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EFFECT OF TANNIC ACID ON THE DYEING PROCESS OF NYLON 6 FABRIC WITH CATIONIC DYE

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Abstract

Nylon 6 fabric was pretreated with polar surfactant, tannic acid and subsequently dyeing with the cationic dye (Rhodamine B) solution as well as emulsion phase. The emulsion phase consisted of n-hexadecane emulsified by isopropyl alcohol and stabilized by tannic acid/Rhodamine B complex. Different factors affect the pretreatment and dyeing process have been studied. Moisture regain, tensile strength, elongation and binding stiffness were investigated. The colour strength values showed that the pretreated fabric with tannic acid which dyed with cationic dye solution as well as emulsion system were enhanced. However, dyeing the nylon 6 fabric using emulsion system improved the dyeability and fastness properties. Interestingly, the dyed fabrics with cationic dye emulsion system exhibited excellent fastness properties aftertreating with the commercial anionic fluorescent whitening agent Uvitex RSB 150%. FTIR spectra have been studied.

Introduction

In particular, nylon 6 is the most widely used synthetic fibres for commercial fabric production. It is a macromolecule whose structural units are interlinked by the amide linkage (-NHCO-) and has a highly crystalline structure. Nylon 6 contains amine and carboxylic acid terminal groups. By selecting appropriate dyeing conditions, the anionic, nonionic and cationic dyes can be used [1].

Nylon 6 fibres are not easily dyed with cationic dyes, in order to improve their dyeability toward cationic dyes some anionically modification on these fibres must be done either by chemically anionic agent [2,3] or by physico-chemical surface treatments (low temperature plasma treatment and UV excimer laser irradiation) and polymerization of acrylic acid and other monomers [4-6].

Dyeing nylon 6 film and leacril fabric pretreated with tannic acid using cationic dye solution has been studied previously [7,8]. However , the obtained results indicate that the absorption was enhanced but the fixation was not satisfactory. Hence , it is worthmention to investigate a new possibilities to increase both the dye uptake and dye fixation on nylon fabric with good fastness properties.

Earlier studies also reported that full backtan, syntan and syntan-polymeric cationic agent aftertreatments can improve the wet fastness properties of premetallised acid dyes on nylon [9,10] .This two-stage aftertreatment process results in the formation of a large molecular size, low aqueous solubility complex between the anionic syntan and the cationic compound within the dyed fabric. Furthermore, two-stage syntan-metal salt aftertreatment raised the level of wash fastness of the dyed sample closely approaching that expected to full backtan [11,12].

Other studies reported that the application of tannic acid/ Rhodamine B cationic dye emulsion system can improve the dye adsorption on leacril fabric [13] .

The preset work was undertaken to investigate the dyeing of nylon 6 fabric with cationic dye emulsion system . The efficiency of this cationic dye emulsion system could enhance the wet fastness properties of the dyed nylon 6 fabric .

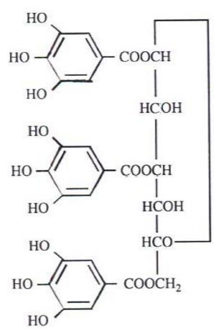
Experimental

Materials

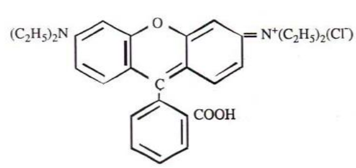
Scoured knitted nylon 6 (200g/m^2) supplied from El-Shourbagy Co., Cairo, Egypt was employed. The fabric samples were washed with a solution containing 5g/l non-ionic detergent (Hostapal® CV-ET, Clariant), and 2g/l sodium carbonate at 60°C for 30 min. Then the fabric was thoroughly washed with water and air dried at room temperature, before the dyeing process.

Chemicals and dyestuff

I-The tannic acid ($C_{76}H_{52}O_{46}$) (1) and the cationic dye Rhodamine B (C.I. Basic Violet 10) (2) used in these experiments were A.R. grade from Sigma Chemical Co. (Merk) used without further purification. All solutions of both tannic acid and Rhodamine B were prepared with bidistilled water. The molecular structure of both were as follows:



(1)



(2)

II-n-Hexadecane was purchased also from Merck. III- Uvitex® RSB liquid 150% fluorescent whitening agent was purchased from Ciba Co. IV-The used other chemicals and reagents were commonly of laboratory reagent grade.

