



Improving Dyeing Parameters of Polyester/Cotton Blended Fabrics by Caustic Soda, Chitosan, and Their Hybrid



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THE production of polyester fibers has been dynamically increased, accompanied by the common use of polyester-cotton blends. In this research work, polyester (100%), cotton (100%), and polyester-cotton blend fabrics (50/50) have been subjected to treat with caustic soda, chitosan and their hybrid, followed by one-bath two-stage dyeing with reactive dye (Reactive Red 198 RR198), to study their dyeing performance and morphology. SEM images of polyester treated and untreated with caustic soda, it describes the ease of adsorption on the alkaline modified fabric surface. The one bath two step method was used to shorten the dyeing process, increase yield and reduce the utility and chemical cost. In the final stage, the dyed fabrics were tested for color strength (K/S) value, dye intensity, fixation% in addition to, fastness properties evaluated by testing the light, washing and perspiration fastness properties of the dye. The results clearly show that for all pre-treated fabrics using caustic soda (90 g/dm³ of caustic soda, 100 min ; 80°C), favorable absorption of reactive dye RR198, resulting a high color strength and fixation % due to surface modification of polyester and mercerization of cotton fabrics. The effect of chitosan pretreatment on dyeability, fastness, and some physicochemical properties has been investigated. Hybrid treatments of textile materials by combining alkali and chitosan are potentially the most effective for improved dye color strength and fixation % and it could be applied as a novel approach to textile dyeing and finishing.

Keywords: Dyeing, Reactive dye, Polyester/ cotton blend, Mass loss, Chitosan caustic soda and fastness.

Introduction

The development of polyester fibers has rapidly increased in recent years, followed by the widespread use of polyester-cotton blends. Textiles made from blends of polyester and cotton fibers constitute about 10% of the total consumption of textile materials. Polyester and cotton fiber blends show the advantages of both components of the textile fabric. Due to significant differences in the chemical structure of both types of fibers, the problems of effectively dyeing such blends have been extensively studied. Polyester fibers show a definitive hydrophobic character and a high degree of crystallinity, being difficult to penetrate

with dyes, however it has wide use in production of garments because of their low production cost and good fiber properties [1]. Polyester has a poor tolerance to hot alkali allowing the polymer to be completely depolymerized by alkaline hydrolysis. The attacking hydroxide ion is not believed to diffuse into the polymer but rather act on the surface, splitting the ester bonds yielding an alcohol and a carboxylic acid [2]. Alkaline finishing of polyester fabric with caustic soda changes fabric weight, strength, wettability and aesthetics [3]. Cotton cellulose is readily depolymerized in acidic environments; however, its resistance to alkaline environments is higher

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[4]. Despite their ease of use, acceptance of polyester / cotton blended fabrics is growing day after day. Polyester has performance qualities such as breathable properties and quick-dry properties, and it can be used to enhance the absorption of cotton sweat. In the other side, polyester is not breathable and cotton is, but you can obtain a fabric that is both breathable and sweat-resistant by combining the two materials. Therefore, the choice of these fibers provides the necessary level of comfort and better resistance because of the different material properties. The presence of both polyester / cotton substrates in textiles does however cause difficulties in the dyeing process [5]. By treating polyester/cotton fabrics in alkaline conditions (using sodiumhydroxide) further improvement in the fabric properties can be obtained. Alkali treatment of polyester fabric is a well-known and conventional process in textile industry. Sodium hydroxide can hydrolyze the ester groups in the polyester chains. Several researchers have proposed different mechanisms for alkaline hydrolysis of polyester fiber [6,7]. The weight reduction of polyester ranging between 15 and 30%, gives a silky handle, luster, soil repellency and anti-static properties to the polyester fabric, and its stiffness, tenacity and elongation are decreased [8]. Although alkaline hydrolysis decreases the diameter of the fibers, the density of the fibers is not affected. It has been stated by some researchers that the rate of polyester hydrolysis can be improved by using certain accelerants [9]. Currently recognized dyeing methods for binary textile blends of polyester / natural fiber use two types of dyes to dye components in one or two solutions, creating large volumes of untreated wastewater that disgrace the entire environment. The global initiative for rational utilization of natural resources and increasingly more stringent environmental legislation impose the need for development of new methods more superior compared to traditional dyeing in terms of technological productivity, economy and protection of water courses. Moreover, the new dyeing methods should provide high performances of dyes on textile, and from this point of view the main challenge in dyeing of blends is matching color intensities on blend components. In order to rationalize dyeing of polyester/cotton blends, recently, dyes with new coloristic properties have been developed or pretreatment improving dye affinity for fibers have been applied [10-15]. Chitin and chitosan are highly versatile biomaterials that have gained considerable

interest in a wide range of applications due to their specific characteristics such as nontoxicity, biocompatibility, biodegradability, low teratogenicity effects, biological activity, low cost etc. [16]. Chitosan particles are well implemented in the cotton and PES/CO blend fabrics. Five washing cycles confirmed the presence of chitosan, but in lower quantity. However, achieved antimicrobial activity is persistent. The application of NaOH led to the swelling and mercerization of cotton cellulose, and the hydrolysis of polyester, as well as the neutralization of forthcoming acid bath, which led to better mechanical properties. It opened the structure for better chitosan particles implementation into the cotton fiber and onto the polyester component of Polyester/Cotton blend, which is certainly a major contribution in the field of achieving persistent processing of cellulosic materials [17]. In this work, commercial polyester, cotton and factory blended polyester/cotton fabrics were treated with alkali and chitosan solutions of various concentrations, with the aim to improve dyeing with reactive dye. The effects of alkali on surface structure and chemical composition of polyester have been studied. The improved dyeing of components and factory blended polyester/cotton blends after combined pretreatment was evaluated by color intensity, fixation degree and dye fastness.

Materials and Methods

Materials

Fabric

100% polyester fabric with surface mass of 149,5 g/m², 100% cotton fabric with surface mass of 206, 67 g/m² and, 50/50 polyester/cotton blend with surface mass of 172,53 g/m² were supplied by El-Mahalla El-Kobra Spinning & weaving company. The fabrics were scoured in aqueous solution with a liquor ratio 1: 50 containing 2g/l nonionic detergent solution at 50° C for 30 min to remove waxes and impurities, then rinsed thoroughly in cold water and dried at room temperature.

Dye and Chemicals

Reactive Red 198, RR198 (C.I. 18221) was purchased from CLARIANT (M.wt = 984.22 g), its chemical structure is shown in Scheme 1. Hydrochloric acid, sodium hydroxide, glacial acetic acid, sodium chloride and sodium carbonate mentioned elsewhere in this study were of analytical grade. Chitosan used was obtained from Primex. It has the following characteristics:

degree of deacetylation 96%, viscosity 102 cP, solubility 99.9%, (Molecular weight, C3646-100G).

