Application of Taguchi and Full Factorial Experimental Design to Model the Color Yield of Cotton Fabric Dyed with Six Selected Direct Dyes

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ABSTRACT

This paper describes the modeling of the color yield (F_k) of 100% cotton fabric dyed with six selected direct dyes (two from each groups of *A*, *B* and *C*) using Taguchi and factorial experimental designs as well as a response surface regression method. The factors chosen were dye concentration, electrolyte (sodium chloride) concentration, temperature and time of dying. To conduct the tests using the Taguchi approach, two levels were chosen for each factor. After obtaining the data (F_k), the significant factors were determined by an analysis of variance (ANOVA). Then, the level of significant factors was increased from two to three and the supplementary tests were carried out using full factorial design. ANOVA was applied again and, finally, the initial response surface regression model was produced considering the significant factors. After verifying the validity of the initial models, the BOX-COX transformation was implemented until the models achieved validity.

Keywords: Cotton fabric; Direct dyes; Color yield; Taguchi experimental design; Full factorial design; Box-Cox transformation; Response surface regression.

INTRODUCTION

Cotton, the most important natural fiber, is the purest form of cellulose found in nature. The content of cellulose in cotton is about 91% and increases to 95% after removing the natural impurities. The remaining 5% consists of other materials such as protein, pectin, ash and minerals [1]. The microstructure of crystalline cotton is defined as cellulose I, consisting of about 70% crystalline and 30% amorphous regions. The hydrophilic nature of cotton makes it possible to dye it with different classes of dyes [2].

Direct dyes, also called substantive dyes, can be applied to cotton fibers easily. These dyes are less expensive than others and are suitable for cellulosic fibers. However, their low washing fastness is a major drawback. Direct dyes are of an anionic type and dissolve in water. These dyes have an aromatic structure and contain chromophore and groups rendering them soluble. The chromophore of direct dyes can be divided into monoazo, diazo, triazo, polyazo, stilbene derivative, thiazole derivative and phethalocyanin derivative groups [3-5]. The Society of Dyers and Colorists *(SDC)* has also classified direct dyes, according to their leveling and migration behavior, into classes *A*, *B* and *C* [6]. The main parameters affecting the color yield, in the exhaust dyeing of cotton with direct dyes, are dye and electrolyte concentration, dyeing time and temperature, as well as the liquor ratio (L:R).

In many branches of the industry including textiles, process optimization, which has a considerable impact on cost minimization, has gained importance. To fulfill this task for the dyeing operation, employing more efficient machines, new dyeing techniques, as well as new products, play an important role. However, another technique that can help optimization is to find the optimum conditions of the dyeing bath which lead to a certain color yield. This requires a model representing the way that each factor, as well as the interaction between them, plays a part in determining the color yield (response). Sound and reliable modeling should be based on an appropriate experimental design.

The literature review revealed a number of efforts concerning modeling in dyeing. A summary of these works follows next. A mathematical model was proposed by Rys and Sperb and described the behavior of the fixation efficiency of mono-functional reactive dyes for various dyeing conditions [7]. Cegarra and Puente produced isoreactivity equations to determine the conditions of the temperature to achieve a constant sorption at different sorption times [8]. Huang and Yu used fuzzy models to provide a systematic approach to controlling the dye bath concentration, pH, and temperature in dyeing cotton cloth with direct dyes [9]. In another work, in order to improve the control of the process cycle for the application of reactive dyes in package dyeing, Shamey and Nobbs employed mathematical modeling [10]. In four papers, Tavanai, et al., reported on the modeling of the color yield in two-phase wet fixation-reactive dyeing of a cotton fabric (random experimental design) [11], the modeling of the color yield in polyethylene terephthalate dyeing through fuzzy regression [12], the modeling of the color yield in a two-phase pad steam-reactive dyeing of cotton cloth (binomial experimental design) [13] and finally the modeling of the color yield of six direct diazo dyes on a cotton fabric through central composite design [14].

THEORETICAL BACKGROUND

In almost all the fields of inquiry, experiments are carried out in order to discover some findings about the processes or systems. An experiment can be defined as a test or series of tests in which purposeful changes were made to the input factors of a process or a system, so that the reason for the changes were observed and identified. The design concept of the experiments has been in use since Fisher's work in agricultural experimentation. Fisher successfully designed experiments to determine the optimum treatments for the land to achieve a maximum yield [15].

The first step in designing any experiment is recognizing the problem. This is followed by the determination of the effective factors with their levels and specifying a response variable. Then, based on the objectives, one must select a suitable experimental design and carry out the experiments accordingly. The obtained data would be studied using the analysis of variance (ANOVA) method, leading to the determination of the factors with a significant effect on a response variable. Finally, a model can be worked out which represents the response variable as a function of the already determined significant factors. The choice of the experimental design depends on the type of problem, the number of factors, as well as their levels [16].

