

# Improvement of the Dyeing Properties of 100% Polyester Fabrics Via Chitosan Application

Burcu Sancar Beşen  0000-0001-5120-268X

Pınar Parlakyiğit<sup>2</sup>  0000-0001-9235-6239

Adıyaman University, Faculty of Engineering, Department of Textile Engineering, Adıyaman, Türkiye

**Corresponding Author:** Burcu Sancar Beşen, bbesen@adiyaman.edu.tr

## ABSTRACT

In this experimental study, it was aimed to research the possibilities of improving the dyeing properties of 100% polyester fabrics. For this purpose, the polyester fabrics were pretreated with sodium hydroxide and chitosan. The hydrophilicity tests, SEM-EDX, and FTIR-ATR analyses of the pretreated fabrics were done, and they were then dyed with acid dye at 90 °C. After dyeing processes, the CIEL\*a\*b\* color values, color strength, fastness properties (to washing, water, perspiration, and rubbing) of dyed samples were investigated, and the bursting strength of the undyed and dyed samples was also tested. The results showed that it is possible to improve the dyeing properties of 100% polyester fabrics via chitosan application and that the polyester fabrics can be dyed with acid dyes at low temperatures after being cationized with chitosan following the sodium hydroxide application.

## 1. INTRODUCTION

Polyester fibres have been widely used synthetic fibre in textile industry due to their low production costs, high tensile strength, anti-wrinkle properties, resistance to many chemicals, dimensional stability, and high thermal stability [1-3]. Despite their many advantages, their rigid structure, high crystallinity, and lack of chemically active groups give them hydrophobic characters, resulting in difficult dyeing properties [3-6]. Polyester materials are typically dyed with disperse dyes through the exhaustion method, either at boiling temperatures with carriers or at high temperatures around 130 °C [2-4,7,8]. Dyeing under HT conditions had the disadvantages of high energy consumption and oligomer accumulation on the fibre surfaces [9]. Despite the fact that dyeing polyester materials with carriers require less energy due to atmospheric conditions [2], many carriers have significant problems such as toxicity, unpleasant odor, sensitization of human skin and environmental contamination [3,10].

Several approaches have been used to avoid the drawbacks of dyeing at high temperatures or with carriers [11,12]. These are surface modification of polyester with chitosan and polyelectrolytes, etching of polyester

surfaces via plasma, laser [13], and alkaline treatments [14], dyeing at supercritical CO<sub>2</sub> conditions [15], and using new auxiliaries such as Coumarin and O-vanillin [2]. In order to achieve to dye polyester fabrics with acidic dyes at the low temperature, both the alkaline and chitosan treatments were applied in the present study.

Alkaline treatment of polyester fabrics with sodium hydroxide is a well-known process in the textile industry [14,16]. Sodium hydroxide hydrolyzes the ester groups in the polyester chains; thus, the materials gain a silky handle, luster, and anti-static properties, while their stiffness, tenacity and elongation are decreased [14].

Chitosan is an abundant biopolymer with a linear chemical structure consisting of poly-(1-4)- 2-amido-2-deoxy-D-glucose, which is obtained after alkaline deacetylation of the chitin [10,17- 21]. The chitin is found in the exoskeletons of crustaceans (e.g., crab and shrimp shells) [17]. The chitosan is a long, unbranched polysaccharide similar to cellulose, with an aminoacetyl group instead of a hydroxyl group in the C-2 carbon position [10,21]. Due to being nontoxic, biodegradable, biocompatible, and microbe resistant, chitosan has been used in several application fields, such as pharmaceutical, biomedical applications,

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textile dyeing and finishing, fibre formation, waste water treatment, paper production, and cosmetics [22]. There are many studies in the literature about improving the dyeing properties of polyester materials with chitosan applications. Some of the studies are summarized below.

