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Original

Surface activation of polyester fabric using remote plasma: Dyeability with disperse dye properties

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Abstract: The effect of oxygen plasma activation of polyester fabric surface on the dyeability with a dispersed dye was investigated. A scanning electron microscope (SEM) was used to examine changes in fiber surface morphology, and an infrared spectrometer (FTIR) was used to analyze alterations in its chemical composition. Furthermore, the impact of the treatment on the fabric's tearing strength was evaluated. The treated samples were dyed with a dispersed dye at 100°C and atmospheric pressure, and another untreated sample was dyed in the conventional way using an autoclave device at high temperature and pressure (125°C, 0.165 MPa). The color strength of the dyed samples was analyzed using a spectrophotometer. Subsequently, the samples were subjected to a washing-fastness test for both treated and untreated samples. The results revealed that the treated samples had higher color strength and better washing fastness than the untreated samples.

Keywords: **Activating, Dyeability, Hollow cathode plasma, Polyester fabric, Wettability.**

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1 Introduction

The textile industry faces numerous environmental concerns during the finishing process, which involves applying treatments to fabrics to enhance their characteristics. The conventional chemical-bath method has drawbacks, including environmental damage and substantial water and energy use. As a result, there is a growing demand for alternative, eco-friendly technologies for fabric treatment (Naebe et al., 2022; Zouari et al., 2021). Plasma technology is an effective method to modify the surface properties of polymers that are hard to modify through conventional methods (Profili et al., 2020; Su et al., 2019), this method is environmentally conscious, simple to execute, cost-effective, completely free of water waste, and does not generate any contaminated water (Mohamed & EL-HALWAGY, 2021; Yang et al., 2019), and one of its most impressive features is its ability to modify the surface of the substance to achieve specific properties without affecting its bulk characteristics (Bhat et al., 2011; Kulkarni, 2019; Saloum et al., 2021)

Plasma can be classified into two types: hot and cold. For modifying textile materials and treating polymer surfaces, cold plasma is appropriate (Saloum et al., 2019a; Saloum et al., 2019b). Two methods can be employed to create cold plasma: low-pressure and atmospheric-pressure. Although both techniques are effective for altering polymer surfaces, low-pressure plasma is considered more effective due to its highly concentrated active particles and superior chemical selectivity. This method is frequently employed to modify the surfaces of diverse materials, improving their properties, such as wettability, roughness, and adhesion. It is typically implemented within a pressure range of 0.01 to 10 mbar (Yilma et al.,2020; Yilma, 2021). The application of an electric field to a gas can form plasma, which can then interact with polymer surfaces, leading to a range of modifications in their properties. These alterations encompass both physical and chemical transformations, as the high-energy electrons in the plasma can break down the double and triple bonds within the polymer (Klébert et al., 2021; Petkevičiūtė et al., 2022; Zaidy et al., 2019). This process leads to the emergence of free radicals and novel functional groups as a result of the activation process of the fabric surface (Samei et al., 2022; Zhang et al., 2017).

Polyester is a widely used synthetic fiber in the textile industry today. However, one limitation is its low surface energy, which can lead to poor wettability and dyeability. This is because polyester is chemically unreactive and highly crystalline, with limited amorphous regions. To address this issue, dispersed dyes are utilized for polyester dyeing at high temperatures (125°C) and high pressure. During this process, the fiber's amorphous regions swelled, allowing the dyes to penetrate. However, this process requires significant energy and specialized equipment to carry out effectively (Kim et al., 2021; Salem & Morgan, 2014).

