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Fading Characteristics of Chemically Modified Cotton Fabric Dyed with an Acid Dye

Ferial M. Tera¹*, Ashraf I. Hafez², Sayed H. Samaha¹, Angela A. Qutub³, and Ekhlas A. El-Alfy⁴

¹Material Testing and Chemical Surface Analysis Lab., National Institute of Standards (NIS), Egypt. ² Egyptian Electricity Holding Co. Chemistry Sector, Cairo, Egypt

³ King Abdul Aziz University, Faculty of Home Economics, Clothing & Textile Departments, Saudi Arabia ⁴Textile Research Division, National Research Centre, Dokki, Cairo, Egypt *Corresponding author's email: ferial.tera@nis.sci.eg

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Abstract

The study focused on enhancing cotton fabrics using modified starches dyed with an acid dye. The study will analyze and discuss various factors related to dye-ability, colorimetric data, color strength, fading and fastness characteristics. The study will include fastness grade, fading rate order curves, and fading rate constants. Since cotton is the most abundant raw material but has no affinity towards acid dyes. Lot of research and technological works were applied to increase its applications for the different end uses and upgrading its performance to become multifunctional, and accepting different classes of dyes. Samples were finished with (DEAE-O and / or-H) starches and dyed with a blue acid dye at different dye concentrations, then exposed to artificial daylight for different periods. The obtained results showed that treating cotton samples with the aminated starches rendered them accepting acid dyeing with considerable (k/s) and colorimetric values, good light fastness, and low fading rate curves showed class (2) fading order. The (k/s), the blue component (-b), the (L.F), and the (tf) 10% values were in direct proportion with the dye concentrations, while [K, Δ E] values showed the opposite trend. It was concluded that, the values of the blue component (-b) may be taken to represent the actual blue color of the applied dye on the fabric. Also, (K) could be taken as good representation to the light fastness compared to the recorded L.F. ratings values.

Keywords: Fading rate constant, CIE color parameters, Cotton, Chemical modification, Acid dyeing.

1 Introduction

Cotton fabrics are the most widely used cellulosic raw materials. They are greatly popular due to their excellent properties and characterization as biodegradable, moisture absorption, comfort, high breathability, good soft hand together with unique durability besides physicochemical and mechanical properties. Hence lot of research and technological work were applied to increase their applications for the different end uses and upgrade their performance to become multifunctional fabrics. Also, chemical modification of cellulosic materials was frequently carried out to create active centers that impart new and /or diverse properties which

increase the fabrics' applications for different end uses. Much research and technological work were done in this field to make the cellulosic fabrics acquire some additional properties such as flame retardant [1,4], antibacterial and UV protection [5-8], self-cleaning, [9-12] water /oil repellent, [13] crease and wrinkle [14-16] recovery. Also, to accept different classes of dyes [17-24] with remarkable improvement in dyeability and fastness properties. The most applied chemical modification techniques are graft copolymerization, [3,25-27] finishing treatment with different types of finishing agents [28-31], UV/ozone [32,33], microwave [34-36] and plasma. [37-39] Also, the application of nanotechnology has increased rapidly and has become a target of many researchers due to its unique and valuable properties that allow cellulosic materials to become multifunctional. [40-44] Exposure of dyed textiles to light causes serious damage as changes in molecular structure, decrease in mechanical properties, change in color strength, and light fastness and fading. It is well known that the L.F. of colored textiles depends on the quantum efficiency and the adsorption rate. many factors affect that affecting the photo fading of colored textiles as the character of the dye-fiber bond, the chemical structure of the dye and the substrate, the physical state of the dye, the diffusion restriction effect, the dye concentration, the chemical modification, the quality of radiation, the temperature and relative humidity [45,46,48]. The present work was carried out with a view of studying the fading characteristics of acid-dyed cotton fabric treated with DEAE-H-and O-St, as finishing agents to improve the dyeing performance, expressed as the color strength (k/s), the blue color component (-b). The fading characteristics of the modified dyed samples will be studied after exposure to the artificial daylight and discussed together with the light fastness L.F., the time required for 10% fading [tf]10% and also, with the fading rate constant (K) and the overall color change $[\Delta E]$ values.