Methods

Treatment of textile fabrics

Treatment of polyester, cotton, and polyester / cotton blend fabrics with caustic soda

Caustic soda treatment of three textile fabrics types was performed in aqueous NaOH solution at temperatures of 80°C and 100°C, alkali concentrations were 30, 60 and 90 g/dm³ and treatment time 20, 40, 60, 80 and 100 minutes. Sample mass was 5 g and solution volume were 200 cm³. After alkalization, the samples were neutralized with diluted glacial acetic acid followed by distilled water rinsing and drying in air.

Weight loss % of alkali treated polyester fabrics was estimated using the following equation [18]:

$$\text{Weight loss \%} = \frac{w_1 - w_2}{w_1} \cdot 100 \quad \% \quad (1)$$

w_1 is the weight of untreated sample, w_2 is weight of hydrolyzed sample.

Treatment of polyester, cotton and polyester / cotton blend fabrics with chitosan

Chitosan treatment of three textile fabrics types was performed in freshly prepared solutions of chitosan with concentrations of 1, 7 and 14 g/dm³ (chitosan dissolved in 10 % acetic acid at pH 4) at Liquor ratio 1:40, 25°C for 60 min with continuous stirring. The treated samples were squeezed and dried in air at ambient temperature, washed with distilled water followed by diluted glacial acetic acid, then dried again.

Treatment of polyester, cotton, and polyester / cotton blend fabrics with caustic soda – chitosan hybrid

Hybrid treatments of three textile fabrics types was performed by combining 60 g/dm³ of caustic soda and 7 g/dm³ of chitosan at liquor ratio 1:40, 25°C and treatment time 20, 40, 60, 80 and 100 min with continuous stirring. The treated samples were squeezed and dried in air at ambient temperature, washed with distilled water followed by diluted glacial acetic acid, then dried again [19].

Volumetric method for determination of the content of end carboxyl groups (SJK) of treated polyester fabrics.

The content of end carboxyl groups is determined by volumetric method as follows: in an Erlenmeyer flask containing 100 cm³ of 0.01M NaOH solution, 0.1 g of fiber is added. The flask is closed well to prevent reaction between NaOH and CO₂ from air and the sample was subjected to stirring for 1 hour and then 20 cm³ of the solution is taken and titrated with 0.01 M HCl solution with phenolphthalein as indicator. Three measurements are made, and an average of acid volume used is taken. (SJK) is calculated as follows [20]:

$$SJK = 5 \left[\frac{V_{NaOH} \cdot C_{NaOH} - V_{HCl} \cdot C_{HCl}}{m(1 - M)} \right] \left(\frac{mmol}{g} \right) \quad (2)$$

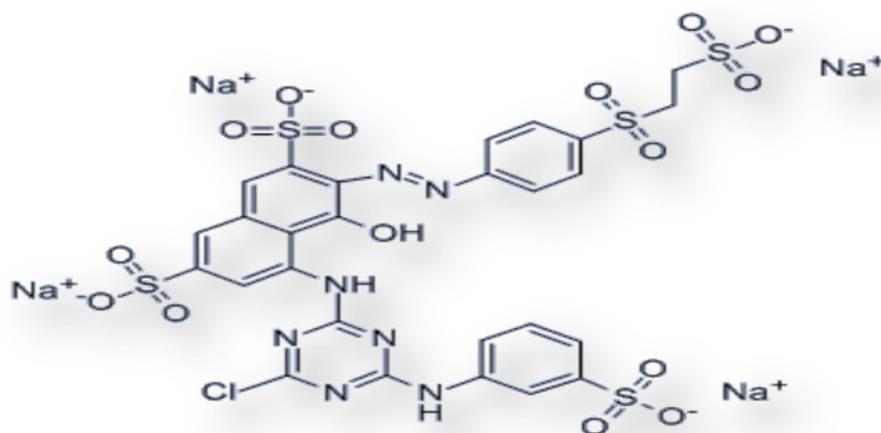
Where, m: fiber mass, g and M: moisture content, %

SEM micrograph and FTIR analysis

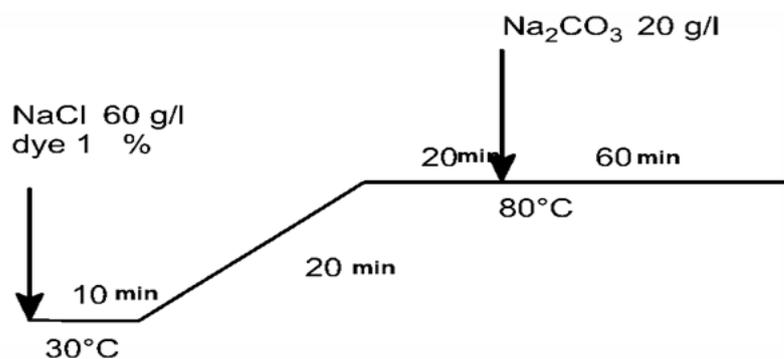
Scanning electron microscopic studies were made on treated and untreated fabric with S-3000H-Hitachi, Japan to study surface modifications if any caused by the caustic soda treatment. The samples were imaged with a magnification of 500X for better understanding of the inner core of the sample. Fourier Transform infrared spectral analysis of the treated and untreated fabrics was recorded in the range of 4000–400 cm⁻¹ using Perkin Elmer spectrometer (spectrum BX, USA) with built-in spectral matching computerized software.

Dyeing of untreated and treated polyester / cotton fabrics with RR198 via one bath two stage dyeing method

Untreated and treated polyester / cotton blend fabrics were dyed in one-bath two stages dyeing using 1% of reactive dye (RR198) with liquor ratio 1:40. The samples were immersed in the dye bath and worked for 10 minutes at 30°C with adding 60 g/l of sodium chloride (exhausting agent). The temperature was gradually raised to 80°C with an interval of 20 min. After 20 minutes, previously dissolved sodium carbonate (fixing agent, 20 g / l) was added in two installations with a 20 min interval and dyeing continued at 80°C for another 60 minutes. The cold running water cleaned the colored samples and then soaped [21]. The dyeing profile of polyester / cotton blend fabrics with RR198 coloring was characterized in Scheme 2.



Scheme 1. Chemical structure of RR 198.



Scheme 2. Dyeing diagram with C.I. Reactive Red 198.

Determination of color strength k/s

The color strength (k/s) was measured using the Win lab software of the Perkin Elmer, Lambda 35 spectrophotometer according to ASTM, D : 2288-93. The measurement was carried out at the respective wavelength of maximum absorption for RR198 ($\lambda = 525 \text{ nm}$)

Determination of percentage of color intensity

Percentage of color intensity increase I of modified samples in relation to original sample was estimated from the following equation (3):

$$I = \frac{\left(\frac{k}{s}\right)_m - \left(\frac{k}{s}\right)_o}{\left(\frac{k}{s}\right)_o} \times 100 \quad [\%] \quad (3)$$

where : subscript m refers to modified fabric samples and subscript o to untreated samples.