Walawska et al. (2003) dyed 100% polyester and polyester/cotton fabrics with direct dye at 95°C after alkaline and chitosan treatments. They concluded that it was possible to color polyester and polyester/cotton fabrics with direct dyestuffs after chitosan treatment with medium wet fastness properties, and the alkaline pre-treatment ensured the greater adhesion of chitosan to the surface of polyester fibres, which is manifested by the greater color strength [10]. Ristic et al. (2012) dyed 100% polyester, cotton, and polyester/cotton blended fabrics with reactive dye after application of alkaline, chitosan, and their hybrid. At the end of the study, they concluded that the hybrid application was the most effective for improving dye uptake [5]. Najafzadeh et al. (2018) applied alkaline and nano-chitosan treatments to the 100% polyester fabrics, and then they dyed the fabrics with reactive dye. The results showed that alkaline and nano-chitosan treatments caused improvements in wettability and coloring polyester with reactive dyes [3]. Ibrahim et al. (2019) dyed polyester and polyester/cotton blended fabrics with disperse and acid dyes after carboxymethyl chitosan applications. They found that using carboxymethyl chitosan improved the dyeability and antibacterial activity of the fabrics [18]. Hilal et al. (2020) treated 100% polyester, cotton and polyester/cotton blended fabrics with sodium hydroxide, chitosan, and their hybrids and dyed the fabrics with reactive dye. They concluded that hybrid treatments of textile materials by combining alkaline and chitosan are the most effective for improving color strength and dye fixation percentage [14]. When the studies are focused, it can be seen that most of the studies deal with coloring polyester/cotton blended fabrics with reactive dyes after treatments with chitosan and chitosan derivatives. However, because of the necessity of using salt and soda in the reactive dyeing process, it was thought that a different dye usage would be more ecological for dyeing 100% polyester materials. For that reason, the polyester fabrics were colored with acid dye in the present study.

In the paper, it was aimed to improve the dyeing properties of 100% polyester fabrics by way of applying alkaline and chitosan treatments. For this purpose, the polyester fabrics were pretreated with sodium hydroxide, and then cationized with chitosan. The pretreated samples were researched via hydrophilicity tests, SEM, EDX, and FTIR analyses. Then the fabrics were dyed with acid dye at 90 °C. Following the dyeing process, the dyed fabrics' CIEL\*a\*b\* color values, color strength (K/S), bursting strength, and color fastness to washing, water, perspiration (acidic-basic), and rubbing (dry-wet) were investigated.

## 2. MATERIAL AND METHOD

### 2.1. Material

Sodium hydroxide (NaOH, liquid form), acetic acid (Sigma-Aldrich), chitosan (medium molecular weight, Sigma-Aldrich), Rucon LFF (low formaldehyde content cross-linker, Rudolf Duraner, combination of modified dihydroxyethylene urea and catalyst), and Bemacid N-TF blue (acid dye-CHT, Turkey) were used in the experiments. The fabric used in the study was knitted (single jersey, 20E) with 100% polyester yarn (30/1 Ne). A laboratory-type fulard (Termal), IR dyeing machine (Termal), and steamer (Ataç) were also used. All of the experiments were carried out with distilled water.

### 2.2. Method

The fabric samples were pretreated with sodium hydroxide and chitosan according to the experimental plan given in Table 1.

**Table 1.** The experimental plan for the pretreatment of the fabric

| Sample Number | Concentration of the NaOH (g/L) | Concentration of the chitosan (g/L) |
|---------------|---------------------------------|-------------------------------------|
| 1             | 0                               | 0                                   |
| 2             | 10                              | 0                                   |
| 3             | 20                              | 0                                   |
| 4             | 0                               | 15                                  |
| 5             | 10                              | 15                                  |
| 6             | 20                              | 15                                  |

Firstly, the alkaline treatments were applied to polyester fabric samples with two concentrations of sodium hydroxide (10 g/L and 20 g/L) at 98 °C for 30 minutes through the IR dyeing machine. The processes were carried out with a liquor ratio of 1:15. After the processes, the fabric samples were washed with boiling water for 10 minutes, subsequently neutralized with 1g/L acidic acid for 5 minutes, and then rinsed with cold water. The fabrics were then dried at room temperature. Some samples were reserved without applying the alkaline treatments.

After the alkaline processes, the fabric samples were cationized with chitosan at a constant concentration of 15 g/L. The processes were carried out on the samples via the pad-dry-cure method. In order to prepare the application solutions, the chitosan was dissolved in %1 (w/v) acetic acid solution, and 50 g/L cross linker agent was added to the solutions. The fabric samples were squeezed up to 60% pick up at the fulard, then dried at 100 °C and cured at 110 °C for 2 minutes at the steamer. In order to investigate the effect of chitosan process on the results, some of the fabric samples were saved without chitosan applications.