Numerous studies have demonstrated the efficiency of plasma technology in enhancing the wettability of polyester fabric surfaces. Kim used atmospheric-pressure air plasma to treat polyester fabric before applying dispersed dyes at high pressure and 125°C. This method effectively augmented the number of COOH groups on the fiber's surface, enhancing its wettability and dyeability. However, he did not examine the color fastness of the dyeing process (Salem & Morgan, 2014). Also, using atmospheric-pressure helium plasma technology, Agrawal and Jassal successfully treated polyester fabric to produce nanogrooves measuring less than 200 nm on the fiber surface. This treatment improved the surface energy and wettability of the fibers through surface-level chemical modifications. However, due to the high cost of helium in the industrial setting, it would be beneficial to explore the use of a more affordable, readily available gas for treatment (Samanta et al., 2009). In addition, Salem utilized air and oxygen plasma at atmospheric pressure to modify the surface of polyester fabric. Following plasma treatment, the fabric was subjected to acid treatment, resulting in improved hydrophilicity and increased capacity for acidic dye absorption. Furthermore, the color strength of the fabric was increased after this treatment (Kim et al., 2021).

However, the studies on improving the wettability and dyeability of polyester fabric are few, and the researchers used plasma technology at atmospheric pressure for this treatment, they did not study the use of low-pressure plasma technology in polyester fabric treatments, nor did they dye the fabric at temperatures lower than 125°C, and in general, studies about the use of low-pressure plasma in fabric finishing to increase their wettability and dyeability are few. Therefore, in this study, pre-treatment of polyester fabric was carried out using *RF* (radio frequency) low-pressure remote hollow cathode plasma system, with different gases, at different treatment times, to demonstrate the possibility of using this plasma system to increase the effectiveness of dyeing polyester fabrics with dispersed dyes at a temperature lower than 125°C.

2 Materials and methods

2.1. Materials

Polyester fabric was used, weighing 85 g/m^2, with a yarn density of 40 warp/cm and 20 weft/cm. O_2 gas was used as the treatment gas with a purity of 99.99%. Disperse dye (C.1. Disperse Orange 30) was used for the fabric dyeing.

2.2. Experimental Procedure

The fabric was treated with O_2 plasma using a remote hollow-cathode plasma, dyed with a dispersed dye, and tested. The surface morphology was tested using a scanning electron microscope Vega II XMU Teskan SEM at a magnification (× 20.000), and the chemical composition of the surface fibers was examined using Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR Thermo Nicolet 6700), as well as the wicking property was determined according to AATCC TM 197 (1997), the tear strength was tested using Elmatear device, and the weight loss after plasma treatment because of the activation process was determined using the equation Eq. (1):

$$\text{weightloss}(\%) = \frac{W1-W2}{W1} \times 100 \quad (1)$$

Where W1 is the initial weight of the sample, W2 is the sample weight after treatment.

In addition, the color strength of the dye was studied using K/S values on a spectrophotometer (Triax-550-Jobin Yvon), RGB Measure, and the ImageJ program's grey scale. Finally, dye fastness to washing was investigated according to ISO 105 C01.

2.3. Plasma treatment

The samples were treated using the remote hollow cathode plasma (RF, 13.56 MHz) HCD (Remote Plasma Consult GmbH, PlasCon HCD-L 300 System), as shown in Fig. 1 and described in another paper (Saloum et al., 2021), with O_2 gas. The plasma system consists of a cathode and an anode, with 30 jets that provide plasma media. The chamber, with dimensions of 50 x 50 x 50 cm^3, is evacuated to 5 x 10^{-4} mbar using vacuum pumps before the treatment. The treatment gas is then introduced at 50 sccm, 100 W, and 0.16 mbar. During this process, the sample is placed on the holder under the plasma source. Three treatment times were used: 5, 10, 15 minutes. After the treatment, the samples were collected and dyed.