Pre-Treatment of nylon 6 fabric with tannic acid

The nylon 6 fabric samples (1g) were pretreated with tannic acid. A range of concentrations of tannic acid from 5%-30 % (owf) was employed at liquor ratio 1:25, in each case for 1hr at 50°C. After treatment, the fabrics were washed with tap water and dried at room temperature.

Preparation of the emulsion system

The emulsion of Rhodamine B was prepared by dissolving 0.15 ml of n-hexadecane in 7.7 ml (1 M) of isopropyl alcohol placed in 100 ml Stoppard flask. Then 10 ml of 10^{-1}, 10^{-2}, 10^{-3} and 10^{-4} M tannic acid was added as a stabilizing and/or mordanting agent in which 10^{-3}M (0.048 g) of the cationic dye was dissolved. The mixture was stirred well and bidistilled water was added up to a volume ca. 50 ml with continuous stirring for 30min. Next, water was added to reach the final volume of 100 ml. Finally, the obtained red emulsion was further stirred at 1000 rpm for another 30 min. [13].

Dyeing conditions

Nylon 6 fabric was dyed with both solution and emulsion of (C.I. Basic Violet 10) cationic dye. In both system the dye concentration was 10^{-3}M (0.048 g/l). The liquor ratio was 1:25 and the pH values of the dyebath was adjusted to different values by the addition of dilute acetic acid and sodium carbonate solution. Dyeing process was carried out at various temperatures (55°C-95°C), for time of different duration (10-180 minutes). The dyed samples were then rinsed with cold water, washed in a bath containing 5 g/l non-ionic detergent with liquor ratio 1 : 25 at 50C° for 30 minutes. Finally the dyed washed fabric was rinsed and allowed to dry in air.

Fixing method

Part of each dyed sample was treated with crosslinking bath containing 1% owf fluorescent whitening agent (Uvitex® RSB liquid 150%), 3 g/l glauber salt at a liquor ratio 1: 20, the pH was adjusted to 5 with acetic acid at 50°C for 30 min. The samples were finally rinsed, dried and conditioned.

Measurements

Measurement of moisture regain

Moisture regain was performed according to the standard ASTM method 2654-76 [14]. It was calculated according to the following eqn. 1:

$$Moisture\ regain\,\% = \frac{w_1 - w_2}{w_2}\, X\, 100 \qquad (1)$$

Where W_1 : Weight of sample (g) after saturation in the standard humidity atmosphere

W_2 : Constant weight of dry sample.

Measurement of tensile strength and elongation

The tensile strength and elongation properties of nylon 6 fabrics before and after treatment with tannic acid were evaluated using a TTM-II-100 tensiometer (Toyo Bldwin, Japan). The average dimensions of the samples were 5x25 cm [15].

Measurement of bending stiffness

The Shirley stiffness tester was used according to the B.S. 3356 : 1961, Determination stiffness of cloth. Three specimens warp way and three weft were tested. Each specimen was tested four times, at each end again with strip turned over. The bending stiffness was calculated according to Eqn.2 and 3 [16].

$$C = l f_1(\theta) \qquad (2)$$

$$f_1(\theta) = \left(\frac{\cos {}^1/_2\ \theta}{8 \tan \theta} \right)^{\frac{1}{3}} \qquad (3)$$

where C : Bending length, l : length and θ : standard angle of deflection of 41.5°, at which f (41.5°) =0.5

Colour measurements of dyed samples

The reflectance of dyed and washed samples was measured by perkin-Elmer Lambda 3B UV/V is spectrophotometer at wavelength 555 nm. The colour strength (K/S) was calculated using the Kubelka- Munk equation (Eqn. 4) [17].

$$K/S = \frac{(1-R)^2}{2R} \qquad (4)$$

FTIR analysis

Fourier-transform infrared (FTIR) spectra were recorded on a Nexus 670 FTIR Spectrometer, Nicolet company, USA using potassium bromide disks. A total of 32 scans for each sample were taken with a resolution of 4 cm^{-1}, which range 4000-400 cm^{-1}.

Fastness testing

The dyed and washed samples were tested, according to ISO standard methods. The specific standard tests were : ISO 106-CO2 (1993) for wash fastness, ISO 105-X12 (1987) for rubbing fastness and ISO 105-EO4 (1989) for fastness to perspiration.