Determination of Fixation, $F\%$

Fixation % was calculated according to equation (4):

$$F = \frac{\left(\frac{k}{s}\right)_T}{\left(\frac{k}{s}\right)_o} \times 100 \quad (4)$$

where : subscript T refers to soap treated fabric and subscript o to untreated fabric

Determination of color fastness properties of dyed textile fabrics

The untreated and treated dyed samples were tested according to ISO standard test methods. The color fastness to light was measured according to ASTM, D : 2053-86, while the color fastness to wash and perspiration was determined according to AATCC (1998) 15-1960 and 36-1961. These properties were measured at the National Institute for Standards, Textile Department, Giza, Egypt.

Results and Discussion*Scanning electron microscopy (SEM) analysis*

Scanning electron microscopy (SEM) was used to determine the effects of caustic soda application on the topography of untreated and

treated polyester fabrics (Fig. 1). Fig. 1a indicates that the untreated polyester fabric has a smooth surface with some clear white particles, possibly due to the incomplete removal of the chemicals during the washing process [22]. These particles are oligomers that migrated and crystallized on the surface during stretching and thermal processing. Fig. 1b demonstrates the SEM photographs of the caustic soda adsorption onto the fabric after the treatment. It is clear to see that the reaction of polyester with alkali starts on fabric surface where high negative charge of fabric acts as a barrier hindering penetration of hydroxyl ions to fabric core [23]. Shorter chains, produced as a result of hydrolysis, are removed into solution resulting in mass loss and changes of surface structure. It is, therefore, presumable that polymer degradation occurs on areas of decreased structure order where polymer density and energy of side bonds are lower facilitating access of alkali.

Mapping scan and EDX analysis

EDX and mapping scan analysis was performed using (TEAM- EDX Model). Fig. 2 represent mapping scanning of treated polyester fabric with caustic soda. The figure shows the homogeneous dispersion of carbon, oxygen and sodium through polyester fabric matrix (carbon has red color, oxygen blue color and sodium white color), mapping scanning technique demonstrate the surface modification of the treated polyester which was confirmed by SEM and EDX analysis. EDX analysis was confirm the elements present in the modified polyester fabric. The analysis reveals the presence of C, O and Na elements as shown in Fig. 3 with a percentage corresponding to each. The obtained analysis prove that the treatment of polyester fabric with caustic soda was successful.

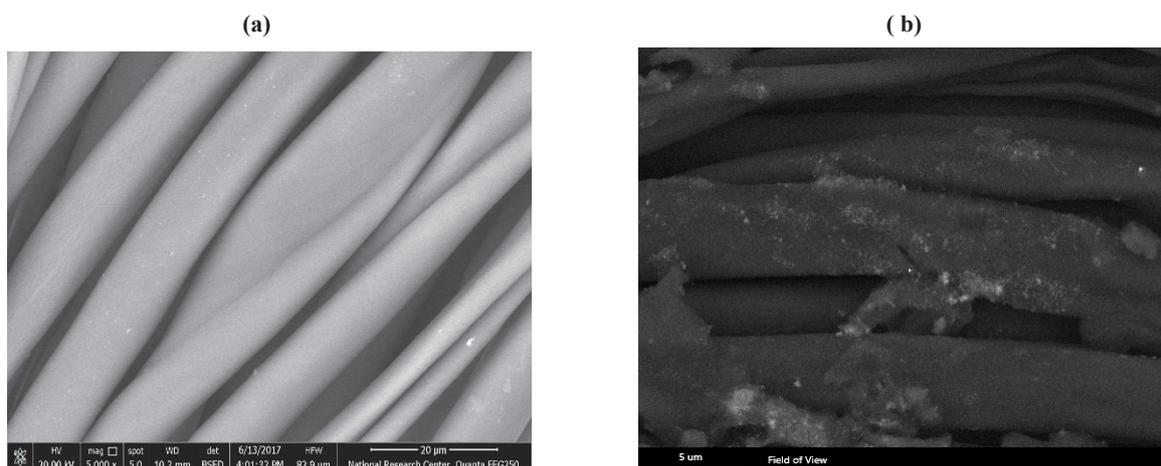


Fig. 1. SEM images of the untreated(a) and treated(b) polyester with caustic soda. [Conc. = 60 g/dm³, 100 °C, 65min].

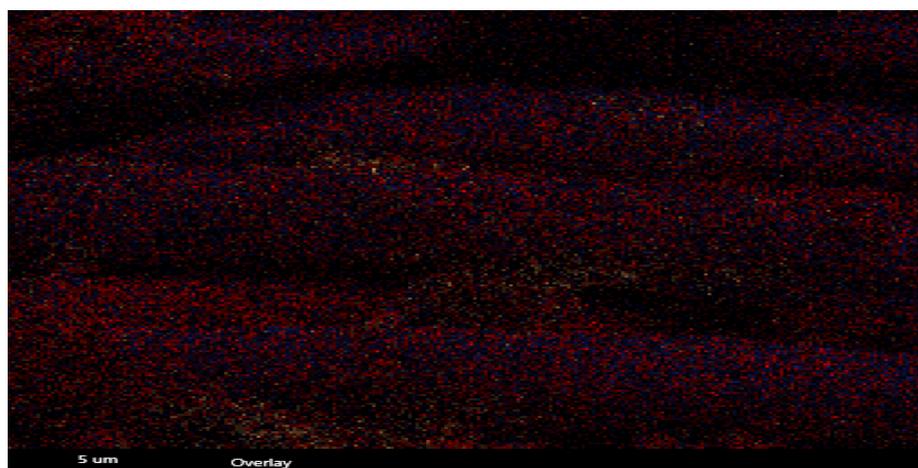


Fig. 2. Mapping scan of the treated polyester fabric with caustic soda. [Conc. = 60 g/dm³, 100 °C, 65min].

FT-IR Studies

The FT-IR spectrum of polyester/ cottonblend fabric before and after caustic soda and chitosan treatment was recorded in Fig.4 (a and b) to assess structural change if any made in the fabric of the alteration of existing functional groups. It was found, from the spectra, that the patterns are almost identical for both treated and untreated samples without any additional peaks. However, on comparing the samples treated with caustic soda, caused a slight shift in the position of the peak to a higher wave number with respect to untreated sample. The extent of shift was found to be dependent on the applied treatment. A broad peak at 1730 cm^{-1} is characteristic of C=O stretching of unsaturated ester, while the broad

bands at 1225 and 1235 cm^{-1} are characteristic of C-O in untreated and treated sample by caustic soda. A small peak in the region between 800 and 1000 cm^{-1} can be accounted for out-of-plane bending of aromatic ring system. An intense peak at $2250\text{--}2118\text{ cm}^{-1}$ can be attributed to methylene C-H stretching. The small peak close to 2955 cm^{-1} can be correlated to C-H stretching of aromatic ring. Sharp small peaks observed at around 3484 and 3797 cm^{-1} corresponds to free -OH groups of cellulose component accounting successful alkali treatment of the fabric. The observed small peaks between the regions $1110\text{--}1150\text{ cm}^{-1}$ were due to cellulosic component of the fabric materials [24]. This trend has been supported by the results of SEM studies.

