

## Dyeing of treated Giza 89 Cotton Fabrics with Direct Dyes

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### Abstract:

The levelness of modified and unmodified Egyptian cotton made from Giza 89 fabric dyed with CI Direct Blue 78 and CI Direct Yellow 106 in the presence and absence of two nonionic surfactants (Rokamin 11 and Igepal CO720) was evaluated calorimetrically. The cotton was modified by grafting with vinyl pyridine (with and without quaternisation with dodecyl bromide). On modified cotton (grafted and quaternised) a significant improvement in levelness was achieved for only a slight decrease in exhaustion.

The results obtained revealed that levelness of dyeing's of CI Direct Blue 78 and CI Direct Yellow 106 on unmodified cotton was slightly improved by the surfactants Rokamin 11 and Igepal CO720. On modified cotton (grafted and quaternised). When dyeing with CI Direct Blue 78, the changes in levelness was achieved for only a "slight decrease in exhaustion, on the other hand, either of the two surfactants, an improvement in levelness was also observed, and consequently both surfactants can be used as effective levelling agents, with Igepal CO720 giving better exhaustion results.

### Keywords:

- *Direct dyes,*
- *Egyptian cotton,*
- *Exhaustion technique,*
- *Surface active agents,*
- *Color components,*
- *K/S of color,*
- *fastness properties*

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### Introduction

Polyethoxylated surfactants, especially nonionic alkyl-phenol and weakly cationic alkylamine types, are often used in the dyeing of wool and cellulosic fibers [1-3]. Such nonionic reagents are capable of forming complexes with dyes, which result in a reduction in the rate at which dye is adsorbed onto the fiber [4-10]. In some cases, however, the complexes formed also caused a decrease in dye uptake, the extent of which can vary depending on the dyes and the surfactant concerned [2]. This means that the textile chemist has to make a careful selection of dyes and surfactants combine good exhaustion with satisfactory dyeing uniformity. The levelness of a dyeing is nowadays frequently evaluated through the use of CIELAB color coordinates [11, 12].

We have previously investigated several dyeing processes involving two direct dyes (CI Direct Yellow 106, CI Direct Blue 78) on cotton [13]. The fiber was grafted with vinyl pyridine and afterwards quaternised with alkyl bromides with the aim of increasing the dye exhaustion. In the present work these modified specimens, which displayed unsatisfactory levelness, were dyed with the, same dyes in a typical dyeing process in presence of two nonionic surfactants (surfactant : dye molar ratios of 1:1 and 1:2). Dye bath exhaustion and dyeing levelness were assessed using CIELAB color coordinates in comparison with results obtained using different observers.

The present investigation studied the levelness of modified and unmodified Egyptian cotton made from Giza 89 fabric dyed with CI Direct Blue 78 and CI Direct Yellow 106 in the presence and absence of two nonionic surfactants (Rokamin 11 and Igepal CO720) modified by grafting with vinyl pyridine (with and without quaternisation with dodecyl bromide).

### Experimental

#### Materials:

Bleached Egyptian cotton fabric made from long stable Giza 89 was purchased from El-Nasr for spinning and textile. The fabric specifications were as followed; Rawcotton of Giza 89Fabric is plain weave 1/1., weight of square meter: 103 gm. Number of threads per cm of warp: 28; weft: 24. Twist factor, warp 4, and weft 3.5.

#### Dyes:

Two commercial dyes Sirius Light Blue G (CI Direct Blue 78) and Sirius Light Yellow 3R-LL (CI Direct Yellow 106) were purified by successive precipitation from sodium acetate [14].The chemical structures for the two dyes were shown in figure 1.

Pure samples of the commercial nonionic surfactants Igepal CO720 (Aldrich) and Rokamin 11 (Shorbagg Co.) as shown in Figure 2 were used.