The pretreated fabric samples were dyed at a constant concentration of %1,5. The dyeing processes were carried out with a liquor ratio of 1:15 in the IR dyeing machine according to the conditions given in Figure 1 as a graph. Following the dyeing processes, the fabric samples were

washed three times in boiling water, then rinsed with cold water. The fabric samples were then dried at 100 °C.

### 2.3. The research methods

**Hydrophilicity test:** The hydrophilicity of the pretreated fabrics was investigated via dropping, and dipping tests. Three measurements were carried out for each sample and the average of results was given. In the dropping test, distilled water is dropped to the fabric and the absorption time of the water is measured [23]. In the dipping test, the fabric sample (5 cm × 5 cm) is left into a water filled beaker from above 1 cm of the water surface. The time when the fabric completely left from the water surface is measured [24]. Since the polyester fabrics have a hydrophobic structure, the dipping time could not be measured, so the test results were given by way of observations.

**SEM and EDX analyses:** The surface morphology of the pretreated fabrics was analyzed through Quanta 400F Field Emission scanning electron microscope. The fabrics were sputtered with gold under vacuum prior to observation. The SEM images were taken at an accelerating voltage of 10.00 kV, and with a magnification of 5000X. In addition, the elemental compositions of the pretreated fabrics were investigated through energy dispersive X-ray spectrometer.

**FTIR-ATR:** Fourier transform infrared-attenuated total reflectance analyses of the pretreated fabrics were carried out by the transmission method using a Perkin-Elmer spectrophotometer (Spectrum 400) between 400-4000 cm<sup>-1</sup>. Resolution for the infrared spectra was 4 cm<sup>-1</sup>, and there were four scans for each spectrum.

**Bursting strength:** The test was applied to dyed and undyed samples according to ISO 13938- 2:2019 standard [25] as three replications and the average of results was calculated.

**CIEL\*a\*b\* color value and color strength (K/S) value:** After the dyeing process, the CIEL\*a\*b\* values and the reflectance (R) values at wavelengths ranging from 400 to 700 nm of the dyed samples were measured. They were measured under D65 daylight and aspect of 10° with spectrophotometer (Datacolor SF 600 model). The results

were measured as three replications, and the average of results was calculated. The K/S values were calculated using Kubelka-Munk equation by using the R value at the wavelengths (600 nm) at which fabric samples had maximum absorption.

**Color fastness test:** In order to investigate the color fastness results of the dyed samples, fastness tests to washing [26], water [27], acidic and alkaline perspiration [28], wet and dry rubbing [29] were carried out.

### 3. Results and Discussion

The hydrophilicity tests were carried out on the pretreated fabric samples to observe whether the applied pretreatments had effects on them. The results are given in Table 2. According to the hydrophilicity test results, it is possible to say that the application of both sodium hydroxide and chitosan increased the hydrophilicity of the polyester fabrics. The situation is thought to be explained by the -NH<sub>2</sub> functional groups found in chitosan, and the incorporation of new -OH groups at the ends of the depolymerized polyester matrix [14] after alkaline treatment.

In order to investigate the surface morphology and chemical compositions of the pretreated fabrics, SEM and EDX analysis were carried out. The SEM images and EDX results of the fabric samples are given in Figure 2 and Figure 3, respectively. From the SEM images of the untreated fabric (Fig. 2a), it can be seen that the untreated fibre has a smooth surface, with some clear oligomer particles on it. Figure 2b and Figure 2c demonstrate that alkaline treatment created some pores and scars on the surface of polyester. Figure 2d, Figure 2e, and Figure 2f proved that the fibre surfaces were coated with chitosan, and the application of alkaline treatment before chitosan treatment ensured a more homogeneous and smooth coating since the sodium hydroxide opened the structure of PES fibres. The EDX results given in Figure 3 also support the fact that the -O element of the polyester fabrics increased after alkaline treatment (Fig. 3b and Fig. 3c), and the -N element (Fig. 3e and Fig. 3f) proved the chitosan presence on the fabrics after chitosan application.