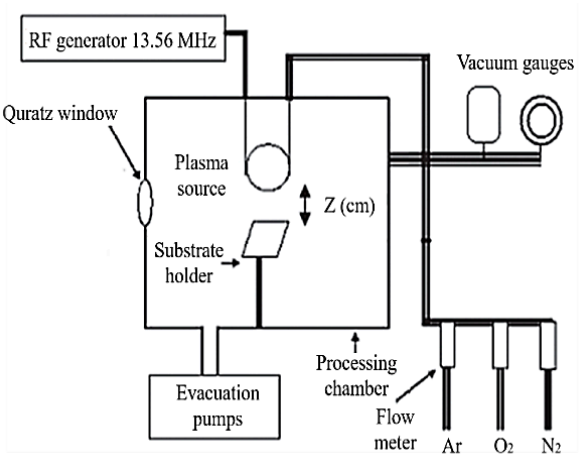


Figure 1. The low-pressure RF remote plasma system.

2.4. Dyeing process

The dyeing process for the treated samples was performed using 1% of the dispersed dye at 100°C, with a bath ratio of 1:100 as follows: The samples were immersed in the bath dye at room temperature, then the temperature was raised by 2°C/min till 100°C, where the dyeing process took 30 min at this temperature. After that, the samples were rinsed with distilled water and dried in the ambient air.

The conventional dyeing for one untreated sample was done using (YX – 28 B) Autoclave device at high temperature, pressure, and power (125°C, 0.165 MPa, 1600 Watt), with the same dye concentration and bath ratio of the dyeing process for the treated samples, and the same dyeing time (30 min).

Results and discussion

3.1 Surface morphology

Fig. 2 shows the morphology of the surface fibers after the treatment at a magnification of ×20.000, where the sample surface was coated with carbon. The coating process was performed in the EMITECH k975X system, which contains two graphite rods that are subjected to high stress in the vacuum chamber. The graphite evaporation coats the samples, and this process takes about 5 × 10 ms; then, the samples were tested using Vega II XMU Teskan SEM.

The untreated sample had a smoother fiber surface with some nanospots, which increased slightly after