Results and Discussion

Physical properties of modified fabric

The physical properties of modified and unmodified nylon 6 fabric pretreated with different amount of tannic acid as an anionic agent are given in Table 1. The moisture regain gradually increases as the amount of tannic acid increased within the range of 5-15%owf ,propably due to the higher content of carboxylic groups of the treated fabric. However, a marginal decrease in moisture regain was detected at higher tannic acid concentration(20-30%).This may be attributed to the higher tannic acid aggregation on the fibre surface which results in the formation of multilayers of the adsorbed tannic acid molecules and this can be also demonstrated by the results of bending stuffiness.

The tensile strength show a good increase as the amount of tannic acid increased from 5-30% owf, probably due to the higher physical interactions between phenolic hydroxy groups of the acid and the carboxyl end groups of nylon 6 fabric. On the other hand, elongation at break was also increased from 137% for untreated to 162% pretreated fibre with 15% owf tannic acid indicating that, this concentration make the surface of the fibre slightly hydrophobic. Although, the pretreated fabric with 20% and 30% owf tannic acid show less elongation at break than the untreated sample. This result may be attributed to a very high concentration of tannic acid showing a weight increase greater than 1% i.e. $\simeq$ more than 10-15% owf tannic acid produces a stiff (not flexible fibres) to the touch.

The results of both flexural rigidity and bending length show the bending stiffness of fabrics. It was found from these results that all the flexural rigidity and binding length increased. However, it was also observed that a higher increase in the bending stiffness with higher concentration of tannic acid (20-30 % o.w.f.) as compared to that of low concentration of it (10-15% o.w.f.). This increment in stiffness may be due to the decrement in the amorphous content of the treated fabric and/or the high molecular weight of tannic acid which fill all the amorphous space in the fibre.

Effect of tannic acid concentration in the emulsion

The results in Figure 1 clearly indicate that as the concentration of tannic acid increased from 10^{-4} to 10^{-1}M, the colour strength (K/S value) also markedly increased till it reached maximum at 10^{-2}M. From 10^{-2}M to 10^{-1}M a slightly increase in K/S value was observed in the emulsion complex. It can be worthy mentioned, that dyeing process of nylon 6 fabrics with emulsion system is of a superior efficiency, especially when 10^{-2}M tannic acid is used as a stabilizing agent.

Dyeing behaviour of emulsion system

Dyeing nylon 6 fabric with a cationic dye, C.I. Basic violet 10, from the emulsion phase and in the presence of tannic acid as a naturally stabilizing agent (10^{-2}M) show a much higher K/S value than the dye solution alone. These high efficiency of the emulsion system as shown in Figure 2 may be due to the following reasons :

1. Hydrocarbon n-Hexadecane emulsified by isopropyl alcohol form a thin uniform protective film coating nylon 6 fabric as a plasticizing agent which can lead to lowering the electron donor interaction of the nylon 6 surface and or shielding of these carboxyl function groups .
2. C.I. Basic Violet 10 molecules interact by hydrogen bonding with tannic acid which in turn ,results in stabilizing the emulsion droplets in hexadecane. This means that the high molecular structure of tannic acid can perform as a good stabilizing agent for both oil in water and water in oil emulsions and these observation support the above discussion.
3. Therefore, the K/S value was highly increased in the case of dyeing nylon 6 fabrics with the emulsion system as a result of lower energy needed for adsorption and faster diffusion of cationic dye from emulsion system than dye solution system.
4. These may conclude that the adsorption process in both systems is a diffusion controlled[13].

Effect of pH

Figure 3 shows the effect of pH on the colour strength of both dye solution and dye emulsion phase. For the determination of the optimum pH, the nylon 6 fabrics was dyed with different pH values (3-9), the maximum colour strength and level dyeing were observed in a moderately acidic medium (3-5) as shown in the figure, using dye emulsion system. Above pH 5, the nylon 6 surface is negatively charged and the K/S value decreased markedly due to the repulsion of the emulsion particles from nylon 6 in alkaline medium when carboxylic groups of tannic acid and nylon 6 are negatively charged. In acidic medium before pH 5, this repulsion force is decreased which supports hydrophobic interactions between the components of the emulsion (hexadecane) from one side and nylon 6 from an other. In acidic pH tannic acid and nylon 6 also interacts due to non polar forces.