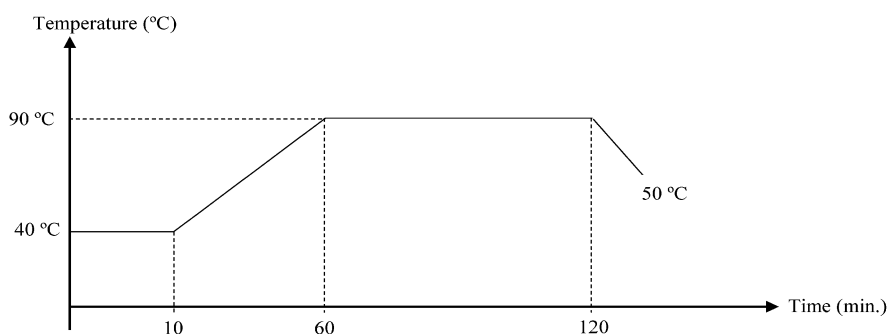
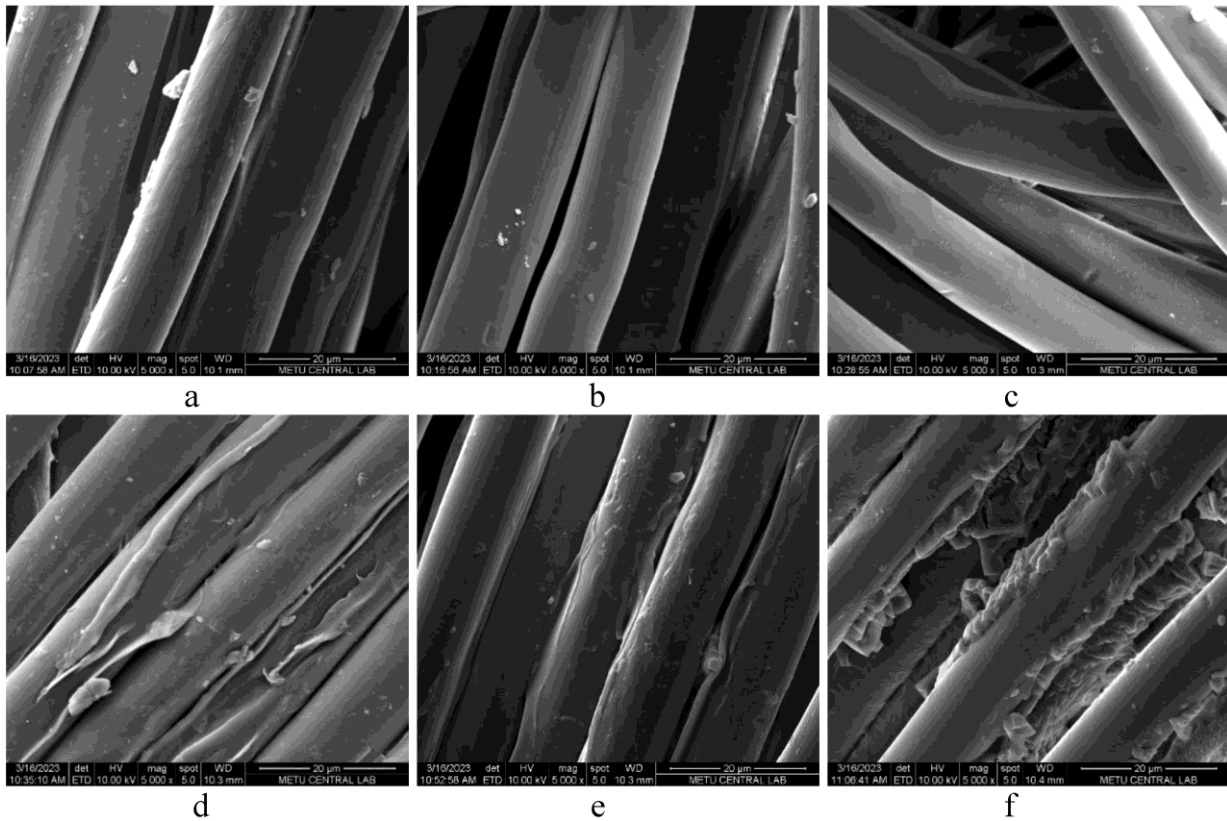