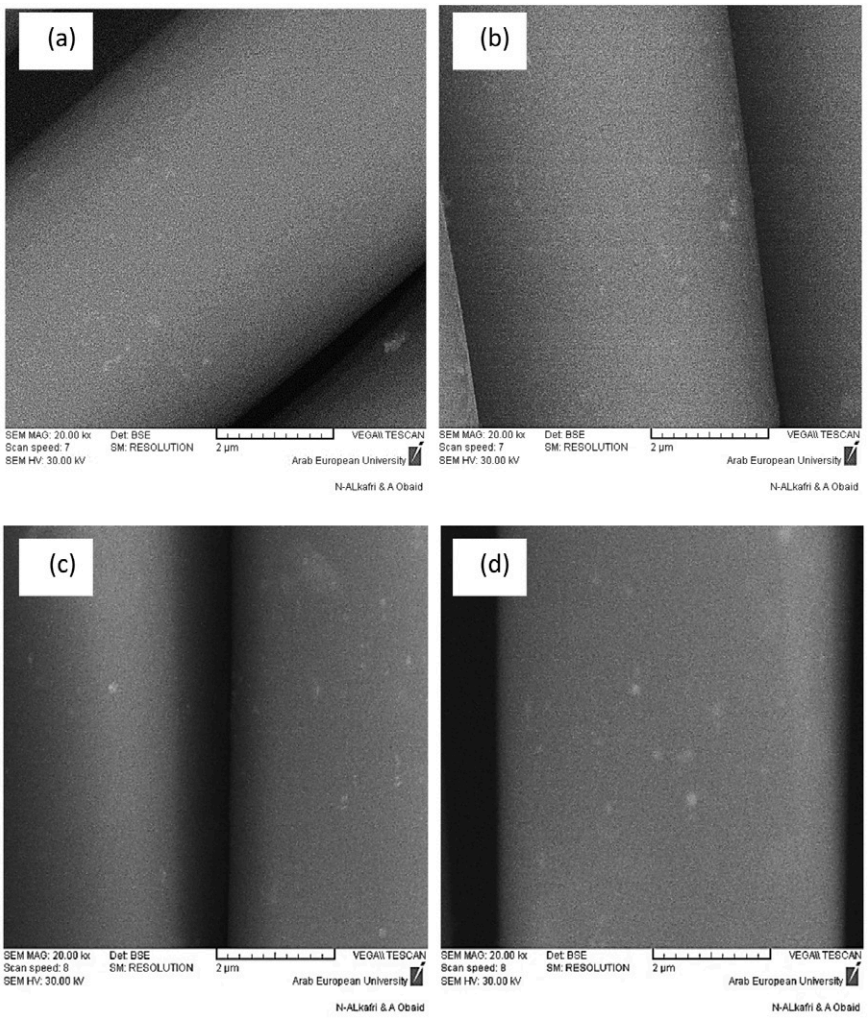


Figure 2. The morphology of the samples: (a) untreated; (b) 5 min plasma-treated sample; (c) 10 min plasma-treated sample; (d) 15 min plasma-treated sample.

plasma treatment. However, there are no significant morphological changes compared with the untreated one; in addition, no difference in fiber surface with respect to treatment time. The increase in nano-spots after treatment can be explained by the interaction of ions in the plasma medium with the fabric surface (Kim et al., 2021). This is consistent with the results of Molina's study (Molina et al., 2015), which showed that the white spots on the surface of the polyester fiber increased slightly after plasma treatment.

3.2 Chemical structure of surface fibers

The chemical formula of the polyester fiber is illustrated in Fig. 3. The fiber's surface is particularly water-repellent thanks to the presence of ether bonds (C-O-C) that constitute its surface. The fiber's core, on the other hand, contains hydrophilic ester oxygen (C=O) (Senthilkumar & Karthik, 2016). To analyze changes in functional groups on the sample surface, Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR; Thermo Nicolet 6700) was employed, with Fig. 4 showing the FTIR spectra of both the untreated and 15-minute-treated samples. Table 1 provides a comprehensive list of the bands and functional groups that are present in the samples.

The spectra of the treated and untreated samples appear similar, but there are some differences. The treated sample shows an increase in the peak at 1340.306 cm-1, indicating a strengthening of the C-O bond. Additionally, there is a slight increase in the peak at 3764.426 cm^{-1}, revealing an increase in the stretching vibration peaks of free O–H radicals (Dai et al., 2023). This suggests an increase in the treated sample's hydrophilicity.

However, there are no new bonds after the treatment, that that O_2 plasma treatment can explain only affects the surface of the sample without affecting its bulk and does not affect the benzene rings, so only the carbonyl, hydroxyl, and ether oxygen bonds on the surface are affected by the plasma treatment (Kan & Man, 2018; Senthilkumar & Karthik, 2016).

Due to the influence of oxygen plasma, active species of plasma can be produced as Eq. (2-7) (Kan & Man, 2018):

Ionization:

$$e^- + O_2 \rightarrow O_2^+ + 2e^- \quad (2)$$

Atom and radical formation:

$$e^- + O_2 \rightarrow O + O^* \quad (3)$$

Heat formation:

$$e^- + O_2 \rightarrow O_2^* + 2e^- \quad (4)$$

$$O_2^* \rightarrow h\nu \quad (5)$$

$$e^- + O \rightarrow O^* \quad (6)$$

$$O^* \rightarrow h\nu \quad (7)$$

It has been discovered that in O_2 plasma present in the hollow cathode discharge reactor, the amount of atomic oxygen flowing toward the sample is considerably higher than the flow of oxygen ions. Therefore, it can be concluded that atomic oxygen (O) plays the primary role in modifying the polymer surface in O_2 plasma (Saloum et al., 2019).

To know the increased amount of the functional groups, FTIR ratio for the untreated and treated samples was investigated by taking the aromatic C-H out-of-plane vibrations to normalize C=O, C-O, and –OH, and the results are shown in Table 2, which showed some intensity differences between the untreated and treated samples, where it can be observed that the reflectance ratio in the treated sample is higher than the untreated sample. This

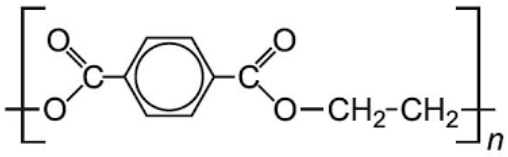


Figure 3. The formula of polyester fiber.