Effect of tannic acid concentration as an anionic agent

Figure 4 shows that as the amount of tannic acid increased from (0-10% o.w.f.) on the pretreated fabric, K/S values also increased markedly on dyeing nylon 6 fabric with cationic dye solution, then leveling occurs above 10% owf of tannic acid. This fact indicates the presence of the additional active sites of the phenolic hydroxy groups of the tannic acid on the pretreated nylon 6 fabric beside the ionized carboxyl groups of the fibre which interact with the cationic dye solution. In our opinion, this increased in K/S value of cationic dye solution beside the electrostatic interaction, probably, is due to the formation of hydrogen bonding between phenolic hydroxy groups of the acid and the carboxylic groups of Rhodamine B of cationic dye on the fibre. On the other hand, increasing the of tannic acid as anionic agent for nylon 6 fabric has no effect on the dyeing process with the cationic dye emulsion system. Since cationic dye complex behaves like a nonionic dye, for this reason, its K/S value is independent on the concentration of tannic acid on the fabric as shown in Figure 4.

To obtain more information about the dyeing process of untreated and 10% owf tannic acid treated nylon 6 fabric by both the solution and emulsion phase of the cationic dye used, different parameters such as dyeing temperature and time, FTIR analysis and fastness properties were determined.

Effect of dyeing temperature

It can be seen from Figure 5, if cationic dye (10^{-3}M) was adsorbed from the emulsion system for both untreated and treated fabric, colour strength increased markedly with increasing temperature, till it reach 95°C. However, in case of dye solution the treated fabric show a higher enhancement in the colour strength than untreated fabrics. Generally, the gradually increase in dyeability at higher temperature for both treated and untreated fabrics using the dye emulsion system was more favour than the fast increment of the treated fabrics with dye solution. These results may be dye to the higher activation energy for the diffusion process in the emulsion system and hence enhanced dye diffusion than in the dye solution [13].

Effect of dyeing time

The effect of dyeing time was investigated, and the results are shown in Figure 6. It can be seen that the colour strength of the treated fabric dyed with both dye solution and emulsion system was much higher than the untreated fabric dyed with dye solution as the time increased. Morever, the dye fixation was significantly higher on the treated fabric dyed with dye solution than the untreated one, but still less than dyeing with emulsion system. The dyed fabric exhibited more fixation using both solution and emulsion system with increasing the concentration of tannic acid as an anionic agent and/or stabilizing agent.

FTIR analysis

FTIR analysis was carried out on all the treated and untreated dyed fabric with both dye solution and emulsion system and the spectra of unmodified and modified fabrics are given in Figure 7. In the spectrum of the nylon 6 fabric, the peak at 3431.70, 3401.32, 3418.43 and 3419.18 cm^{-1} confirm the presence of the broad intermolecular hydrogen bonded (OH) between the phenolic hydroxy groups of tannic acid and the carboxyl groups of the dye molecule and dye complex. However, the absorbance increase from sample A to D which may indicate that the intensity of the hydrogen bond also increased as the fabric was pretreated with tannic acid followed by dyeing with emulsion system. These mean that the intensity of the intermolecular hydrogen bond increased as the concentration of tannic acid increase as anionic and/or stabilizing agent. A distinct band can be observed in spectra C and D within 2923.70 and 2856.54 cm^{-1}, 1454 and 1368.29cm^{-1} corresponding to aliphatic C-H stretching, bending deformation and rocking vibration of methylene groups (CH_2) which were seen to have virtually disappeared in spectrum A and B. These results may be due to the presence of more cationic dye molecules on the fibre. In the modified pretreated fabrics with 10% o.w.f tannic acid certain absorption bands can be observed at 1645.72 or 1625.24, band 1425.06 or 1463.02 cm^{-1} characteristic to amide group (N-C=O) and the stretching vibration of C=O and C-N group. In our opinion, this fact confirm the presence of the electrostatic interaction between the phenolic carboxylic groups of tannic acid and the amine groups of the Rhodamine B on the treated fabric dyed with cationic dye solution system.

Fastness properties

The washing, rubbing and perspiration fastness properties of untreated and treated nylon 6 fabric, followed by dyeing with both dye solution and emulsion system before and after adding 1% o.w.f Uvitex® RSB liquid 150% are given in Tables 2 and 3 respectively. The washing and rubbing fastness properties in Table 2 reveal that dyeing nylon 6 fabric with cationic dye complex in the emulsion system show a better fastness than cationic dye solution system. This may be attributed to the large molecular size of such complex system , preventing the dye to be easily lost in washing. It is also evident from Table 2 that the fluorescent whitening agent Uvitex® RSB liquid 150%, of being an anionic crosslinking agent based on disulphonic acid triazine derivatives would enhance the ionic interaction between the anionic sulphonic groups and the plenty of cationic dye site on the nylon fabric surface. This would be expected to improve the wet fastness properties. Table 3 shows that cationic dye complex in emulsion system dyeing and followed by aftertreatment with anionic crosslinking agent situated at the dyed substrate display a very good perspiration fastness properties. This results confirm and support the presence of more molecules of Rhodamine B onto the fibre surface .