Figure 1. The dyeing conditions

**Table 2.** The hydrophilicity test results

| Sample Number | Dropping test (min.) | Dipping test (min.) | Observation                          |
|---------------|----------------------|---------------------|--------------------------------------|
| 1             | >120                 | >120                | Super hydrophobic                    |
| 2             | >120                 | >120                | Hydrophobic                          |
| 3             | 85                   | >120                | Wetting on the fabric surface        |
| 4             | >120                 | >120                | Slight wetting on the fabric surface |
| 5             | 115                  | >120                | Wetting on the fabric surface        |
| 6             | 110                  | >120                | Wetting on the fabric surface        |

**Figure 2.** SEM images of the pretreated fabrics *a.1, b.2, c.3, d.4, e.5, f.6* (qq. Table1)

The FTIR-ATR analyses were carried out in order to assess structural change in the pretreated fabrics, and the results are given in Figure 4. According to FTIR spectra, it is possible to say that the results are almost identical. A broad peak at  $1712\text{ cm}^{-1}$  is characteristic of C=O stretching of an unsaturated ester, while the peak at  $722\text{ cm}^{-1}$  represents the aromatic  $\text{sp}^2$  C-H bend. The peak at  $1016\text{ cm}^{-1}$  indicates plane vibration of the benzene ring, the peaks at  $1093\text{ cm}^{-1}$  and  $1240\text{ cm}^{-1}$  represent C-O stretch (ester), and absorption at  $2917\text{ cm}^{-1}$  is due to the asymmetric C-H stretching [14,30]. A slight shift in the position of the peak to a higher wave number (from  $2964$  to  $2966\text{ cm}^{-1}$ ) is observed after the alkaline treatments compared to untreated fabric. In addition, on comparing the

fabrics treated with chitosan, a very low, intense peak around  $3400\text{ cm}^{-1}$  is detected, representing N-H stretching [30].

The bursting strength results were tested in order to observe whether the applied pretreatments had any negative effects on the strength properties of the fabric samples. The results of the fabric samples before and after dyeing can be seen in Figure 5. When the bursting strength results are focused, it is seen that all of the values are close to each other. Thus, it can be said that neither the alkaline nor the chitosan treatments have negative effects on the fabrics' strength. In addition, it is possible to say that the chitosan applications cause a slight increase on the fabrics' strength, which is thought to be due to the usage of cross-linker in the chitosan applications.