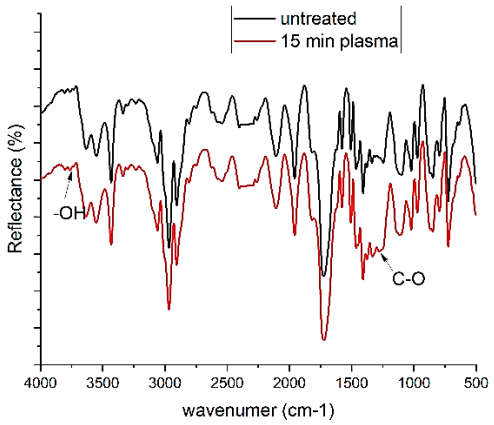


Figure 4. FTIR spectra of the untreated and 15-minute treated sample.

indicates the formation of more polar groups on the sample surface, making the treated sample more hydrophilic than the untreated one.

3.3 Wettability

Water contact angle (WCA) was conducted using the OCA 15PLUS device according to ASTM D5946 standard by dropping a drop of distilled water (5 µl) on the sample surface from a height of 5 mm and then calculating the contact angle using a camera. Five measurements were taken for each sample, and the average was calculated.

Table 1. FTIR peak assignments for the untreated and 15-minute treated samples.

Bond	Wavenumber (cm^{-1})		Reference
	untreated	15 min plasma treated	
C=O	1720.219	1729.862	[12, 23]
C-O	1243.881-1336.448	1263.166-1340.306	[12, 17]
Aromatic ring	1409.731	1411.66	[12]
O=C-O-C ester	1022.104	1020.175	[12]
Aromatic C-H	725.1149	721.2579	[12]
-OH	3436.582	3432.725- 3764.426	[23, 24]

Table 2. The reflectance ratio on the sample surface.

Untreated			15 min plasma treated		
$R_{1243.881}/R_{725.1149}$	$R_{1720.219}/R_{725.1149}$	$R_{3436.582}/R_{725.1149}$	$R_{1263.166}/R_{721.2579}$	$R_{1729.862}/R_{721.2579}$	$R_{3432.725}/R_{721.2579}$
0.661	1.645	0.809	1.001	1.898	0.952

R= Reflectance

The results are in Fig. 5, where the WCA for the untreated sample was 138° while for the treated samples, it was 0°. The wicking property was determined according to AATCC TM 197 (1997), where a strip of the sample (25 x 200 mm^2) is suspended vertically from one end and dipped in a water solution of 2 g/l reactive dye, submerging about 2 cm of the strip from the other end and recording the wicking height as a function of time within 15 min. Five readings were taken for each sample, and the average value was calculated. The results are shown in Fig. 6.

After plasma treatment, the samples show a noticeable improvement in wettability. Interestingly, there is only a minor difference in the results between samples that were treated for 5, 10, and 15 minutes. Therefore, it can be concluded that even a brief treatment time of 5 minutes is sufficient to transform the fabric's surface from hydrophobic to hydrophilic.

The improvement in wettability can be attributed to surface oxidation and ion bombardment, which lead to the formation of free radicals and increase the amount of active species and polar groups on the surface (Kim et al., 2021). Active oxygen rapidly interacts with free radicals formed on the fiber surface, leading to the formation of oxygen-containing polar functional groups (Mihailovic et al., 2010). These groups enhance the fibers' wettability, while the ester oxygen moves closer to the surface (Senthilkumar & Karthik, 2016).