2 Research Methodology

2.1. Materials

Mill-scoured and bleached plain weave cotton fabric [poplin], Kafr El-Dawar, Alexandria was used. Pure Egyptian corn starch was kindly supplied by Egyptian Starch and Glucose Manufacturing Company, Cairo, Egypt. 2-chloroethyl dimethyl amine hydrochloride was supplied by Fluka. Nonionic wetting agents as well as other used chemicals were of reagent grads. Blue acid dye namely Alphan Fast Blue HFL (C.I. Acid Blue 213) Hoechst Co. was used.

2.2. Acid hydrolysis of native starch

Hydrolyzed corn starch having 0.84 m. eq. /100 gm sample was prepared according to the method mentioned elsewhere [49].

2.3. Oxidation of native starch

Oxidized corn starch having analyzed carboxyl content of 18.4 m. eq. /100g sample was obtained as the method [50] described elsewhere.

2.4. Cationization of modified starch

The cationization procedure for both hydrolyzed and oxidized forms were prepared based on the method described elsewhere [51]. The obtained N% were evaluated according to the Kjeldahl method [51] and their recorded values were $1.53 \pm 0.02 \& 1.49 \pm 0.02$ respectively.

2.5. Fabric treatment

Cotton fabric samples were treated with (6%) solution of the prepared cationized starch in both oxidized and hydrolyzed forms using the pad - dry-cure method at a temperature of 160 0C for 3 minutes. The cured samples were then thoroughly washed, soaped, rinsed, and finally dried at room temperature

2.6. Dyeing procedure

The untreated and the previously treated cotton fabric samples with the aminated oxidized and hydrolyzed starch were independently dyed with the acid dye at six different dye bath concentrations using the exhaustion method at a temperature of 90 OC for one hour at a liquor ratio of (1:50) and pH of 4. The dyed samples were rinsed, soaped, and thoroughly washed, then finally dried at ambient conditions.

2.7. Light exposure

All the untreated and treated cotton fabric samples were exposed to the artificial daylight using Tera Light fastness tester [52] for different time intervals ranging from 20 - 180 hours at a temperature of 25 ± 2 ^OC and relative humidity of 60 ± 5 . A Blue Scale was hanged alongside the samples [53].

2.8. Characterization

2.8.1. Fourier Transform Infrared Measurements

FTIR spectrum for the native and aminated modified samples were recorded using a Nicolet 380 spectrophotometer Thermo Scientific USA, at the wave-number range of (4000-400 cm⁻¹).

2.8.2. Zeta potential

Zeta potential is the measurement of the average charge of the colloid solution and represented in millivolts. The potential charges of native corn starch and also the extent of cationization of the hydrolyzed and oxidized starched solutions were measured by using Zetasizer Nano-ZS [Malvern, UK] [61].

2.9. Testing and analysis

2.9.1. Spectrophotometric measurements

The reflection spectra for all examined samples before and after exposure to the artificial daylight were recorded in the visible range [400 - 700 nm] using Optimacth Color –Eye 3100

spectrophotometer SDL, England

2.9.2. Color evaluation

The ability to recognize color of any colored textile samples is developed from their spectral reflection curves. Thus, the color strength (k/s), color components (L*, a* and b*) and the color difference [ΔE^*] were evaluated by applying the Kubelka Munk equation [54, 55,] and the CIE system [56]. The color difference (ΔE^*) values were evaluated using the color component values of the exposed and the unexposed dyed modified samples.

2.9.3. Light fastness assessment

The light fastness rating values of the examined blue acid dye on the dyed modified samples were evaluated visually assessed against the standard Blue Scale patterns. [57]

2.10 Statistical analysis

All results listed in this research are the average of three measurements.

3 Results and Discussions

3.1. FTIR

The FTIR spectra of the native starch, DEAE- O - St and DEAE - H -St are shown in Fig. 1. It is evident that for all studied samples in question, the presence of characteristic peak bands at the range of $3500-3300 \text{ cm}^{-1}$, and $1240-1080 \text{ cm}^{-1}$, representing O–H stretching and C–O–C stretching respectively, assuring the existing of D-glucose units [60]. On the other hand, the presence of characteristic peaks at 1025 cm^{-1} and 2820 cm^{-1} are due to the tertiary amine as well as CH₂-N groups of the modified starch [60].

