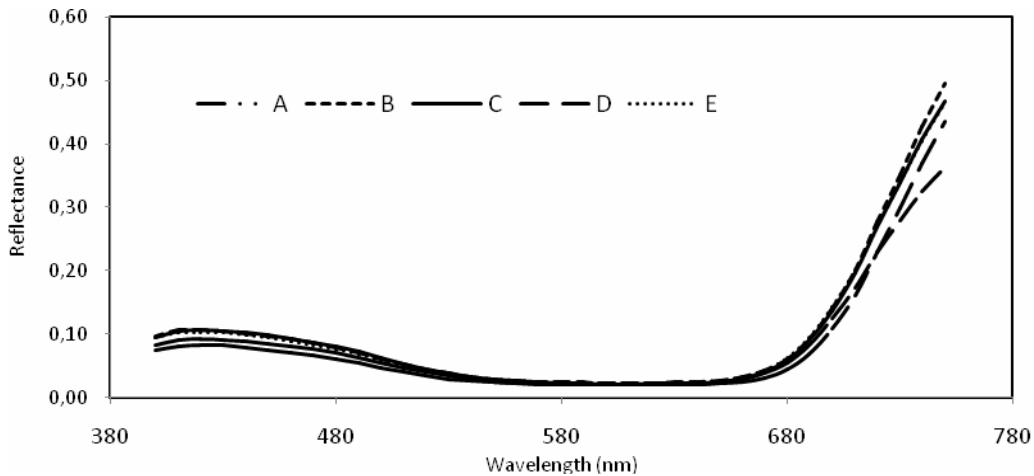
which,  $R_i$  indicates the reflectance factor of  $i$ -th wavelength, and indices  $p$  and  $t$  refer to pristine dyed sample and treated sample, respectively. Also,  $N$  is the number of wavelength which in this research is equal to 36 nm for 10 nm wavelength intervals in the visible spectrum 400 nm – 750 nm.

## 3. RESULTS AND DISCUSSIONS

In order to reduce the surface reflectance of micro-polyester fabric various treatments such as pretreatment, after treatment, and changing the method of dyeing were applied on the micro polyester fabric. The two pretreatments procedure such as alkali treatment and plasma treatment were carried out. Beside, the dyed micro polyester fabric was coated with silica as an after treatment. Increasing the dyeing percentage and liposome dyeing procedure was used as changing the condition of dyeing process for reducing surface reflectance.

Figure 2 shows the reflectance spectra of all dyed samples. It can be inferred from the figure that the pre-treatment procedures and also change in dyeing method result in decreasing the reflectance spectral values. As the results of procedures shown in Fig. 2, it can be seen that the reflectance values decreased bellow 540 nm and above 630 nm, while the reflectance factors are roughly equals for the wavelengths approximately from 540 nm to 630 nm.

Among these treatments for achieving deep coloring, the liposome dyeing method and increasing the dyestuff percentage have the highest effect on the surface reflectance spectra of the micropolyester fabric. Applying liposomes in dyeing procedure can lead to increase the color strength of dyed sample, which means that the dyed



**Fig. 2.** Reflectance spectra of dyed sample using 2 % (owf) Disperse Blue 56 and pretreated by: A – liposome dyeing sample; B – alkali pretreatment; C – untreated sample; D – dyed by 3 %; E – pretreated by plasma

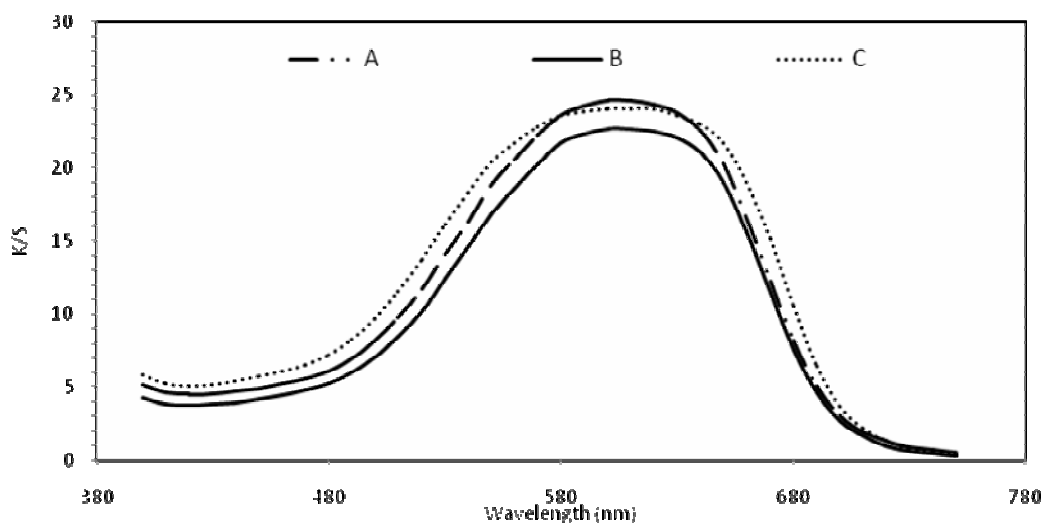
sample has lower reflectance spectra [10]. The liposome gradually converted to small particle size at above the 85 °C and lead to coating the surface of polyester fibers with a layer of phospholipids, which leads to decrease the amounts of reflectance spectral values [15 – 16].