This is similar to what the result obtained by Yilma in his study (Yilma et al., 2021), where he stated that the hydrophilicity increases due to the increase in polar groups and the appearance of grooves or nano-spots on the surface of the polyester fiber after treatment with oxygen plasma, as these formed structures increase the capillary pressure of water in the fibers, and thus treatment with oxygen plasma improves the wettability.

3.4 Tearing strength

This test was conducted following ASTM D1424-21 standard, using Elmatear device, which has a fixed jaw and a moving jaw. To perform the test, the sample with dimensions of 10 x 7.5 cm^2 was placed in the warp direction between the jaws of the device, and an initial incision of 2 cm was made towards the warp, and the tear strength for each sample was measured. Three readings were taken, and the average value was calculated. The results are shown in Fig. 7.

There is a slight decrease in the tear strength of fabric samples as the treatment time increases. This could be due to the softness and structure of the fabric, as well as the length and density of the yarns used. Additionally, the activation of the fabric surface using plasma can slightly affect the fabric structure, which may cause a decrease in the tear strength (Rosace et al., 2010).

3.5 Weight loss

Table 3 shows the weight loss of the treated samples, which were tested according to Eq. (1) described in the experimental procedure section. Weight loss increases with treatment time, ranging from 0.45% to 1.2%. The high kinetic energy of the active species in plasma media

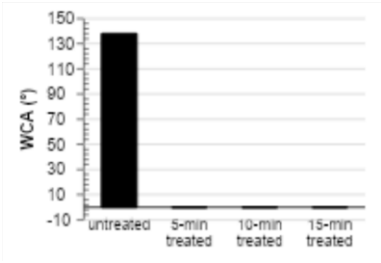


Figure 5. WCA of samples.

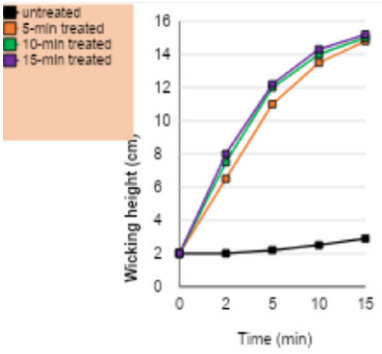


Figure 6. Wicking height of the samples.

explains this phenomenon. When these active species react with the sample surface, they lose energy to strike it, and, as a result of ionic bombardment, atoms with lower bonding energy are removed from the surface. This sputtering process reduces the sample's weight. Weight loss begins in the amorphous areas, and as energy increases, the crystalline areas degrade as well. This result is consistent with the findings of other researchers (Aboltakhty et al., 2018; Öteyaka et al., 2012).

Table 3. The weight loss of the samples.

The sample	Weight loss (%)
5-min treated	0.45
10-min treated	0.8
15-min treated	1.2

3.6 Color strength

To evaluate the effect of plasma treatment on the fabric's dye absorption, the color intensity of the dyed fabric was measured by analyzing *K/S* values of the samples. This analysis was conducted within a 400-700 nm spectrum range, which is the visible spectrum field, using a custom-made device that includes a spectrophotometer (Triax-550-Jobin Yvon), a halogen-tungsten lamp as a light source, a silicon detector, lenses, and mirrors. The device was calibrated using a silver mirror.

The maximum wavelength (λ_{ma}) of the color of dyed samples was determined using this device; it was 600 nm, and *K/S* values were calculated at the maximum wavelength according to Kubelka-Munk Eq. (8):

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \quad (8)$$

Where *K* is the absorption coefficient, *S* is the scattering coefficient, and *R* is the reflection factor of the sample. *K/S* value depends on the dye concentration on the fabric (Kerkeni et al., 2012; Zhang et al., 2017). The K/S values are presented in Fig. 8; the dyed samples are shown in Fig. 9; and Fig. 10 shows the chemical formula of the used dye.

It can be seen in Fig. 8 that the treated samples have higher *K/S* values than the conventional dyed sample, and among the treated samples, the 15-minute treated sample has the highest value, indicating that this sample has the most color strength. The increase in K/S values can be explained by the decrease in the reflectance of the O_2 plasma-treated samples' surface due to the modification of the refractive index on the sample surface (Saloum et al., 2019c).