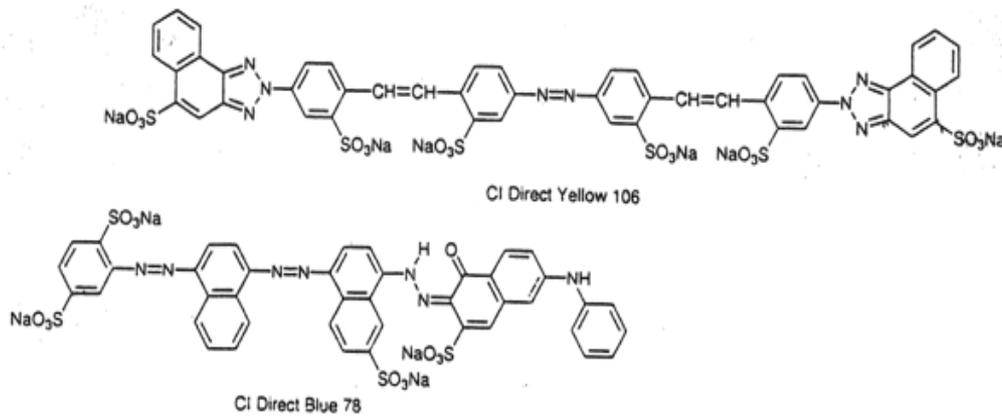


Figure 1. Chemical formula of CI Direct Blue 78 and CI Direct Yellow 106.

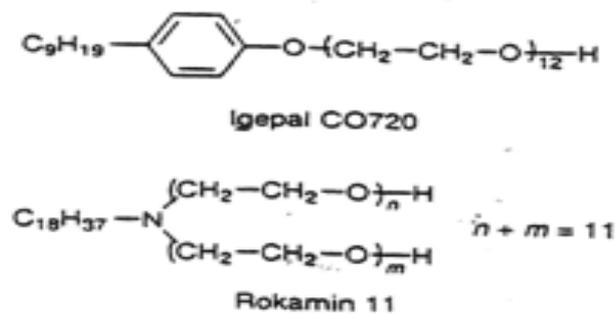


Figure 2. Chemical formula of nonionic surfactants Igepal CO720 and Rokamin 11

### Methods:

Dyeing was carried out using 1% dye, with 20% owf sodium chloride in Rotadyer machine, liquor ratio 50:1. A typical dyeing profile was adopted, beginning at 40 °C and raising the temperature to 98 °C over 30 min. and maintaining this temperature for a further 75 min. after which the dyeings were squeezed above the dyeing liquor, carefully washed with 20 ml distilled water, squeezed and finally washed thoroughly with tap water and dried.

The grafting and quaternisation process was carried out using the method described by Eleftheriadis et al., [13]. Pretreated specimen into an emulsion of 70 ml distilled water, 1ml 30% hydrogen peroxide, 1ml 2-vinylpyridine and five drops of nonionic emulsifier (Levegal KNS, Bayer), and maintaining it for 25 min with stirring from time to time in a water bath at 83-87°C. Then it was successively washed with methanol and water/methanol mixture, squeezed and extracted with methanol in a Soxhlet apparatus for 24 h to remove the homopolymer and monomer, and finally dried at 80 ± 2 °C and weighed. The percentage graft yield G was determined according to the following equation:

$$G = \frac{W_g - W_u}{W_u} \times 100 \quad [1]$$

Where  $W_g$  = dry weight of grafted cotton  $W_u$  = dry weight of unmodified cotton. Two further grafted samples (10.3 and 14.9% using 10ml and 12ml 2-vinylpyridine respectively) were obtained using the above procedure but with larger amounts of the emulsion components (300ml distilled water, 6ml 30% hydrogen peroxide, 30 drops emulsifier). Quaternisation of 4.6% grafted sample with alkyl bromides or epichlorohydrin was achieved by immersing a pretreated specimen into 50ml methanol containing 1.8 mol alkyl bromide or epichlorohydrin per mole of grafted vinylpyridine and refluxing for 24 h. The resultant sample was thoroughly washed with methanol/water and dried at 80°C to constant weight. The quaternisation yield was found to be approximately 0.2-0.3% on the basis of elemental analysis of specimens before and after quaternisation, since the weight difference between them were very low. The samples grafted to 10.3 and 14.9% were quaternised as above using, 1.8 mol I-bromodecane per mole of grafted vinylpyridine.

Two different molar ratios of surfactant to dye (1:1 and 2:1) were applied. The percentage dyebath exhaustion was indirectly determined by

measuring the absorbance ( $I_{\max}$ ) of the final dyebath using a Shimadzu UV-2101 PC spectrophotometer.