Conclusions

This study focuses on the role of tannic acid as stabilizing agent with a cationic dye complex Rhodamine B, from the emulsion system and as anionic agent in the treatment of nylon 6 fabric before dyeing process. The pretreated fabric with 10% o.w.f. tannic acid showed slightly increase in the tensile strength and elongation %. Higher moisture regian and binding stiffness were observed with increasing the amount of tannic acid. Nylon 6 fabric treated with 10% o.w.f. tannic acid followed by dyeing with cationic dye solution enhance the dyeability. These results may be due to the formation of the hydrogen bonding between the carboxyl group of the dye molecule and the hydroxy phenolic groups of tannic acid, besides electrostatic interaction between the amine groups of Rhodamine B and the carboxylic groups of the treated fabric. Moreover a higher dye uptake was achieved in dyeing nylon 6 fabric with emulsion system complex and also lead to an enhancement in the wet fastness properties than dyeing with dye solution. FTIR analysis confirm this result.

The washing and rubbing fastness of nylon 6 fabrics dyed with emulsion system complex are generally very good. Interestingly, The fixation can reach 90-95% in the emulsion system after adding anionic crosslinking fluorescent whitening agent Uvitex RSB. The perispiration fastness showed a good improvement specially in the acidic medium. Future work with this dyeing system will studies on using an other stabilizing agent to understand the dyeing mechanism of different fabric.

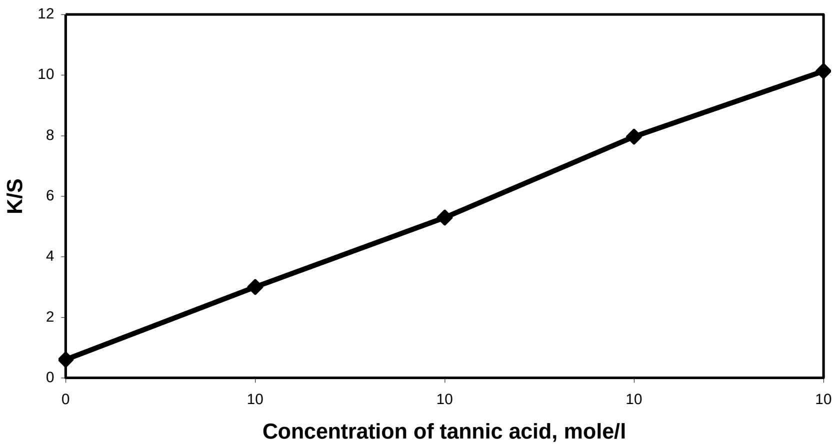


Figure 1 Effect of the concentration of tannic acid in the emulsion system of the cationic dye on the colour strength of nylon 6 fabrics. Dyeing conditions: 10^{-3} M of C.I. Basic Violt 10, liquor ratio 1 : 25, pH = 6 at 95°C for 60 .