3.2. Zeta potential

The zeta potential values of native corn starch as well as DEAE-O & H-starches forms are represented in Fig. 2. It is evident that native corn starch showed slightly negative charge equal, -0.504 mV, whereas both of DEAE-H & O -starches exhibited high positive charges of the order 19.2 &13.4 mV respectively. Although both cationic starch derivatives [hydrolysed and oxidized forms] were carefully prepared with approximately equal nitrogen content values $[1.53 \pm 0.02 \& 1.49 \pm 0.02]$, there is a remarked higher zeta potential value for the DEAE-H-St than that of the DEAE-O-St. Most probably the presence of anionic carboxyl groups, having negatively charged character, in the molecular structure of the oxidized form lead to the decrement of its zeta potential value. [61]



Figure 1: FTIR of native starch [a], DEAE- H – St [b]; DEAE - O -St [c].



Figure 2: Zeta potential of native starch, DEAE-H-St. DEAE-O-St.

3.3. The dyeability, the color strength (k/s), and the blue color component (-b)

The obtained results of Table 1 represent the dyeability of the examined cotton samples finished with the aminated oxidized or hydrolyzed starch and dyed with the blue acid dye at six different dye bath concentrations. Considerable dyeability represented by the values of the color strength (k/s) of the order (0.16 - 2.15) and the blue color component (-b) of the order (21.89 -29.10) were obtained for the aminated dyed samples while untreated samples did not accept the acid dyeing. The improved dyeability and the observed blue coloration of the modified cotton samples may be attributed to the presence of the reactive tertiary aminated groups bonded to the cellulose macromolecules creating: self-active, built-in catalyst, basic leading to the increase of cellulose swell-ability through opening its structure that enhanced the dye adsorption. Accordingly, the acid dye molecules diffused, penetrated, accumulated, and aggregated in the intermicelles of the cellulose leading to better dye-ability, higher values of the color strength, and the blue color component (-b). These values were increased with the increment of the dye bath concentrations which may be attributed to the fact that the reaction of the dye with the fabric relies on he availability of the dye molecules in the vicinity of the fabric and this can be achieved by increasing the dye bath concentration. [20,21,45-48]. Also, the aminated hydrolyzed samples having active (CHO) groups showed higher dye-ability values than those of the oxidized samples having (COOH) groups which were attributed to the effect of the slightly higher nitrogen content of the cationic hydrolyzed starch samples, and also due to the presence of the acid dye that repulsing the similar acid groups (COOH) of the oxidized samples leading to minimizing their dye particles aggregation, and hence lowering their color strength and color component (-b) values.

Table 1: shows the values of: the dye bath concentrations, [k/s, the color strength]; [-b, the blue color component]; [L.F, the light fastness]; [tf10, the 10% fading time]; [k, the fading rate constant] and [ΔE , the overall color difference] of the treated cotton samples with hydrolyzed and/or oxidized starch and dyed with acid dye.

Dye	Blank	DEAE –Hydrolyzed -Starch dyed samples					DEAE –Oxidized-Starch dyed samples						
bath.	sample												
Conc.g/l	S	k/s	-b	L.F.]tf]1	Kx10	$\Delta \mathbf{E}$	k/s	-b	L.]tf]1	Kx10	$\Delta \mathbf{E}$
x102					0%	3				F	0%	3	
0.05	BDL*	0.29	24.05	3	32	2.5	7.8	0.16	21.89	3	30	4.2	8.14
0.10	BDL*	0.44	26.60	4	38	2.2	7.3	0.24	25.05	3	35	3.1	8.11
0.40	BDL*	1.01	27.50	4-5	48	2.0	6.5	0.70	26.50	3-4	42	2.6	7.15
1.25	BDL*	1.20	28.10	4-5	50	1.8	6.1	0.88	27.80	4	44	2.4	6.84
5.00	BDL*	1.75	28.90	5	60	0.9	5.3	1.40	28.40	4-5	56	1.4	5.99
10.00	BDL*	2.15	29.10	5	66	0.7	5.0	2.00	28.90	4-5	60	1.1	5.42

BDL= Below the detection limit.

For fastness properties 5 means the Highest fastness while 1 is the least fastness on the standard grey scale rating.