Test methods:

A Macbeth CE 3000 Color eye spectrophotometer (PC software Matchprobe 200, UV and specular component included, large area view 25.4 mm diameter) was used to measure the reflectance of the dyed fabrics, as well as dyeing levelness, expressed in terms of  $\Delta E$ . The percentage dye bath exhaustion  $E$  was calculated according to equation 2:

$$E = \frac{A_o - A_d}{A_o} \times 100 \quad (2)$$

Where  $A_o$  and  $A_d$  is the absorbances ( $I_{\max}$ ) of dye originally in the dye bath and of residual dye in the dye bath respectively.

The adsorbed dye on the fabric was calculated according to Kubelka - Munk function (equation 3):

$$K / S = \frac{(1 - R)^2}{2R} = ac \quad (3)$$

Where  $K$  = absorbance,  $S$  = scattering,  $R$  = reflectance,  $a$  = absorption coefficient and  $c$  = dye concentration [15,16]. The values obtained by this method, were identical to those obtained previously (Table 1).

The uniformity of the dyeing's were assessed visually by placing the samples in a Verified (Leslie Hubble Ltd) color assessment cabinet under an artificial daylight D<sub>65</sub> lamp. Samples for colour measurements were prepared by folding the dyed cotton fabric three times to give samples of about 3x3 cm.

The human eye can detect small differences in color, but it is difficult to quantify color differences accurately. Instrumental measurements can overcome this problem by using modern instruments that are able to measure tristimulus values accurately and reproducibly, and the  $\Delta E$  (equation 6) is used, the function of which is to provide a single number which is more precise and nearly equivalent to the grade of the visual difference between different colors [17].

$$\Delta E = \sqrt{[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]} \quad (4)$$

$\Delta L^* = L^* \text{ sample} - L^* \text{ standard}$ ,  $\Delta a^* = a^* \text{ sample} - a^* \text{ standard}$ ,  $\Delta b^* = b^* \text{ sample} - b^* \text{ standard}$

Where: standard refers to the dyed fabric; sample refers to the dyed sample after exposure to artificial day light. Color difference was evaluated by means of CIELAB 1994. Five points on a trial sample were measured. The first point was taken as the standard with which the other four points were compared to give the  $\Delta E$  values. If the maximum  $\Delta E$  was less than unit the dyeing was considered to be acceptable.

## Results And Discussions

Adsorption of CI Direct Yellow 106 and CI Direct Blue 78:

Table 1 shows the exhaustion % values for the samples dyed with both direct yellow 106, and blue 78 in the absence (control) as well as in the presence of both surfactants. It has been noted that the exhaustion of CI Direct Yellow 106 on the unmodified sample was approx. 23% lower than the corresponding values for CI Direct Blue 78. The grafted samples also showed a considerably (25-50%) higher exhaustion than did the unmodified samples, increasing with the dyeing temperature; generally recorded values were some 10-21 % lower than for CI Direct Blue 78 on grafted cotton. Again the uptake of CI Direct Yellow 106 by the variously quaternised specimens was virtually independent of whether a surfactant was used as the quaternising agent, except with isothermal dyeings at 40°C. This behavior could be attributed to the higher solubility and lower substantivity of the hexasulphonated yellow dye compared with the blue tetrasulphonate dye.

On the other hand, both dyes showed much higher exhaustion on the grafted and quaternised samples owing to the cationic character of the cotton, as discussed elsewhere [13]. However, it is notable that in the presence or the weakly cationic agent Rokamin 11 (2:1), both the unmodified and modified specimens showed a decrease in exhaustion compared with the control dyeing's in the absence of agent. This may have been attributable to mutual repulsion between the weakly cationic micelles containing some dye molecules and the cationic surface of the substrate. The results obtained revealed that quaternisation and dyeing were mainly carried out on the grafted specimens with the lowest vinylpyridine content, (4.6%), since these showed least yellowing, greatest uniformity and dye exhaustion values in the typical dyeing process.