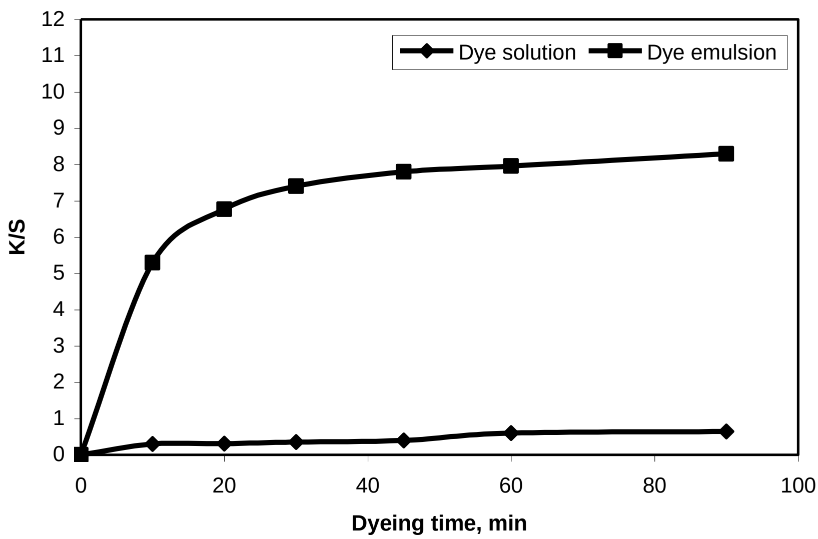


Figure 2 Effect of dyeing process with the emulsion system of the cationic dye on the colour strength of nylon 6 fabrics. Dyeing condition: 10^{-3}M C.I. Basic Violet 10, liquor ratio 1 : 25, pH = 6 at 95°C for 60 min.

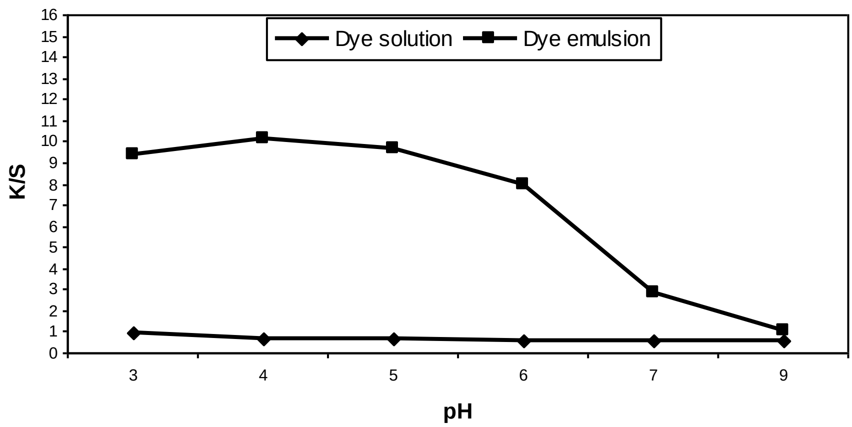


Figure 3 Effect of pH values on the colour strength of dyed nylon 6 fabrics with cationic dye. Dyeing conditions : 10^{-3}M C.I. Basic Violet 10, liquor ratio 1 : 25, at 95°C for 60 min.

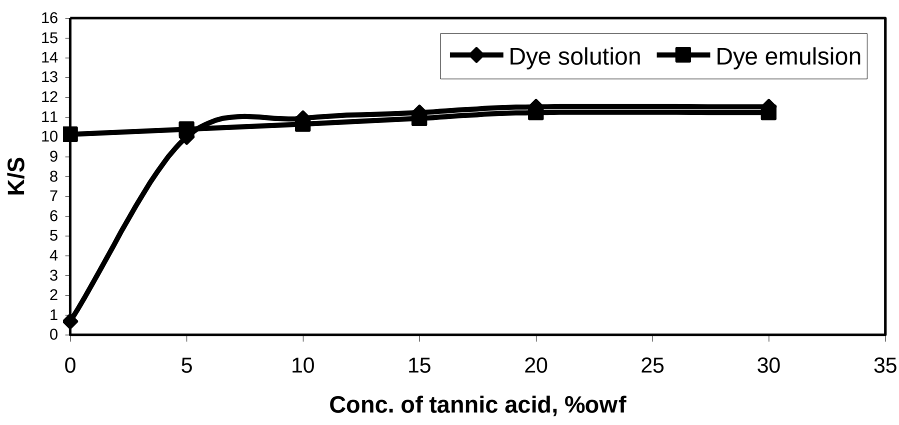


Figure 4 Effect of different concentration of tannic acid on the colour strength of dyed nylon 6 fabrics. Pretreatment conditions : Liquor ratio. 1:25 at 50°C for 60 min. Dyeing conditions : 10^{-3}M C.I. Basic Violet 10, liquor ratio 1 : 25, pH = 4 at 95°C for 60 min.