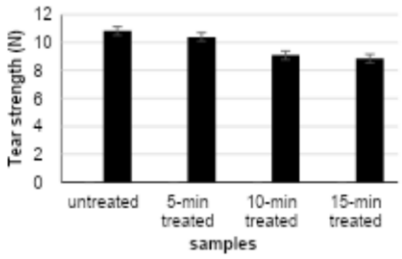


Figure 7. Tear strenght of the samples.

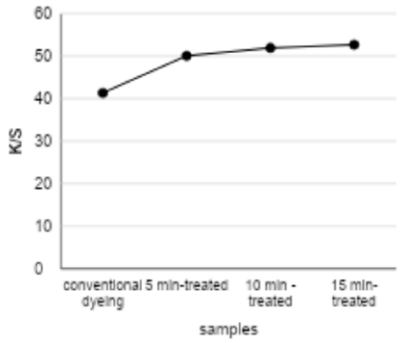


Figure 8. K/S values of the samples at λ_{max} = 600 nm.

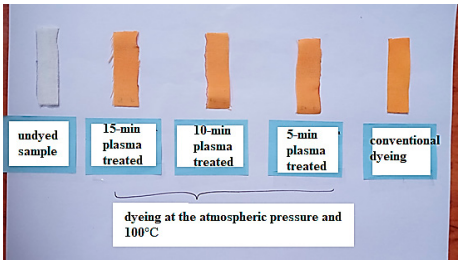


Figure 9. The undyed and dyed samples.

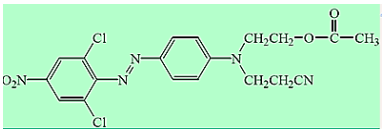


Figure 10. C.I. Disperse orange 30.

K/S values of the plasma-treated samples increased over the conventional dyed sample by approximately 21.08%, 25.57%, and 27.39% after 5, 10, and 15 minutes of treatment, respectively. There seems to be an equation that correlates *K/S* values with the treatment time. Equation (9) displays this correlation, where *'y* represents the *K/S* value and *'x'* represents the treatment time in minutes, with an *R-squared* value of 0.9439.

$$y = 1.3046x + 48.948 \quad (9)$$

For a more in-depth study, the color depth of the dyed samples and the white undyed sample was determined using the ImageJ program. First, the samples were scanned. Then, the color constants of red, green, and blue were calculated from RGB Measure.

To make a comparison, the three colors were transformed into a gray scale using Eq. (10) that takes into account the varying sensitivity of the human eye to each of the colors. The resulting values fall within the range of 0 to 255, where 0 represents black and 255 represents white. This produces a range of shades that can be used for comparison purposes (Karma, 2020; Safarik et al., 2019).

$$\text{Gray scale} = 0.299R + 0.587G + 0.114B \quad (10)$$

Where *R*, *G* and *B* represent red, green, and blue, respectively. The results are shown in Table 4, where it can be seen that the treated dyed samples are darker in color than the untreated, conventional dyed sample, as indicated by the gray scale values.

Table 4. Color constants and grayscale values for samples.

Sample	Red	Green	Blue	Grayscale
White undyed	253.742	253.256	253.471	253.399
Conventional-dyed	239.288	133.427	54.135	156.038
5-min treated	243.98	130.23	53.56	153.21
10-min treated	243.688	125.798	52.97	155.743
15-min treated	242.62	131.15	50.437	152.275

After analyzing the results, it was found that treating the fiber surface with O_2 plasma yields a more intense color at the maximum wavelength. This is because the covalent bonds and surface functional groups increase the dye's absorption. Moreover, it was found that the treatment duration does not significantly affect color intensity. Even a short treatment time of 5 minutes is enough to enhance the color depth. This finding is consistent with the wettability test results. The dyeing enhancement of the treated sample is likely due to the linkage of functional groups on the sample surface, such as -OH and C-O, to the dye via proposed bonds, including -C=N-, -C=O-, or CH_2-.