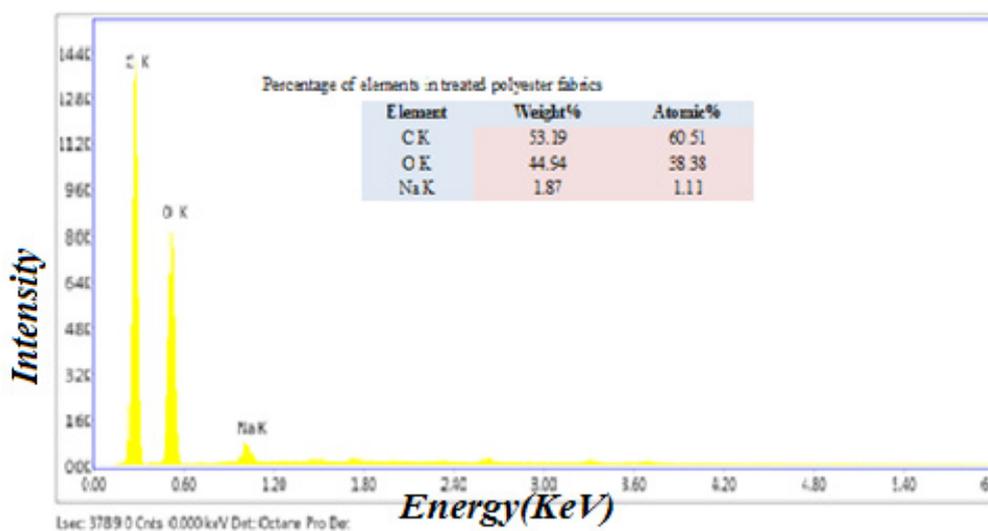


Fig. 3. EDX analysis of treated polyester fabric with caustic soda [C = 60 g/dm^3 , 100°C , 65min].

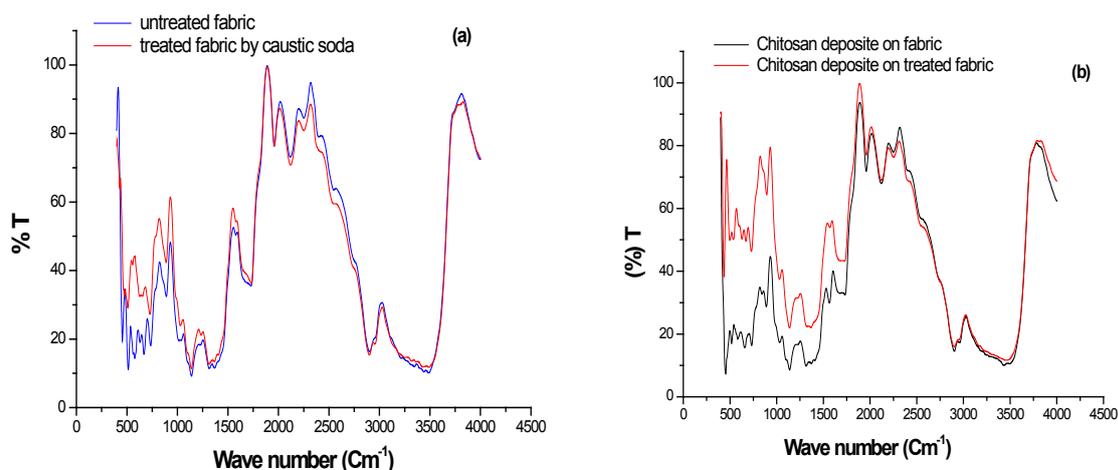


Fig. 4. FT-IR spectrum for untreated and treated polyester/ cotton blend fabric with (a) caustic soda [C = 60 g/dm^3 , 100°C , 60 min] (b) chitosan [7 g/dm^3 , 25°C , 60 min].

Determination of the content of end carboxyl groups of modified polyester fabric

During the treatment of polyester fabric in alkaline solution, modified polyester fragments with terminating carboxyl or hydroxyl groups remain in polyester structure after removal of the products of hydrolytic degradation. Table 1 shows the relative increase of terminating carboxyl groups in alkali treated samples compared to the original sample. The relative increase is 1.0–3.194 times higher, because it is not possible to establish a simple relation between the number of terminal carboxyl groups and mass loss, i.e. higher mass loss does not necessarily mean higher number of terminal groups. Since the mass loss occurs when hydrolysis takes place at the ends of molecules, and that the number of terminal groups increases when hydrolysis takes place inside the polyester macromolecule, it can be concluded that polyester hydrolysis is a statistically random process for defined reaction conditions.

Kinetics of mass loss of treated polyester fabrics with caustic soda

The effect of caustic soda on polyester fabric is important in dyeing process of polyester/cotton blend fabrics, where low content of polyester carboxyl groups give rise to obvious hydrophobic polyester fabric surface and therefore it is inert to dyes dissociating in dyeing bath. Hydrophilization of polyester, introducing polar oxygen groups into macromolecular structure, can alter polyester behavior, i.e. enable dyeing at 100°C and possibly using dyes with low thermal stability, e.g. reactive dyes. Caustic soda treatment of polyester fabrics has favorable effects on some textile characteristics of practical importance, because fabrics have better aesthetic appearance, they are less prone to pilling, have higher resistance

to staining and better hydrophilic properties [17,25]. Figs. 5a, b show graphic presentation of fabric mass loss % versus time with different concentrations of caustic soda at 80 and 100°C. Prolonged reaction time progressively increases weight loss. A higher caustic soda concentration and higher solution temperature, in all cases, weight loss variation with time is linear. When polyester is treated in aqueous alkaline solution it loses weight due to nucleophilic substitution in such a way that hydroxyl ions attack electron deficient carbon atoms in carbonyl groups inducing ester group hydrolysis (Fig. 5b). Alkaline hydrolysis of polymers produces water soluble depolymerized polyester fragments that separate from fiber surface and transfer to solution, which is observed as a weight loss. Higher weight loss at higher temperatures is explained by increased alkaline diffusion in polyester fabric and is particularly pronounced at temperatures higher than polyester glass transition temperature. The sodium terephthalate (TPA-Na₂) and ethylene glycol are formed due to hydrolysis, by acidifying the reaction solution TPA-Na₂ is converted to terephthalate anion (TPA) as illustrated in the following mechanism (Scheme 3) [26]. Water droplet adsorption following alkaline treatment of polyester fabric. This may be due to physical or chemical variations in polyester fiber treated with alkaline. Alkaline treatment by developing pores and cracks improves the surface structure of the fibre. This increases polyester hydrophilicity and facilitates wetting of surfaces. The alkaline hydrolysis, in addition to physical changes, induces chain cleavage as OH⁻ attacks ester-bonded carbonyl carbon resulting in hydroxyl (-OH) and -COO groups on the fiber surface as shown in Scheme 3 [27].