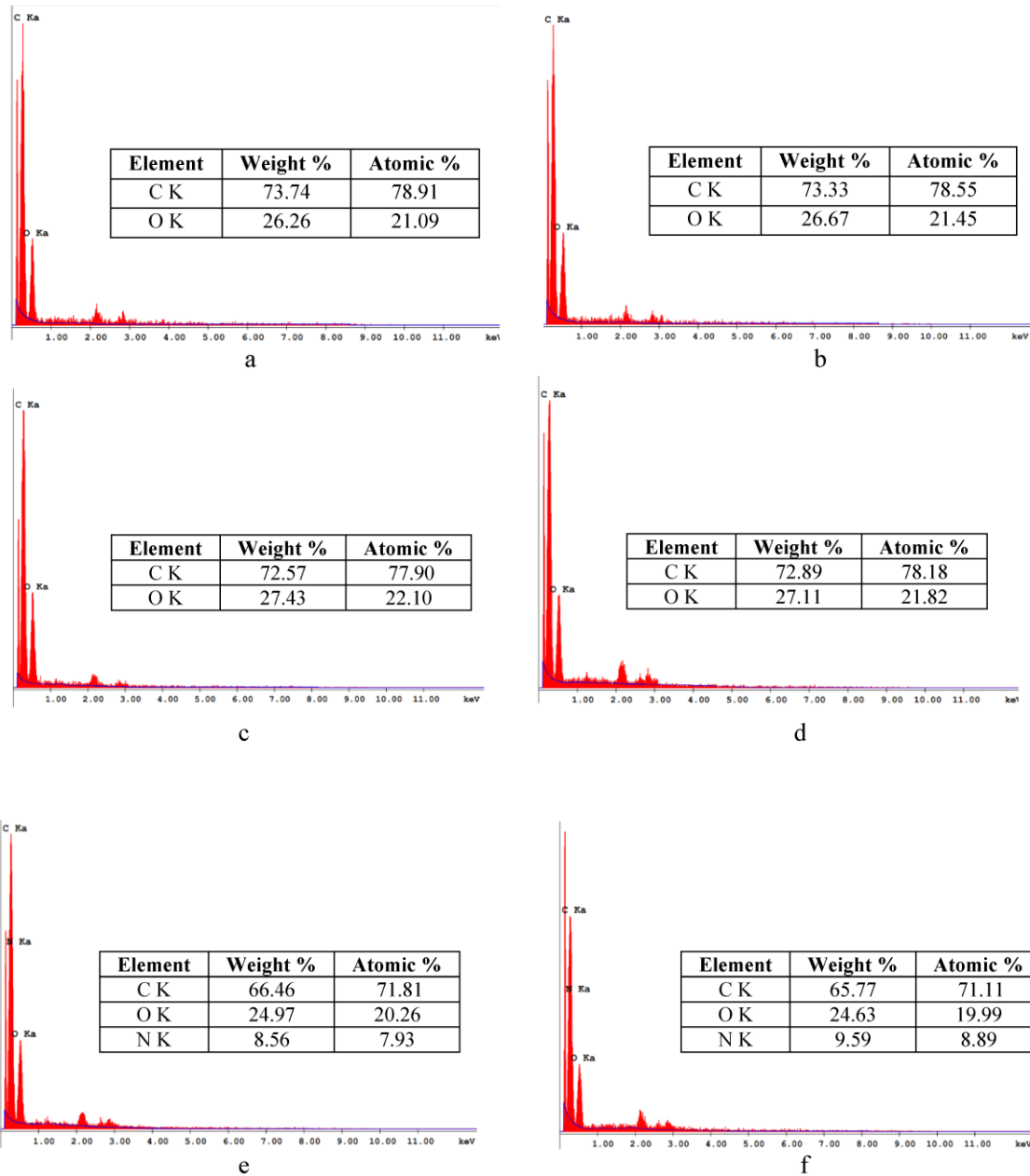


Figure 3. EDX results of the pretreated fabrics a.1, b.2, c.3, d.4, e.5, f.6 (qq. Table1)

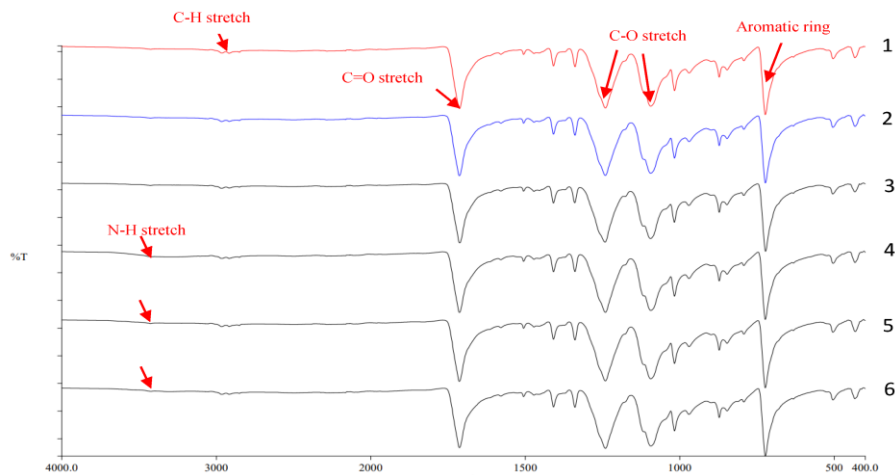


Figure 4. FTIR-ATR spectra of the pretreated fabrics



Table 3 shows CIEL\*a\*b\* color values, as well as the color strength (K/S) values of the dyed samples. It is commonly known that while the L\* value indicates the lightness and darkness, the b\* value gives information about the yellowness and blueness, and the a\* value is about the reddish and greenish of the fabric samples in the CIEL\*a\*b\* color system. The L\* value changes between 0 and 100, and the color becomes lighter as the L\* value increases. The increase in the a\* and b\* values mean that the color goes to the reddish and yellowness, respectively. In addition, the increase in the K/S value means that the fabric is darker. When the results in Table 3 are examined, it is possible to conclude that the dye uptake did not increase when only the alkaline treatments were applied, but increased when the chitosan treatments were only applied. The situation is thought to be derived from the groups formed on the polyester after the