The surface reflectance spectral values of the dyed micropolyester fabric are slightly reduced by the alkali and plasma treatments, however, their effects are insignificant as their reflectance spectra in all wavelengths are nearly coincided. The plasma procedure can leave an etching effect on the surface of fibers which leads to light trapping effect that decreases the reflectance values [7, 8]. According to the obtained results of this research, the power of applied plasma might be lower than that of effective value. That is why it did not leave a significant etching effect on the fibers and then it slightly reduced the reflectance factors.

Increase the percentage of dyes in dyeing bath, results in reducing the reflectance spectral values of the micro-polyester fabric and then increasing the color strength of dyed sample (Fig. 3). Furthermore, liposome dyeing

reduces the reflectance as well as using higher percentage dyestuff in dyeing bath solution. At liposome dyeing method the color strength of dyed sample reached to 24.62 which are 8.5 % higher than untreated sample at the maximum wavelength. Increasing dyestuff to 3 % gives rise to improve on color strength, which is reached to 24.08 and this value is 2.4 % less than liposome dyeing method. So, liposome dyeing could be a suitable method for conventional dyeing procedure which leads to use of lower percentage of dyestuffs.

Figure 4 shows the effects of after treatment on reflectance spectra of the micro-polyester fabric which is dyed by 2 % (owf) Disperse Blue 56. It can be inferred from the figure that the consequences of this procedure are different from the pretreatment and change in dyeing method. According to the results shown in Fig. 4, the silica coating reduced the reflectance factors only at limited range of wavelengths, approximately beyond from 510 nm up to 710 nm. Preparing a non-reflective coating on polyester fabric requires a resin coat having low reflective index about 1.313 that can minimize reflectance values in



**Fig. 3.** *K/S* spectra of sample: A – liposome dyeing sample dyed by 2 % (owf); B – untreated sample dyed by 2 % (owf); C – dyed by 3 % (owf) Disperse Blue 56

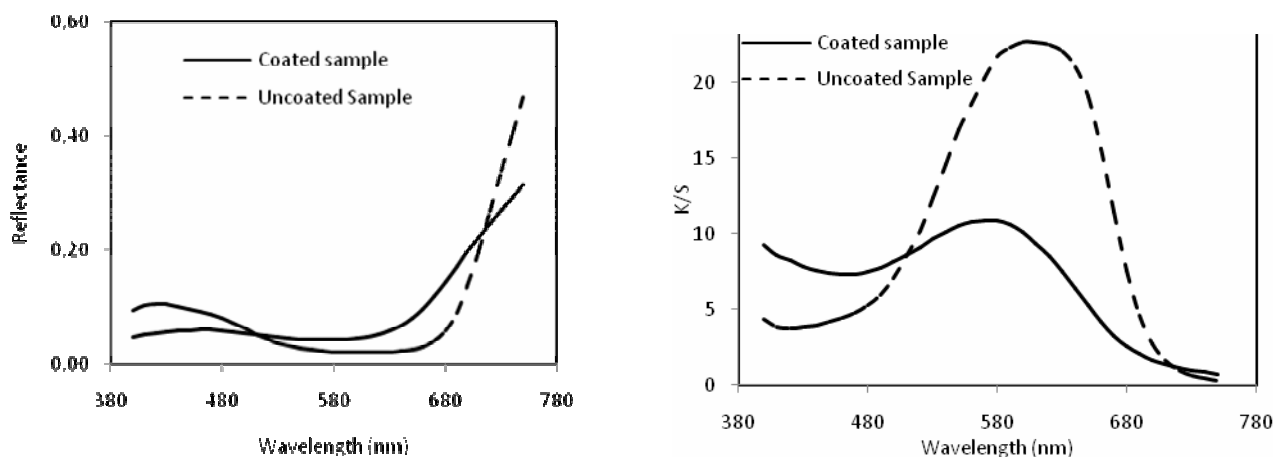


Fig. 4.  $K/S$  and reflectance spectra of dyed micropolyester fabric using 2 % (owf) Disperse Blue 56

the visible wavelength region [8]. So, it cannot be achieved a non-reflective coating only by a single layer [17–18].