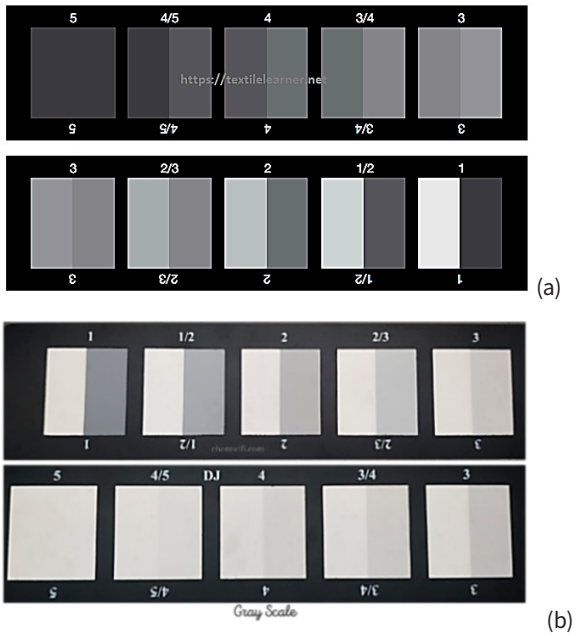
3.7 Dye fastness to washing

To evaluate the color change and color staining of the dyed samples, washing fastness tests were conducted. The washing process was carried out at 40 ± 2 °C for 30 min, according to ISO 105 C01. The color change was evaluated by comparing the washed dyed sample with the unwashed one according to ISO 105-A02: 1993 standard (Fig. 11-a) using grayscale 1, where grade (1) indicates a significant change between the washed and unwashed dyed samples, and the color difference gradually decreases till grade (5) that indicates no change in color between the samples.

The staining level of the dyed fabric is evaluated by placing the dyed sample between two pieces of white fabric of the same type. These three pieces are then sewn together, and the staining degree over the undyed white sample is evaluated according to ISO 105-A03: 1993 standard using grayscale 2 (Fig. 11-b), where grade (1) indicates that the white sample is darkly stained, and the staining decreases gradually until grade (5), where no spot occurs on the white sample. The results are in Table 5, where it can be seen that the washing fastness of the dyed samples is good as that of the conventional dyed sample.

Table 5. The dye fastness to washing.

The sample	color change	Staining degree
Conventional-dyed	5	5
5-min treated	5	5
10-min treated	5	5
15-min treated	5	5



(a)

(b)

Figure 11: (a) Grayscale (1); (b) Grayscale (2).

Conclusion

The surface of the polyester fabric was activated by utilizing oxygen plasma generated from a hollow cathode discharge. Subsequently, the samples underwent a dyeing process using a dispersed dye at atmospheric pressure and boiling point. Moreover, a comparison was made with an untreated sample dyed using the conventional polyester dyeing method in an autoclave at high temperature and pressure. The treatment successfully enhanced the fabric's wettability, with the water contact angle (WCA) decreasing from 138° in the untreated sample to 0° after treatment. Also, the color strength indicated that the K/S values of the treated samples at the maximum wavelength were higher than those of the untreated samples. It was found that treatment time does not affect wettability and color strength much, and a short time is sufficient to enhance them.

FTIR analysis showed an increase in C-O, -C=O, and –OH groups, thus increasing the surface hydrophilicity of the fabric. SEM analysis showed no significant difference in the fabric's surface morphology after treatment. The fabric's washing fastness was good, similar to that of the conventional dyed sample. However, the tear strength decreased slightly after the treatment, and the weight loss increased slightly after it.

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Conflict of interest

The authors did not receive any type of conflict of interest to declare.

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The authors contributed to the design and implementation of the research, the analysis of the results, and the writing of the manuscript.

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