The obtained results showed also that, the blue color component (-b) values could represent the actual accurate color of the applied dye on the fabric better than the color strength (k/s) values which are obtained by application of Kubelka Munk [55] equation at only one wave length of the reflection spectrum, while the color components are evaluated from the three tristimulus values of the C.I.E [55,56] system which are evaluated applying the whole reflection [400-700]nm according to the equations :

$$X = \int_{400}^{700} S\lambda \cdot R\lambda \cdot x\lambda \, d\lambda \tag{1}$$
$$Y = \int_{400}^{700} S\lambda \, R\lambda \cdot y\lambda \, d\lambda \tag{2}$$

$$Z = \int_{400}^{700} S\lambda \ R\lambda \,.\, z\lambda \,d\lambda \tag{3}$$

$$\mathbf{b} = [[\mathbf{Y}/\mathbf{Y}_{\rm o}]^{1/3} - [\mathbf{Z}/\mathbf{Z}_{\rm o}]^{1/3}] \qquad (4)$$

where, 400-700 nm is the visible spectrum range

X, Y, Z are the tristimulus values

x λ , y λ , z λ are the stimulus values gf the equal energy spectrum

 $S\lambda$, is the energy distribution of the illumine falling on the color at a given wavelength

 $R\lambda$, $\,$ Is the reflectance of the color at a given wavelength

3.4. Light fastness [L.F] and the characteristic fastness grade curves (C.F.G.)

The L.F. characteristics deals with the examination of the state of the dye molecules in the fiber with rise of the dye concentration using physical measurements. The aggregation of the dye in a polymer substrate plays an important role in controlling the photo fading. Thus, the size and the form of the aggregates determine the extent of the exposed air-dye interface and this in turn determine the rate of degradation. Visual L.F. rating of the examined modified dyed cotton samples based on the Blue Scale were evaluated and given in Table 1. These results showed that the L.F. values were in direct proportion with the dye bath concentration, i.e. the dye particles are present as large aggregates [20,21, 45,46,48]. Nevertheless, the L.F. values of the modified hydrolyzed samples were slightly higher than those of the oxidized ones due to the reasons mentioned before that leading to minimizing their dye particles aggregation, and hence

lowering their color strength and color component (-b) values and in return their L.F. rating values.

When the L.F. values were plotted against their relative log (k/s) values, and also against (-b) values, the characteristic fastness grade curves were obtained. The examined dyed samples showed linear (C.F.G) curves with positive slopes and lay at moderate positions as represented in Fig. 3 [A, B.] Both the slopes and positions of the [C.F.G] curves depend upon the change in the size and the distribution of the dye particles inside the fabric with the rise of the dye bath concentration. The obtained positive slopes of these curves call for improvement of [L. F.] with the rise in dye bath concentration as a result of the increment of proportions of the dye large particles with reduction of the surface /weight ratio [20, 21, 45,46,48]. Also, the obtained (C.F.G) curves of the hydrolyzed modified samples were at slightly higher positions than those of the oxidized ones, and their slope values were 0.7770 and 0.7333, respectively when k/s values were applied while 0.3257 and 0.2139 when (-b) values were used.

3.5. Fading kinetics

3.5.1. Fading rate order

The fading rate order curves were obtained when the color strength (k/s) values at the different dye concentrations were plotted against the exposure time. Fig. 4 [A, B] shows such plots for the hydrolyzed and oxidized dyed samples. The obtained curves may be represented by class two [II] fading which is quite common representing an initial first or second-order fading followed by a zero-order. This means that only small proportions of the dye particles were accessible to light while the remains were either highly aggregated or firmly embedded in the micelles of the cellulose substrate. Also, the obtained curves showed that the fading of the oxidized dyed samples was slightly higher than those of the hydrolyzed ones i.e. they were more affected by the light exposure.

3.5.2. Fading rate curves

These curves were obtained by plotting the values of (k/s)t/(k/s)o representing the color strength values of the dyed exposed and the initial unexposed dyed samples against the exposure time (t) at the different dye bath concentrations [20, 45,46,48]. The obtained curves for both modified cotton samples were similar for the different examined dye bath concentrations and were represented in Fig. 5 (A-F). The obtained curves revealed that the fading rate for all the samples under investigation may be represented by class (1) fading [20, 45,46,48] i.e. first or second-order kinetic curves and that the treated oxidized samples were more affected by light and were more sensitive to photo- fading,



Figure 3: Characteristic Fastness Grad Curves: Light Fastness Against log k/s [A], Light Fastness Against -b [B] [▲: DEAE - H- St; ■ DEAE-O-St.].



Figure 4: Fading rate order curves for acid dyed fabric treated with: DEAE H-St [A]; DEAE-O-St at different dye bath concentrations [g/Lx102].