Coating procedure changes the reflectance spectra significantly. The reflectance factors decreased at wavelengths lower than 510 nm and increased at upper wavelength, namely 510 nm up to 710 nm by coating procedure. It would be related to the characterization of employed material for coating in which the thickness layer and refractive index are of important considerations to predict the optical behavior of coat layer. The coated fabric which was dyed by 2 % dyestuffs has the less color strength than the untreated sample from 510 nm up to 710 nm, which is beyond of this range, the coating method increases the color strength.

The RMS values of reflectance spectra, chroma, color difference, lightness, color strength and hue of prepared samples are presented in Table 2. The coated sample which was dyed by 2 % (owf) Disperse Blue 56 has the highest amount of RMS (0.052) among the listed sample. So, the coating procedure shows significant effect on changing the reflectance spectra than the other applied method. Coating procedure can enhance the lightness of dyed sample, which is reach to 26.37, while this value for the pristine dyed sample is equal to 22.65. Moreover, this sample has the lowest chroma (saturation) and cause to achieve the duller shade, while it causes to great hue differences and its color strength at maximum wavelength ( $\lambda_{max} = 600$  nm) is lower than the other samples which it means only by one layer coating cannot achieve higher color strength at all wavelengths.

Figure 5 shows the  $K/S$  spectra of all coated sample which can be inferred that there is a same trend for coating

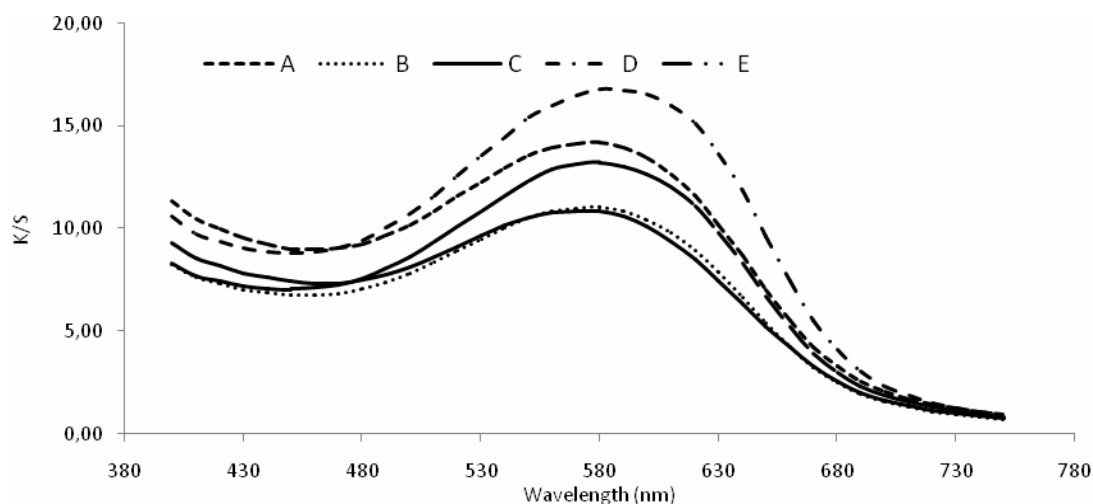
sample as there was for uncoated sample. The coated sample, which is dyed by 3 % (owf) Disperse Blue 56 has the highest color strength among the all samples.

Figure 6 shows the reflectance spectra of pristine micropolyester fabric, which was dyed by 2 % (owf) Disperse Blue 56, coated liposome dyeing sample, and coated micro-polyester fabric which was dyed by 3 % (owf) Disperse Blue 56. The reflectance spectra of coated sample decrease by increase the wavelength up to 510 nm, after this wavelength it is increase up to 710 nm. The spectra of coated samples differs from the uncoated ones and also the spectra of coated liposome dyeing sample is less than untreated sample in all wavelengths, exactly like what presented for uncoated samples in Fig. 2. However, it effects on lowering the reflectance spectra is as well as the coated sample which was dyed by 3 % (owf) Disperse Blue 56. Liposome dyeing method was increased the reflectance spectra more than increasing dye percentage method in 510 nm to 710 nm.