treatments. While the acidic dyes have no affinity for -OH groups formed after the alkaline treatments, they have affinity for -NH<sub>2</sub> groups. Besides, chitosan applications following alkaline treatments have positive effects on the dye uptake, and the fabric sample (coded as 5) is darker than other samples. The results are not amazing since the chitosan can bond to polyester through -OH groups, and the groups on the polyester fibres increases with alkaline processes. In addition, when the L\*, a\*, b\*, and K/S values of the samples coded as 5 and 6 are focused, the importance of the sodium hydroxide concentration applied is realized. This situation demonstrates that the alkaline treatment conditions applied before chitosan treatment are crucial to the dyeing properties of 100% polyester fabrics, and it is not necessary to use more sodium hydroxide than 10 g/L.

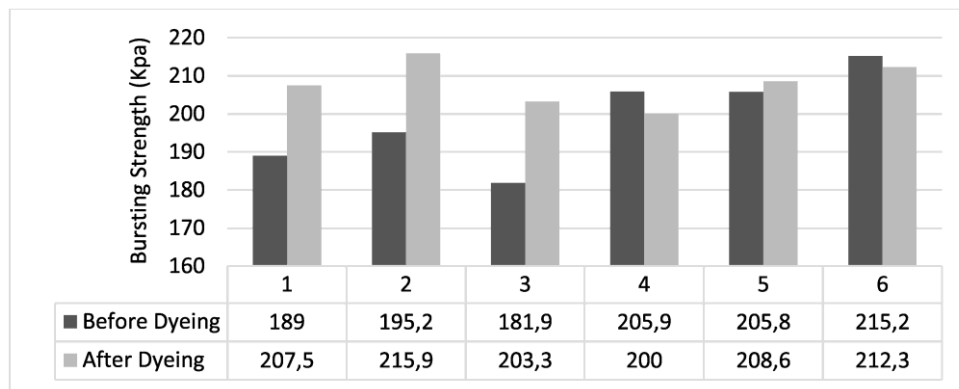








Figure 5. The bursting strength results

Table 3. The results of CIEL\*a\*b\* color values and color strength (K/S) values

| Sample number | Photos  | L*    | a*    | b*     | R    | K/S  |
|---------------|---|-------|-------|--------|------|------|
| 1             |  | 82,16 | -4,70 | -9,88  | 0,52 | 0,22 |
| 2             |  | 84,04 | -4,05 | -7,56  | 0,57 | 0,16 |
| 3             |  | 84,35 | -3,81 | -7,17  | 0,58 | 0,15 |
| 4             |  | 64,42 | -3,80 | -27,95 | 0,23 | 1,25 |
| 5             |  | 55,37 | -1,00 | -31,49 | 0,16 | 2,30 |
| 6             |  | 66,15 | -4,24 | -24,23 | 0,26 | 1,06 |

The color fastness to washing and water, as well as to perspiration and rubbing of the dyed samples are given in Table 4 and Table 5, respectively. According to the fastness results, it is possible to say that the all of the investigated fastnesses are lower for darker samples, as expected. This situation is clearer for the wet fastness results, and it has been thought to be concluded that the cross-linker used in chitosan applications. The chitosan, accordingly dyes cannot hold on to the fibres with strong bonds, thus the dyes removed from fabrics by wet treatments. Therefore, it is possible to say that the cross-linker used in the chitosan applications is so crucial for obtaining high wet fastness. In order to improve the fastness properties of the dyed samples, either the cross-linker's concentration may be increased or a stronger cross-linker should be used.

#### 4. CONCLUSION

Although polyester fibres have wide usage in the textile industry due to their many advantages, they are problematic in dyeing treatments because of their rigid structure, high crystallinity, and lack of chemically active groups. In order to solve that problem, several approaches have been studied. The current study also attempted to improve the dyeing properties of polyester fabrics by using chitosan. For this purpose, both the alkaline and chitosan applications were carried out on the 100% polyester fabrics. Following those treatments, the fabric samples were dyed with acid dyes at 90 °C. The color values and fastness properties of the dyed samples, as well as the bursting strength and hydrophilicity of the fabric samples, were investigated. The results can be concluded as given below.