TABLE 1. Relative increase of terminating carboxyl group of polyester fabric treated with caustic soda at different time.

| Sample No. | Time treatment, min | 30 g/dm ³ of caustic soda | | 60 g/dm ³ of caustic soda | | 90 g/dm ³ of caustic soda | |
|------------|---------------------|--------------------------------------|---------------------------------------|--------------------------------------|---------------------------------------|--------------------------------------|---------------------------------------|
| | | Terminating carboxylic group at 80°C | Terminating carboxylic group at 100°C | Terminating carboxylic group at 80°C | Terminating carboxylic group at 100°C | Terminating carboxylic group at 80°C | Terminating carboxylic group at 100°C |
| 1 | 0 | 1 | 1 | 1 | 1 | 1 | 1 |
| 2 | 25 | 1.024 | 1.198 | 1.780 | 1.804 | 2.640 | 2.989 |
| 3 | 45 | 1.023 | 1.354 | 1.716 | 1.945 | 2.712 | 2.954 |
| 4 | 65 | 1.102 | 1.323 | 1.841 | 1.997 | 2.700 | 3.012 |
| 5 | 85 | 1.187 | 1.498 | 1.840 | 2.112 | 2.797 | 3.194 |
| 6 | 100 | 1.185 | 1.601 | 1.841 | 2.356 | 2.823 | 3.112 |

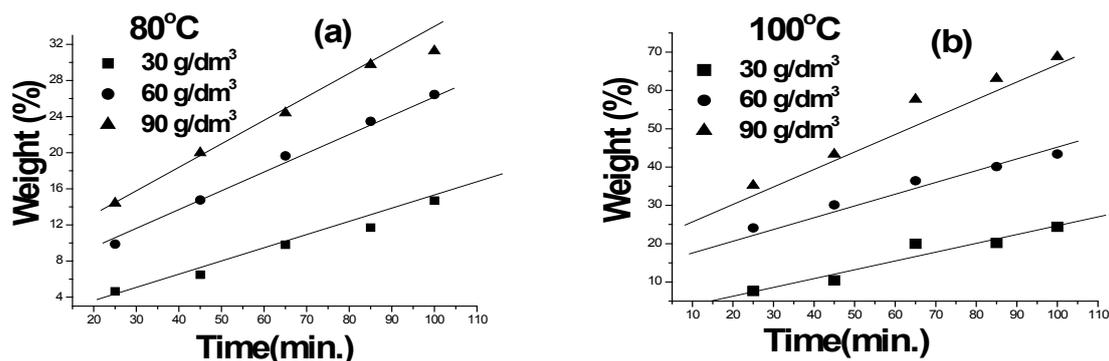
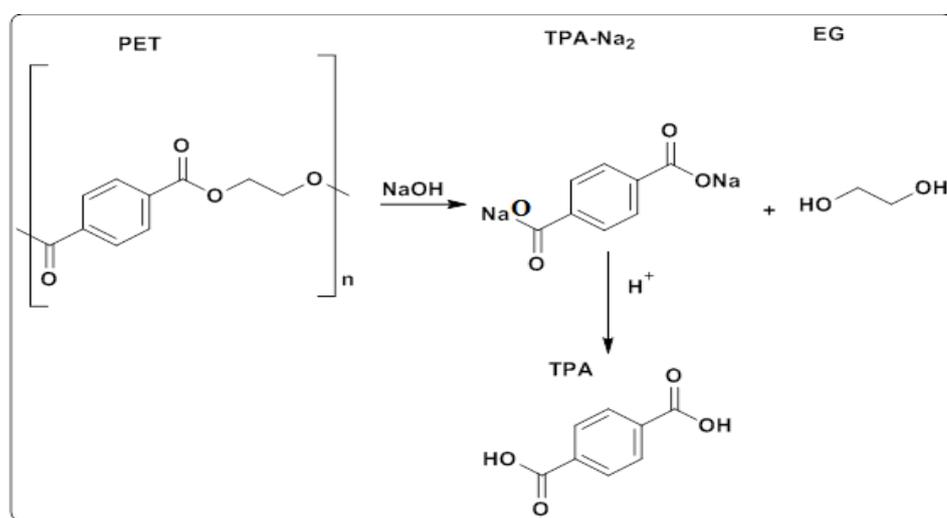


Fig. 5. Kinetic of weight loss % of polyester fabric treated with different concentrations of caustic soda at 80°C and 100°C.



Scheme 3. Polyester chain alkaline hydrolysis mechanism [27].

Dyeing of Polyester (100%), cotton (100%), and polyester/cotton blend fabrics (50 / 50) with RR198

To study the technical feasibility of dyeing process and functional finishing of textile fabrics as well as to attain proper dyeing and functional properties, caustic soda, chitosan as well as their hybrid have been investigated as modifiers. Results obtained along with their appropriate discussion follow.

One-bath two-stage dyeing of caustic soda treated polyester, cotton and polyester-cotton blend fabrics (50 / 50) with RR198

Polyester, cotton and polyester / cotton blend fabrics were dyed sequentially after caustic soda treatment with RR198 dye solution. Set of fabrics treated under conditions (different concentrations of caustic soda 0, 30, 60 and 90 g/dm³, treatment time 0, 20, 40, 60, 80 and 100 min)

at temperature 80°C were selected for dyeing purpose. Reactive dyes are the most important class of dyeing cellulosic fabric and their ability of chemical reaction with various textile fabrics makes them different from other dye classes. The importance of using one dye class for dyeing polyester-cotton blend fabric enhances the dye ability to be fixed by covalent bond on fabric substrate [11]. On the other hand, the use of one-bath two-stage rather than the two-bath dyeing method, reduce the cost of dyes and chemicals and causes reduction in process time. Also, it offers lower usage of water and chemical and a reduction in effluent volume [28,29].

The results in Table 2 indicate that the caustic soda treatments have improved the color strength *k/s* value of the dyed textile fabrics. This may be since the caustic soda pretreatments have improved the penetration of the dyestuff

molecules into the interior of the fabric matrix [30] and have improved the stability of dye-fiber bond [17].

The cotton fabric showed the highest k/s value due to the improved dye uptake by the reaction between reactive dye and the high reactive groups in the fabric cotton substrate. The caustic soda treated polyester/cotton blend fabric with 50% cotton portion gives high k/s values compared with those of the 100% polyester fabric. The 100% polyester fabric also shows good improvement ink/s values after caustic sodatreatment despite their lower values compared with the other two fabrics type.