Table 1. Exhaustion % of CI Direct Yellow 106 and CI Direct Blue 78 on Giza 89 cotton fabric-

Combination	CI Direct	Modification	Surfactant	molar ratios of surfactant to dye		
				0:1	1:1	2:1
Y1	Yellow 106	Control	Igepal CO720	33.8	32.2	35.3
Y2	Yellow 106	Control	Rokamin 11	33.7	24.2	21.2
Y3	Yellow 106	Modification A	Igepal CO720	88.2	95.2	96.7
Y4	Yellow 106	Modification A	Rokamin 11	87.0	92.5	83.2
Y5	Yellow 106	Modification B	Rokamin 11	97.5	99.2	88.2
B1	Blue 78	Control	Igepal Control CO720	62.3	56.2	58.5
B2	Blue 78	Control	Rokamin 11	62.3	61.2	53.3
B3	Blue 78	Modification A	Igepal CO720	97.5	99.56	99.3
B4	Blue 78	Modification A	Rokamin 11	97.6	95.8	82.3
B5	Blue 78	Modification B	Rokamin 11	99.3	98.5	93.5

Dyeing of 1% owf with 20% sodium chloride at 98°C

Modification A = cotton grafted with vinyl pyridine and dyed in presence of respective surfactants

Modification B = cotton grafted with vinyl pyridine, quaternised with dodecyl bromide and dyed in presence of Rokamin 11

Color coordinates of dyeing:

It has been noted that the highest the levelness the lowest  $\Delta E$  values, on other words, homogenous distribution of the dye on fabric renders the dye more tightly adhered to the fabrics, while non-homogenous distribution of the dye.

The levelness parameter values of the samples Y1 and Y2 were the higher compared with the other yellow dyed samples. On the other hand, the levelness parameter values of the samples B1, B2 and B3 were the higher compared with the other yellow dyed samples.

With the blue dye, the grafted samples, which displayed poorer Levelness, showed a great improvement when dyed in presence of Rokamin 11, compared with those dyed in the presence of Igepal CO720 (Table 2). With the yellow dye, both surfactants improved the levelness of grafted samples to about the same extent.

The Levelness of the unmodified cotton controls for both dyes was already acceptable (below unity) and the surfactants improved this slightly.

Table 2. Color coordinates of dyeing on various Giza 89 cotton fabric in a molar ratios of surfactant to dye (1:1), and grafting and vinylpyridine content, (4.6%)

Combination	L*	A*	B*	$\Delta E$	Levelness
Y1	74.3	18.7	67.2		
Y2	74.9	16.2	64.5	1.1878	Very small difference
Y3	75.5	18.5	67.9	0.6459	Normally invisible difference
Y4	61.8	14.3	54.2	7.012	Very obvious difference
Y5	62.2	14.8	49.8	7.3243	Very obvious difference
B1	60.5	14.2	53.2	7.6899	Very obvious difference
B2	61.4	11.2	50.2	7.8629	Very obvious difference
B3	61.1	13.1	51.3	7.6768	Very obvious difference
B4	62.5	18.6	57.3	6.4446	Very obvious difference
B5	61.2	21.2	57.9	7.3032	Very obvious difference

As mentioned in Table 1

Mean value among five points on the same sample.

Color differences ( $\Delta E$ ) by CIE 94 method.

The levelness values were according to the universal  $\Delta E$

The following delta E values are valid universally

- Color differences ( $\Delta E$ ) by CIE 94 method :

Meaning	Delta E value
A normally invisible difference	0 - 1
Very small difference, only obvious to a trained eye	1 - 2
Medium difference, also obvious to an untrained eye	2 - 3.5
An obvious difference	3.5 - 5
A very obvious difference	> 6

## CONCLUSIONS

The results obtained by studying the levelness with related to  $\Delta E$  using fabric dyed with CI Direct Blue 78 and CI Direct Yellow 106 in the presence and absence of two nonionic surfactants (Rokamin 11 and Igepal CO720) was evaluated calorimetrically. Factors affecting exhaustion of CI Direct Yellow 106 and CI Direct Blue 78 on Giza 89 cotton fabric was evaluated. The levelness parameter values of the samples *Y1* and *Y2* were the higher compared with the other yellow dyed samples. On the other hand, the levelness parameter values of the samples *B1*, *B2* and *B3* were the higher compared with the other yellow dyed samples.

The results obtained revealed that levelness of dyeing's of CI Direct Blue 78 and CI Direct Yellow 106 on unmodified cotton was slightly improved by the surfactants Rokamin 11 and Igepal CO720. On modified cotton (grafted and quaternised). When dyeing with CI Direct Blue 78, the changes in levelness was achieved for only a "slight decrease in exhaustion, on the other hand, either of the two surfactants, an improvement in levelness was also observed, and consequently both surfactants can be used as effective levelling agents, with Igepal CO720 giving better exhaustion results.

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