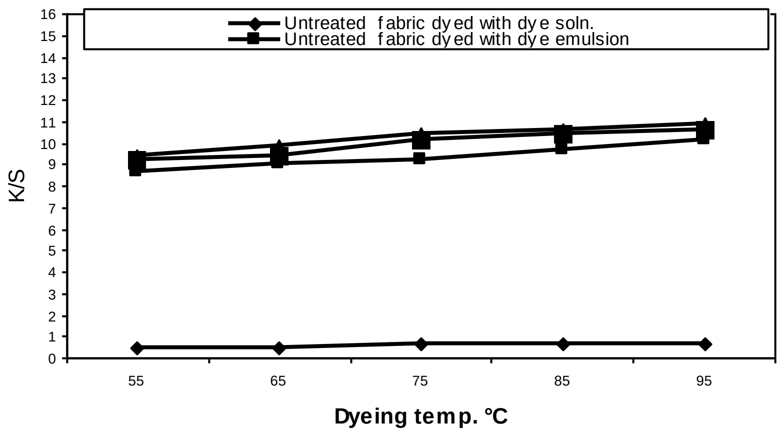


Figure 5 Effect of different dyeing temperature of untreated and pretreated nylon 6 fabric on the colour strength of dyed nylon 6 fabrics. Pretreatment conditions : 10 % owf tannic acid, liquor ratio. 1:25 at 50°C for 60min. Dyeing conditions: 10^{-3} M C.I. Basic Violet 10, liquor ratio 1 : 25, pH = 4 for 60 min.

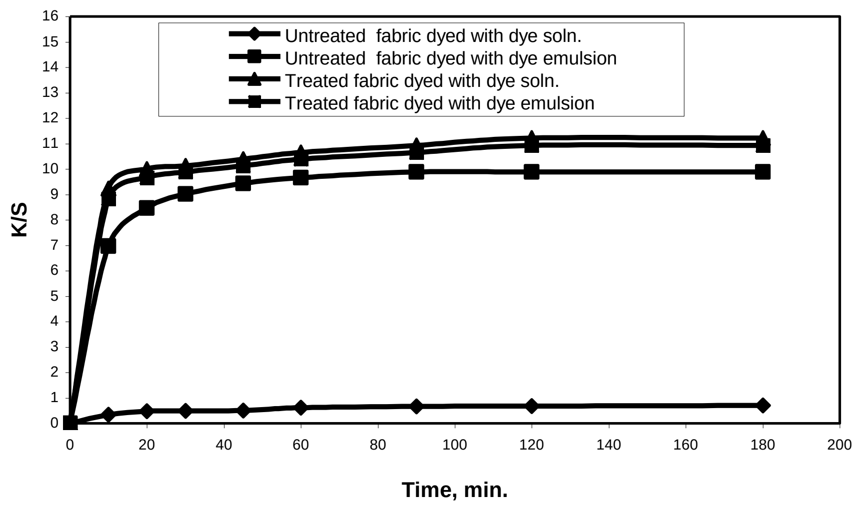
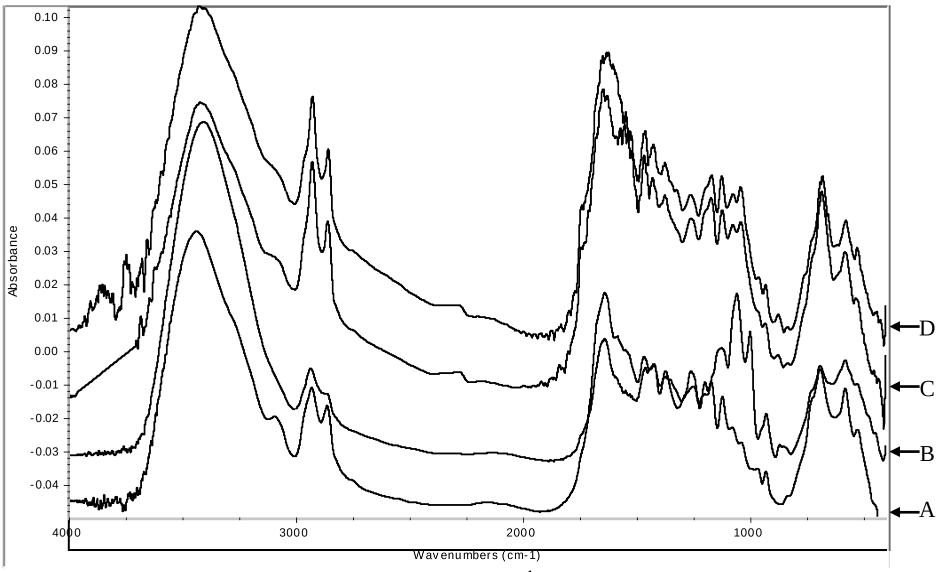