Data of the percentage of color intensity increase was also recorded in Table 2 before and after caustic soda treatment. An increased trend of color intensity percentage for all modified samples was observed by increasing concentration of caustic soda and treatment time. For treated polyester fabric dyed with RR198, I%, increased from 3.54 to 100.51%, this may be attributed to the incorporation of new OH groups at the ends of depolymerized polyester matrix

[19]. Cotton fabric samples after caustic treatment give also higher values of the percentage of color intensity increase I% (1.42–88.04%) as a result of changes in supramolecular structure during treatment. When cotton is treated with caustic soda a good mercerization effect is achieved because cellulose crystal structure is modified, and percentage of amorphous regions is increased [31], enhance cotton swelling property and hence RR198 dye penetrates more easily through fabric interior layers. Modified polyester /cotton blend fabric have also better concurrent dyeing parameters and it can be asserted that I increase from 7.2–63.36 %. On modified samples, standard values for fixation F% Table 2 are observed, indicating again the stability of dye-fabric bond for the three types of textile materials. It is also clear that the dyeing results are more satisfactory with one- bath two- stage dyeing method using RR198. Therefore, it is anticipated one-bath two-stage method (Scheme 2) using the bifunctional reactive dye RR198 would be convenient for dyeing, polyester, cotton, and polyester/cotton blend fabrics throughout the dyeing cycle.

TABLE 2. Color strength, intensity and fixation % of reactive dyed polyester, cotton and polyester/cotton fabrics in presence of different concentrations of caustic soda at different time.

| Concs. of caustic soda | Time treatment, min. | Polyester fabrics | | | Cotton fabrics | | | Polyester/cotton fabrics | | |
|------------------------|----------------------|-------------------|--------|------------|----------------|-------|------------|--------------------------|-------|------------|
| | | K/S (525 nm) | I % | Fixation % | K/S (525 nm) | I % | Fixation % | K/S (525 nm) | I % | Fixation % |
| 0 | 0 | 1.98 | - | 52.56 | 9.87 | - | 79.82 | 6.25 | - | 62.23 |
| | 25 | 2.05 | 3.54 | 60.33 | 10.01 | 1.42 | 81.55 | 6.70 | 7.20 | 70.21 |
| | 45 | 2.07 | 4.55 | 63.66 | 10.56 | 6.99 | 85.32 | 6.89 | 10.24 | 70.89 |
| | 65 | 2.13 | 7.58 | 69.88 | 10.77 | 9.12 | 86.55 | 7.12 | 13.92 | 72.23 |
| | 85 | 2.19 | 10.61 | 72.31 | 10.88 | 10.23 | 88.11 | 7.34 | 17.44 | 72.87 |
| 30 gm/dm ³ | 100 | 2.25 | 13.64 | 78.33 | 11.05 | 11.96 | 88.78 | 7.54 | 20.64 | 73.28 |
| | 25 | 2.11 | 6.57 | 72.33 | 15.56 | 57.65 | 90.22 | 7.87 | 25.92 | 74.23 |
| | 45 | 2.29 | 15.66 | 75.12 | 16.01 | 62.21 | 94.11 | 8.14 | 30.24 | 75.61 |
| | 65 | 2.37 | 19.70 | 75.55 | 16.90 | 71.23 | 95.32 | 8.55 | 36.80 | 75.88 |
| | 85 | 2.56 | 29.29 | 75.99 | 17.43 | 76.60 | 95.99 | 8.97 | 43.52 | 76.01 |
| 60 gm/dm ³ | 100 | 2.74 | 38.38 | 76.11 | 17.56 | 77.91 | 96.56 | 9.12 | 45.92 | 76.45 |
| | 25 | 3.28 | 65.66 | 74.55 | 16.33 | 65.45 | 94.23 | 8.54 | 36.64 | 81.23 |
| | 45 | 3.45 | 74.24 | 76.33 | 16.98 | 72.04 | 94.88 | 8.97 | 43.52 | 82.12 |
| | 65 | 3.87 | 95.46 | 76.84 | 17.15 | 73.76 | 97.22 | 9.23 | 47.68 | 83.24 |
| | 85 | 3.88 | 95.96 | 77.14 | 18.00 | 82.37 | 98.99 | 9.78 | 56.48 | 83.88 |
| 90 gm/dm ³ | 100 | 3.97 | 100.51 | 77.88 | 18.56 | 88.04 | 99.56 | 10.21 | 63.36 | 84.56 |

One-bath dyeing of chitosan treated polyester, cotton and polyester / cotton blend fabrics (50 / 50) with RR198

The deposition of biodegradable polymer ecofriendly chitosan is one way to achieve multifunctional properties of textile, including better absorption. Chitosan is a biopolymer possessing reactive amino and hydroxyl groups and owing to its unique characteristics, chitosan has recently attracted scientific and industrial interests as a suitable adsorbent for dyes in textile dyeing and wastewater treatment. The concentrations selected from chitosan in our investigation was 1,7 and 14 g/dm³. k/s value of a dyed materials has a close relationship to the amount of dye absorbed by the fabric. k/s values, the percentage of color intensity increase I and fixation F % of chitosan treated polyester, cotton and polyester / cotton blend dyed samples with RR198 are shown in Table 3. It was observed that the color strength measurements of untreated fabrics have the lowest values. Before treatment the textile fabric samples when immersed in water produce a negative zeta potential. The negative charge on the fabric repels the RR 198 dye ions and consequently the exhaustion of the dye bath was limited which lead to the decrease of the color strength measurements. Cotton fabric record the highest k/s values followed by its blend with polyester and 100% polyester give the lowest values of color strength measurements with the chitosan pretreatment. This enhancement in k/s values of chitosan treated fabrics shows that

the chitosan has an incremental effect in dyeing processes. The improved dye ability is postulated due to the presence of both hydroxyl groups of chitosan layer on the surface of pretreated fabric that represent binding sites for additional quantities of reactive dye and amide groups (-CONH₂) available from the (chitosan). It is observed Table 3 that by increasing the chitosan concentration the k/s values have been increased up to 7 g/dm³ of chitosan concentration and then decreased when the concentration of chitosan reaches 14 g/dm³. This detraction in k/s values of chitosan treated fabrics is associated with the saturation of fabrics surfaces by chitosan.

For all samples, values of fixation F% were high especially for cotton fabric. Higher dye fixation means lower dye content in wastewater and this is based on the reaction of chitosan hydroxyl group with reactive center of monochlorotriazine mono azo tetra sodium sulphonyl dye. It is known that RR198 dye can react with cotton fabric hydroxyl group after adjusting pH with Na₂CO₃. Moreover, under alkaline conditions deprotonation of chitosan hydroxyl group also occurs [32].