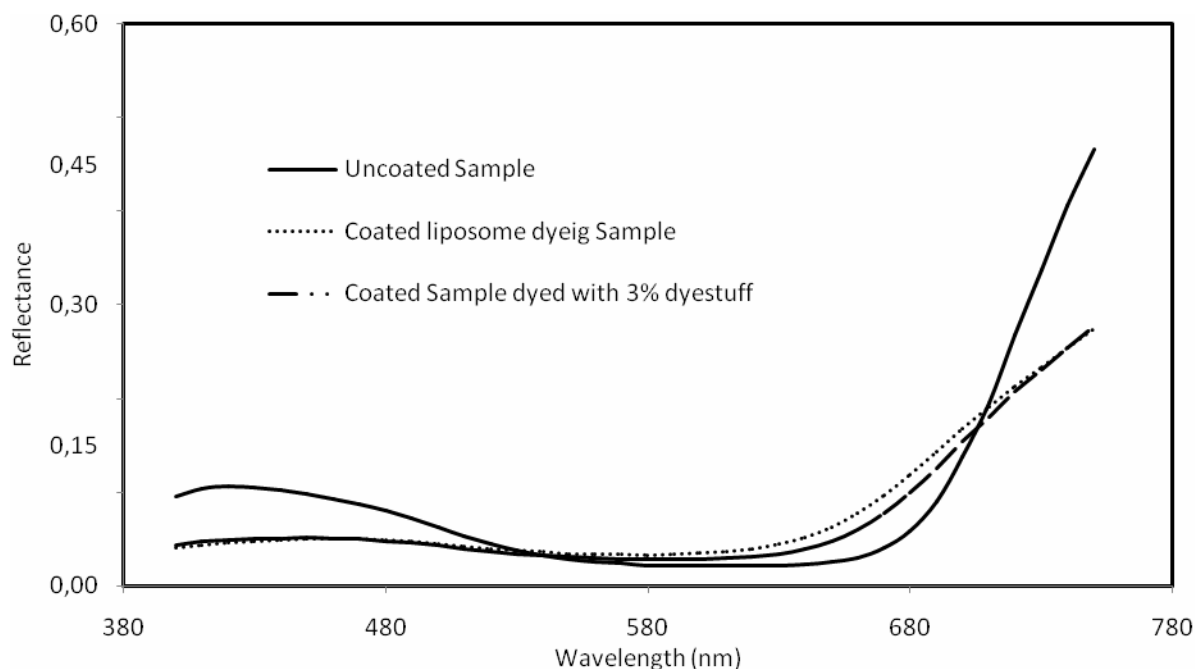
To study the effect of coating layer on color appearance of the prepared samples, color specifications of the specimens were calculated and listed in Table 3. It can be inferred from Table 3 that coating procedure gives rise to decreasing chroma of color. The root men square of reflectance spectra of coated sample are nearly close to each other, and their chroma decreased significantly than the standard sample. Among these samples the coated the pristine sample dyed with 2 % dyestuff and coated liposomes dyeing with 2 % dyestuff have the lowest amount of chroma. In addition the Coated the pristine sample dyed with 2 % dyestuff has the highest color differences and difference hue.

Table 2. RMS values of reflectance spectra, color difference and hue, and chroma of dyed sample using 2 % (owf) Disperse Blue 56

Sample	RMS	$\Delta E$	C*	L*	$\Delta H^*$	$K/S$
Untreated sample	–	–	24.420	22.65	–	22.69
Alkali pretreatment	0.007	1.01	23.780	22.92	0.7402	20.61
Liposome dyeing	0.026	2.22	22.700	21.24	0.0495	24.61
Dyed by 3% dyestuff	0.020	4.79	21.360	20.06	1.4848	24.08
Plasma pretreated	0.003	0.99	23.874	22.31	0.7625	22.06
Coated sample	0.052	20.26	5.494	26.37	6.2360	10.09



**Fig. 5.** *K/S* spectra of coated sample dyed using 2 % (owf) Disperse Blue 56 and pretreated by: A – liposome dyeing sample; B – alkali pretreatment; C – untreated sample; D – dyed by 3 %; E – pretreated by plasma



**Fig. 6.** Reflectance Spectra of micro-polyester fabric dyed using 2 % (owf) Disperse Blue 56

**Table 3.** The RMS values of reflectance spectra, color difference and hue, and chroma of dyed and coated sample using 2 % (owf) Disperse

Sample	RMS	$\Delta E$	$C^*$	$L^*$	$\Delta H^*$
Untreated sample	–	–	24.42	22.65	–
Alkali pretreatment and coated	0.050	18.423	7.029	26.44	4.782
Liposome dyeing and coated	0.056	19.025	5.952	23.24	4.557
Dyed by 3% dyestuff and coated	0.054	15.983	8.533	21.73	1.553
Plasma pretreated and coated	0.051	15.969	8.916	24.60	3.321
Coated sample	0.052	20.266	5.494	26.37	6.236

## CONCLUSIONS

Deep color and color strength of micropolyester fabrics were enhanced by pre and changing the dyeing procedure such as liposome dyeing. Liposome dyeing method increases the color strength of dyed sample to

24.62 which is 8.5 % higher than untreated sample at the maximum wavelength. Although, increasing dyestuff lead to increase the color strength, while in liposome dyeing by lower dyestuff can reach to higher color strength at maximum wavelength. Plasma and alkali treatment have

very low effect on reducing the reflectance spectra which lead the root mean square of reflectance spectra reach only to 0.007 and 0.003 accordingly. The silica coating as an after treatment method causes to reduce reflectance spectra significantly which the root mean square reached to 0.052 and the chroma reduce significantly.

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