Figure 6 Effect of dyeing time of untreated and pretreated nylon 6 fabric on the colour strength of dyed nylon 6 fabrics. Pretreatment conditions : 10% owf tannic acid, L.R. 1:25 at 50°C for 60min. Dyeing conditions : 10^{-3}M C.I. Basic Violet 10, liquor ratio 1 : 25, pH = 4 at 85 °C.



wave numbers cm^{-1}

Figure 7 FTIR spectra of untreated and pretreated dyed nylon 6 fabrics. (A) – untreated nylon 6 fabric dyed with dye solution, (B) – untreated nylon 6 fabric dyed with dye emulsion, (C)- Treated nylon 6 fabric with 10% owf tannic acid dyed with dye solution and (D) – Treated nylon 6 fabric with 10% owf tannic acid dyed with dye emulsion. Dyeing conditions: (10^{-3} M C.I. Basic Viole 10, liquor ratio 1 : 25, pH = 4 at 85°C for 60 min.

Table 1 Physical properties of untreated and pretreated nylon 6 fabrics with tannic acid.

Amount of tannic acid %owf	Moisture regain (%)	Tensile strength (kg/cm^2)	Elongation (%)	Flexural rigidity		Bending length	
				Warp mg/cm	Weft mg/cm	Warp cm	Weft cm
Untreated(0)	3.8	0.45	137	20.00	67.5	1	3
5	4.2	0.50	139	28.75	97.03	1.1	3
10	4.7	0.57	155	60.60	190.40	1.4	4.1
15	4.8	0.62	162	68.90	271.30	1.3	4.6
20	4.4	0.71	94	65.10	238.40	1.35	4.4
30	4.2	0.86	73	75.80	310.30	1.50	4.8

Pretreatment conditions : 10% owf tannic acid, liquor ratio 1:25 at 50°C for 60min .

Table 2: Wash and rubbing fastness properties of untreated and treated nylon 6 fabrics.

Samples	Washing										Rubbing			
	Before using Uvitex® RSB					After using Uvitex® RSB					Before using Uvitex® RSB		After using Uvitex® RSB	
	Change	SW	SN	SA	SC	Change	SW	SN	SA	SC	Wet	Dry	Wet	Dry
Untreated nylon 6 fabric dyed with dye solution	3-4	2	4	4	3-4	4-5	4-5	4-5	4-5	4	4-5	5	4-5	5
Untreated nylon 6 fabric dyed with emulsion system	4	2-3	4-5	4-5	4-5	4-5	4-5	5	5	4-5	3	3-4	4	4-5
Treated nylon 6 fabric dyed with dye solution	3-4	2-3	3-4	4-5	3-4	4	3-4	4	4-5	3-4	3-4	4	4-5	4-5
Treated nylon 6 fabric dyed with emulsion system	4	3	4-5	4-5	4	4-5	4-5	5	5	4-5	3-4	4	4	4-5

Change : Alteration in shade, SW= staining on wool, SN=staining on Nylon, SA= Staining on acrylic, SC = staining on cotton.

Table 3 Perspiration fastness properties of untreated and treated nylon 6 fabrics

Samples	Perspiration before using Uvitex® RSB										Perspiration after using Uvitex® RSB									
	Acidic					Alkaline					Acidic					Alkaline				
	Alt	SW	SN	SA	SC	Alt	SW	SN	SA	SC	Alt	SW	SN	SA	SC	Alt	SW	SN	SA	SC
Untreated nylon 6 fabric dyed with dye solution	3-4	2-3	3	4	2-3	3-4	2-3	3	4	2-3	4-5	4	4-5	4-5	4	4-5	4	4	4-5	4
Untreated nylon 6 fabric dyed with emulsion system	4	3	4	4	3	4	2-3	3-4	4	3	4-5	4	4-5	4-5	4	4-5	3-4	4	4-5	3-4
Treated nylon 6 fabric dyed with dye solution	4	2-3	4	4	3	4	2-3	3-4	4	3	4-5	4	4-5	4-5	4	4-5	3-4	4	4-5	4
Treated nylon 6 fabric dyed with emulsion system	4	3	4	4-5	3-4	4	3	3-4	4-5	3-4	4-5	4	4-5	4-5	4	4-5	3-4	4-5	4-5	4

Alt: Alteration in shade, SW= staining on wool, SN=staining on Nylon, SA= Staining on acrylic, SC = staining on cotton. Fixing agent: 1% Uvitex RSB, 3g/l glauber salt , liquor ratio 1:20, pH 5,at 50ºC.

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