One-bath dyeing of caustic soda – chitosan treated polyester, cotton and polyester / cotton blend fabrics (50/50) with RR198

Hybrid treatments of textile material combining surface modification of fabrics and deposition of compound with higher dye adsorption capacity are potentially the most effective for improved dye yield from technological solution. Polyester,

TABLE 3. Color strength, intensity and fixation % of reactive dyed polyester, cotton and polyester/cotton blend fabrics in presence of different concentrations of chitosan (Time treatment 60 min, at 25° C).

| Concs. of chitosan | Polyester fabrics | | | Cotton fabrics | | | Polyester/cotton blend fabrics | | |
|-----------------------|-------------------|--------|------------|----------------|-------|------------|--------------------------------|-------|------------|
| | K/S (525nm) | I % | Fixation % | K/S (525 nm) | I % | Fixation % | K/S (525 nm) | I % | Fixation % |
| 0 | 1.98 | - | 52.56 | 9.87 | - | 79.82 | 6.25 | - | 62.23 |
| 1 gm/dm ³ | 3.78 | 90.91 | 62.33 | 12.36 | 25.23 | 81.55 | 8.56 | 21.91 | 70.01 |
| 7 gm/dm ³ | 5.01 | 135.35 | 70.88 | 16.02 | 56.33 | 83.23 | 10.89 | 66.56 | 74.12 |
| 14 gm/dm ³ | 4.66 | 214.65 | 79.88 | 15.34 | 62.31 | 87.99 | 10.09 | 74.24 | 78.78 |

cotton and polyester / cotton blend fabrics were treated with 60 g/dm³ of caustic soda and 7 g/dm³ chitosan solutions at different time interval from 0-100 min at 25 °C, then dyed with RR198. An increase of all dyeing parameters was observed as it given in Table 4. Polyester fabric, sample treated with hybrid caustic soda-chitosan as well as cotton and its blend proportion 50/50 have higher color strength values reaches 5.48, 18.70 and 11.40 compared with 5.01, 16.02 and 10.89 when only chitosan for 60 min treatment time was used. The percentage of reactive dye intensity *I* of blended fabric was varied from 47.52–93.44% when the treatment time increases from 20-100 min Table 4 and it is significant in terms of economy and environmental protection, because with higher dye utilization degree from technological solution, the required dye quantity to achieve a specified color shade is reduced and dye quantity reaching waste water is also reduced. The fixation % was also higher and greater than the corresponding values obtained when separate utilization of caustic soda and chitosan was applied. For cotton fabrics, higher fixation % reaches 98.78 as it given in Table 4. This was attributed because cellulosic cotton material and chitosan hydroxyl groups fix the dye by covalent bond.

Fastness Properties of treated and untreated dyed textile Fabrics

Untreated and treated dyed polyester, cotton and polyester / cotton blend samples were tested

for various fastness properties such as light, wash and perspiration. Three measurements were carried out for each sample according to its specific ISO standard test methods. An average value given in Tables 5, 6 and 7 for caustic soda, chitosan, and caustic soda-chitosan hybrid-treatment respectively. It is inferred from the data in Tables 5, 6 and 7 that there is a maximum light and washing grade fastness value for treated cotton fabric as compared to the polyester and its blend with cotton. It is perhaps due to the strong attachment of the added modifier and the RR 198 active sites to all over the whole structure of the cotton fabric. The lower fastness grade properties of polyester with respect to the other fabric sample, might be due to the unwell-washed adhered dye particles onto fabric.

The caustic soda, chitosan and their hybrid deposited on polyester and its blend give remarkable improvement in the light and washing grade fastness properties in continuation of their good color values. Higher values mean lower dye content in wastewater as previously explained. The results in Tables 5, 6 and 7 show very good color fastness grade for cotton, good for its blend and poor for polyester towards both acidic and alkaline perspiration. The perspiration data of cotton sample assets the important requirement for comfort properties. In general, the results give very good indication for the treatment applied as a novel approach to textile dyeing and finishing.

TABLE 4. Color strength, intensity and fixation degree of reactive dyed polyester, cotton and polyester/cotton fabrics in presence of hybrid chitosan and caustic soda (60 g/dm³ of caustic soda and 7 gm/dm³ of chitosan).

| Sample No. | Time treatment, min. | Polyester fabrics | | | Cotton fabrics | | | Polyester/cotton blend fabrics | | |
|------------|----------------------|-------------------|--------|------------|----------------|-------|------------|--------------------------------|-------|------------|
| | | K/S (525 nm) | I % | Fixation % | K/S (525 nm) | I % | Fixation % | K/S (525 nm) | I % | Fixation % |
| 1 | 0 | 1.98 | - | 52.56 | 9.87 | - | 79.82 | 6.25 | - | 62.23 |
| 2 | 20 | 4.88 | 146.46 | 82.11 | 17.33 | 75.58 | 91.55 | 9.22 | 47.52 | 70.01 |
| 3 | 40 | 5.11 | 158.08 | 87.04 | 18.03 | 82.67 | 95.23 | 10.45 | 67.20 | 75.01 |
| 4 | 60 | 5.98 | 176.77 | 89.35 | 18.70 | 89.46 | 96.55 | 11.40 | 76.64 | 76.66 |
| 5 | 80 | 6.02 | 204.04 | 91.23 | 19.01 | 92.60 | 98.11 | 11.56 | 84.96 | 80.88 |
| 6 | 100 | 6.78 | 242.42 | 91.55 | 19.56 | 98.18 | 98.78 | 12.09 | 93.44 | 84.12 |

TABLE 5. Light, washing and perspiration fastness of reactive dyed polyester, cotton and polyester/cotton fabrics in presence of different concentrations of caustic soda.

| Concs.of caustic soda,g/dm ³ | Polyester fabrics | | | | Cotton fabrics | | | | Polyester/cotton fabrics | | | |
|---|-------------------|------|--------------|--------|----------------|------|--------------|--------|--------------------------|------|--------------|--------|
| | Light | Wash | Perspiration | | Light | Wash | Perspiration | | Light | Wash | Perspiration | |
| | | | acidic | alkali | | | acidic | alkali | | | acidic | alkali |
| 0 | 1-2 | 2 | 3-4 | 2-3 | 3-4 | 4 | 4 | 3 | 3 | 4 | 3 | 3 |
| 30 | 1-2 | 2 | 3-4 | 2-3 | 3-4 | 4 | 4 | 3 | 3 | 3-4 | 4 | 3 |
| 60 | 2 | 2 | 3-4 | 2-3 | 4-5 | 4-5 | 4-5 | 3-4 | 3-4 | 4 | 4 | 3 |
| 90 | 2 | 2 | 4 | 2-3 | 5 | 5 | 4-5 | 3-4 | 4 | 4-5 | 4 | 3 |

TABLE 6 . Light, washing and perspiration fastness of reactive dyed polyester, cotton and polyester/cotton fabrics in presence of different concentrations of chitosan (Time treatment 65 min).

| Concs.of chitosan g/dm ³ | Polyester fabrics | | | | Cotton fabrics | | | | Polyester/cotton fabrics | | | |
|-------------------------------------|-------------------|------|--------------|--------|----------------|------|--------------|--------|--------------------------|------|--------------|--------|
| | Light | Wash | Perspiration | | Light | Wash | Perspiration | | Light | Wash | Perspiration | |
| | | | acidic | alkali | | | acidic | alkali | | | acidic | alkali |
| 0 | 1-2 | 2 | 3-4 | 2-3 | 3-4 | 4 | 4 | 3-4 | 2-3 | 3 | 4 | 3-4 |
| 1 | 2 | 2-3 | 3-4 | 2-3 | 4 | 5 | 4-5 | 3-4 | 3-4 | 4-5 | 4 | 3-4 |
| 7 | 2 | 3 | 4 | 3 | 5 | 5 | 4-5 | 4 | 4 | 5 | 4 | 3-4 |
| 14 | 2-3 | 3-4 | 4 | 3 | 6 | 5 | 4-5 | 4 | 5 | 5-6 | 4 | 3-4 |