3.5.3. Time for 10% fading loss [t_f]_{10%}

The time required for 10 % fading [46] was evaluated from the curves of the fading rate order of Fig. 4 and is given in Table 1. The (tf)10% values increased with the rise of the dye bath concentration and also with the increment of the color strength. The hydrolyzed samples recorded higher (tf)10% values than those of the oxidized ones at nearly comparable color strength values i.e. their particles were more aggregated and needed more time to fade.

3.5.4. Fading rate constant (K)

Theoretically the rate of light fading can be represented by the following Equation: [60]

-dc/dt = [k/c₀] _C where, k/c₀ = K
∴ -dc/dt =Kc c and c₀ represent the exposed and the initial concentrations respectively
∴ dc/c = -K dt t is the exposure time k is the rate constant of light fading K is the fading rate constant

By integration,

 $\text{Log c/c}_0 = - \text{ K t } / 2.303$ (5)

When the values of $\log c/c0$ are plotted against the exposure time [t] straight lines are obtained and K values are evaluated from the slopes of these lines.

Thus, when the values of log (k/s) t / (k/s) o were plotted against the exposure time [t], straight lines with positive slopes were obtained indicating that more aggregations of the dye particles with the rise of the dye concentrations had occurred. [21, 45,46,48,].and Fig. 6 (a-f) shows such plots for the different dye concentrations. The values of the fading rate constant K were evaluated from the slopes of these curves and are given in Table 1. It is evident that the values of K decreased with the increment of dye bath concentration and also with the increase of the light fastness values. Also, K values of the hydrolyzed samples were lower than their mates of the oxidized ones. The obtained K values are concentration dependent and gave finite values for each dye bath concentration. It is clear that they could represent the L.F. much more accurate than that of the Standard Blue Scale visual ratings which gave same L.F. values for some different dye bath concentrations.



Figure 5: Fading rate curves: Variation of (k/s)t/(k/s)o against the exposure time for treated cotton fabric with [▲] DEAE-O-St.; [■] DEAE-H-St. dyed at different acid dye bath concentrations [a-f].



Figure [6]: Variation of log (k/s)t/(k/s)o against exposure time for treated cotton fabric with $[\blacktriangle]$ DEAE-O-St.; $[\blacksquare]$ DEAE-H-St. dyed at different acid dye bath concentrations [a-f].

3.6. The color difference $[\Delta E]$

This value was evaluated according to the CIE [56] system applying the following Equation:

$$\Delta E = \sqrt{[\Delta a]^2 + [\Delta b]^2 + [\Delta L]^2} \qquad (6)$$

where:

 Δ (a, b & L) = the difference between the color components of the exposed and the unexposed samples.

These values were calculated from the recorded color components (L. a, b) and the obtained results are shown in Table 1. It is clear that ΔE values were decreased with the increment of each of: the color strength, the light fastness, the blue color component (-b), the time for 10% fading, but with the decrement of the fading rate constant values (K). Also, the values of the color change ΔE of the hydrolyzed samples were lower than those of the oxidized ones indicating that they resist photo fading parameters slightly more than the oxidized samples.

4 Conclusion

This study introduced a new application of the blue color component (-b) values to represent the actual and accurate true color strength of the dyed fabric better than the color strength (k/s) values. The study showed also that the values of the fading rate constant (K) could be taken as

a good representation of L.F. values better than the Blue Scale L.F. ratings. Moreover, the obtained results showed also, that pretreatment of cotton fabrics with (DEAE-H-St) or (DEAE-O-St) rendered the treated samples susceptible to acquiring significant dyeability towards the examined acid dye with considerable color strength (k/s) and color component (-b) values and good light fastness ratings. Also, stability to the artificial daylight exposure and resist fading with low fading rate constant values Nevertheless, the color difference values were found to decrease as the concentration was increased.

5 Declarations

5.1 Credit authorship contribution statement

Ferial Tera: Conceptualization, Acquisition, Data curation, Methodology, Writing – review, editing & Supervising the whole work. Sayed H. Samaha: Methodology, investigation, Writing original draft, Formal analysis. Ashraf Hafez: Methodology & Formal analysis. Ekhlass ElAlfy: Methodology, Writing, review & editing. Angela Angela Qutub: did the experimental work.

5.2 competing of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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