- It is possible to improve the dyeing properties of polyester fabrics by way of chitosan applications. It is recommended to apply an alkaline treatment at convenient conditions before chitosan in order to increase dye uptake.
- Because the polar groups on the polyester formed after the treatments, both alkaline and chitosan applications have positive effects on the hydrophilicity of the fabric samples.
- The strength properties of the fabric samples are not negatively affected by the sodium hydroxide and chitosan treatments. Moreover, their strength improved due to the cross-linker agent used in the chitosan treatments.
- The wet fastness results obtained in the present study are insufficient, however they can be improved by the way of stronger bonding of chitosan to the fibres.

As a conclusion, the experimental study shows that it is possible to improve the dyeability properties of 100% polyester fabrics with acid dyes via the combined application of alkaline and chitosan without having any negative effects on the fabrics' strength. In order to improve the wet fastness results the chitosan must be better bonded stronger to the fibres. For that purpose, it is recommended to use a stronger cross-linker, higher concentration levels of cross-linker, or apply the curing treatment at a higher curing temperature or time.

#### Conflicts of Interest

The authors declare that they have no conflict of interest.

**Table 4.** The color fastness to washing and water of the dyed samples

| Sample number | Washing |     |     |     |     |     |     | Water  |     |     |     |     |     |     |
|---------------|---------|-----|-----|-----|-----|-----|-----|--------|-----|-----|-----|-----|-----|-----|
|               | Fading  | As  | Co  | PA  | PES | PAC | WO  | Fading | As  | Co  | PA  | PES | PAC | WO  |
| 1             | 4       | 4/5 | 4/5 | 4   | 4/5 | 4/5 | 4/5 | 3/4    | 4/5 | 4   | 4   | 4   | 4/5 | 4/5 |
| 2             | 4       | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 3/4    | 4/5 | 4/5 | 4   | 4/5 | 4/5 | 4/5 |
| 3             | 4       | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4      | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 | 4/5 |
| 4             | 3/4     | 4/5 | 4/5 | 3   | 4/5 | 4/5 | 4/5 | 3      | 3/4 | 2   | 2   | 3/4 | 3/4 | 3   |
| 5             | 3       | 4/5 | 4   | 3   | 4   | 4/5 | 4/5 | 2/3    | 3   | 2   | 2   | 3/4 | 3/4 | 3   |
| 6             | 3       | 4/5 | 4/5 | 2/3 | 4/5 | 4/5 | 4/5 | 2/3    | 3   | 2   | 2   | 3/4 | 3/4 | 3   |

**Table 5.** The color fastness to perspiration and rubbing of dyed samples

| Sample number | Perspiration |     |     |    |     |     |     |          |     |     |    |     |     |     | Rubbing |     |
|---------------|--------------|-----|-----|----|-----|-----|-----|----------|-----|-----|----|-----|-----|-----|---------|-----|
|               | Acidic       |     |     |    |     |     |     | Alkaline |     |     |    |     |     |     | Wet     | Dry |
|               | Fading       | As  | Co  | PA | PES | PAC | WO  | Fading   | As  | Co  | PA | PES | PAC | WO  |         |     |
| 1             | 3/4          | 4/5 | 4/5 | 4  | 4/5 | 4/5 | 4/5 | 3/4      | 4/5 | 4/5 | 4  | 4/5 | 4/5 | 4/5 | 4       | 4/5 |
| 2             | 3/4          | 4/5 | 4/5 | 4  | 4/5 | 4/5 | 4/5 | 3/4      | 4/5 | 4/5 | 4  | 4/5 | 4/5 | 4/5 | 4       | 4/5 |
| 3             | 3/4          | 4/5 | 4/5 | 4  | 4   | 4/5 | 4/5 | 3/4      | 4/5 | 4/5 | 4  | 4/5 | 4/5 | 4/5 | 4       | 4/5 |
| 4             | 3            | 3/4 | 3   | 2  | 3/4 | 4   | 2/3 | 2/3      | 3/4 | 2   | 2  | 3   | 3/4 | 2/3 | 2       | 4   |
| 5             | 2/3          | 3/4 | 3   | 2  | 3/4 | 4   | 2/3 | 2        | 2   | 1/2 | 2  | 2/3 | 3/4 | 3   | 2       | 4   |
| 6             | 2/3          | 3/4 | 2   | 2  | 3   | 3/4 | 2/3 | 2/3      | 3/4 | 3   | 2  | 3/4 | 4   | 2/3 | 2       | 4   |



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