TABLE 7. Light, washing and perspiration fastness of reactive dyed polyester, cotton and polyester/cotton fabrics in presence of hybrid chitosan and caustic soda (60 g/dm³ of caustic soda and 7gm/dm³ of chitosan).

| Time treatment, min. | Polyester fabrics | | | | Cotton fabrics | | | | Polyester/cotton fabrics | | | |
|----------------------|-------------------|------|--------------|--------|----------------|------|--------------|--------|--------------------------|------|--------------|--------|
| | Light | Wash | Perspiration | | Light | Wash | Perspiration | | Light | Wash | Perspiration | |
| | | | acidic | alkali | | | acidic | alkali | | | acidic | alkali |
| 0 | 1-2 | 2 | 3-4 | 3 | 3-4 | 4 | 4 | 3-4 | 2-3 | 3 | 4-5 | 3-4 |
| 25 | 2 | 2-3 | 3-4 | 3 | 4 | 4 | 4 | 4 | 3-4 | 4-5 | 4-5 | 3-4 |
| 45 | 2-3 | 2-3 | 4 | 3 | 4-5 | 4-5 | 4-5 | 4 | 3-4 | 5 | 4-5 | 3-4 |
| 65 | 3 | 3 | 4 | 3 | 5 | 5-6 | 5 | 4 | 4 | 5 | 5 | 4 |
| 85 | 3 | 3-4 | 4 | 3 | 5-6 | 6 | 5 | 4 | 4-5 | 5 | 5 | 4 |
| 100 | 3-4 | 4 | 4 | 3 | 6 | 6 | 5 | 4 | 5 | 5 | 5 | 4 |

Conclusion

In this study, the process of dyeing polyester, cotton and polyester/cotton fabrics (50/50) using reactive dye (RR198) in one bath- two-stage method dyeing process was investigated.

In the order to improve the surface functional and finishing properties of polyester, cotton and polyester/cotton (50/50) fabrics, pre-treatment with caustic soda, chitosan and their hybrid was

performed. The viability of using caustic soda as an auxiliary for alkaline dyeing of polyester and its cotton blend is explored. The result indicates the effectiveness of using caustic soda. An increased trend of color strength and fastness percentage for all modified samples was observed by increasing concentration of caustic soda and treatment time., this may be attributed to the incorporation of new OH groups at the ends of depolymerized polyester matrix and the good mercerization effect that is

achieved by cotton. This success in process optimization would further suggest future work for the use of caustic soda in textile coloration. The chitosan-deposited, polyester, cotton, and polyester/cotton blend fabrics were assessed. by increasing the chitosan concentration, the (K/S) values has been increased up to 7 g/dm³ of chitosan concentration and then decreased when the concentration of chitosan reaches 14 g/dm³. This detraction in (K/S) values of chitosan treated fabrics is associated with the saturation of fabrics surfaces by chitosan. The data obtained shows that it is possible to dye polyester/cotton fabrics finished by chitosan with only one reactive dyestuff.

Hybrid treatments of textile material by combining 60 g/dm³ of caustic soda and 7 g/dm³ chitosan solutions at different time interval from 0-100 min at 25°C are potentially the most effective for improved dyeing parameters. The percentage of reactive dye intensity I of blended fabric was varied from 47.52–93.44% when the treatment time increases from 20-100 min and it is significant in terms of economy and environmental protection. For cotton fabrics, higher fixation % reaches 98.78 was obtained. This was attributed because cellulosic cotton material and chitosan hydroxyl groups fix the dye by covalent bond. Untreated and treated dyed polyester, cotton and polyester / cotton blend samples were tested for various fastness properties such as light, wash and perspiration. A significant improvement of the color fastness of washing, and light has been observed owing to the covalent bond formed between the reactive group of dye and the functional group of textile fabrics. The washing grade fastness value exhibit excellent rates at 4-5 which achieved the requirements for industrial use. The lower fastness grade properties of polyester with respect to the other fabric sample might be due to the unwell-washed adhered dye particles onto fabric. The perspiration data of cotton sample assets the important requirement for comfort properties. In general, the results give very good indication for the treatment applied as a novel approach to textile dyeing and finishing

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تحسين معاملات صباغة الأقمشة مزيج البوليستر / القطن بواسطة الصودا الكاوية ، الشيتوزان ، ومزيجهما معا

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تم زيادة إنتاج ألياف البوليستر ديناميكياً ، بسبب الاستخدام الشائع لمزج القطن والبوليستر. في هذا البحث ، أقمشة البوليستر (100%) ، القطن (100%) ، أقمشة البوليستر والقطن (50/50) (تعرضوا للمعالجة باستخدام الصودا الكاوية ، الشيتوزان ومزيج منهما ، يعقبة الصباغة في حمام واحد ذومرحلتين مع الصبغة الحمراء النشطة (RR) 198 ، لدراسة أداء الصباغة والشكل الخارجي للنسيج . صور SEM للبوليستر المعالج والغير المعالج بواسطة الصودا الكاوية تصف سهولة امتزاز القلوي علي سطح القماش المعدل. تم استخدام طريقة حمام واحد من خطوتين ومن هذا توصلنا لتقليل وقت عملية الصباغة ، وزيادة المنتج وتقليل التكلفة الكيميائية. في المرحلة النهائية ، تم اختبار الأقمشة المصبوغة لمعرفة شدة اللون (ك / ث) ، وكثافة الصبغة ، ونسبة استنفادها بالإضافة إلى خصائص الثبات التي تم تقييمها عن طريق اختبار الثبات ضد الضوء والغسيل والعرق للصبغة. أظهرت النتائج بوضوح أنه بالنسبة للنسيج المعالج بالصودا الكاوية بالشروط
(90g/dm³ of caustic soda, 100 min ; 80oC)

أمتصاص ايجابي للصبغة النشطة الحمراء RR 198 ، مما ينتج عنه شدة لون عالية ونسبة استنفاد الصبغة بسبب تعديل سطح البوليستر وتقوية نسجة القطن. تم التحقق في تأثير المعالجة بالشيتوزان علي قابلية الصبغة وخصائص الثبات ، وبعض الخصائص الفيزيائية والكيميائية. من المحتمل أن تكون المعالجات المختلطة للمواد النسيجية عن طريق الجمع بين القلوبات والشيتوزان هي الأكثر فاعلية لتحسين شدة اللون ونسبة استنفاد الصبغة ويمكن تطبيقها كنهج جديد لصباغة النسيج.