The Taguchi approach is one experimental design which has achieved a great deal of success. The overall aim of the Taguchi design is to find factor levels that maximize the *S/N* ratio. In statistical terms, "*S*" is called a "signal" and "*N*" is called a "noise". The higher the *S/N* ratio, the better the quality; in general, the *S/N* ratio could be considered in three modes where smaller is better, nominal is better or larger is better. The "larger is better" mode is found to be appropriate for this experimental design. *S/N* is shown in Eq. (1).

$$S/N = -10\log\left(\frac{\sum_{i=1}^{n} 1/y_i^2}{n}\right) \tag{1}$$

In Eq. (1), n is the number of repetitions for an experimental combination, i is a numerator, and y_i is the performance value of the ith experiment [17]. Generally speaking, the application of the Taguchi method leads to economy in cost and time by decreasing the number of experiments.

Contrary to the Taguchi approach, the full factorial design considers all possible combinations of a given set of factors. Since most of the industrial experiments usually involve a significant number of factors, a full factorial design results in a large number of experiments [18]. The response surface methodology, a collection of mathematical and statistical techniques, is useful for the modeling and analysis of problems in which a response of interest is influenced by several factors. If the response is modeled by a linear function of the independent factors, then the approximating function is the first-order model Eq. (2).

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \ldots + \beta_k x_k + \varepsilon \tag{2}$$

Where ε represents the noise or error observed in the response y. In this model, the regression coefficient, β_i, is a measure of the change in the response y due to a change in the input variable x_i. If there is curvature in the system, then a polynomial of a higher degree, such as a second-order model Eq. (3), must be used [18]:

$$y = \beta_0 + \sum_{i=1}^{k}\beta_i x_i + \sum_{i=1}^{k}\beta_{ii} x_i^2 + \sum\sum \beta_{ij} x_i x_j + \varepsilon \tag{3}$$

Transformations are often applied to the data to achieve certain objectives such as normalizing the data, stabilizing the variance, or eliminating the interaction effects. The most commonly used transformer is the power family given by Box-Cox as shown in Eq. (4):

$$y^{(\lambda)} = \begin{cases} \dfrac{y^{\lambda}-1}{\lambda} & \lambda \neq 0 \\ \log(y) & \lambda = 0 \end{cases} \tag{4}$$

Box-Cox proposes a maximum likelihood procedure to estimate the power (λ). This proposal is equivalent to minimizing $\sqrt{MSE}$ over the choices of λ. 10-12 points are usually chosen for λ. These points are in the range of -2 to +2. Then for each λ, a model is fitted to the data. The λ related to the model with the lowest MSE is chosen as the power used to modify the original model [18].

As literature review showed scarce information on the modeling of the color yield of the dyed cotton fabric through the Taguchi design, this research aimed at modeling the color yield (F_k) of 100% cotton fabric dyed with six selected direct dyes as a function of the dye concentration, the electrolyte concentration, the time and temperature of dyeing.

EXPERIMENTAL

Materials, Dyeing of the Samples and Color Yield Measurement

Samples of 100% bleached (no optical brightener) cotton fabric (128 g/m^2), weighing 2g each were dyed according to the Taguchi experimental design with two levels, namely level 1 (minimum value) and level 2 (maximum value) for each of the control factors. The control factors affect the response. The six diazo direct dyes chosen for this work were as follows:

- C. I. Direct Blue 67 and C. I. Direct Red 31 (Class A)
- C. I. Direct Blue 1 and C. I. Direct Red 224 (Class B)
- C. I. Direct Blue 2 and C. I. Direct Red 23 (Class C)

As previously mentioned, in the direct dyeing process, the main factors that affected the color yield were the dye and electrolyte concentration and the time and temperature of the dyeing. It is worth mentioning that the water hardness, in the levels of 1 for hard water (hardness =195 ppm) and 2 for soft water (hardness 40 ppm), was chosen as an uncontrollable factor. Based on the results obtained by Zavare et al. [14], we have assumed that the brainstorming sessions have led to the conclusion that for the red dyes, only the four main effects as well as the four interaction effects (dye concentration & time, dye concentration & electrolyte concentration, electrolyte concentration & time and temperature & time) were significant. This led to ten degrees of freedom requiring the L_{12} Taguchi scheme. Similarly, in the case of the blue dyes, we assumed that the brainstorming sessions led to the conclusion that, apart from the main effects, only two of the interactions, namely the dye and electrolyte concentration as well as the electrolyte concentration & temperature, have been significant. This led to seven degrees of freedom which required the Taguchi L_8 scheme.

Table I shows twelve runs for red dyes with the conditions of the variables stated as a coded factor value whose real amount is shown in *Table II*.

The samples were dyed in a Polymat (AHIBA1000) laboratory dyeing machine. Each dyeing (run) was carried out with hard water (Block 1) as well as soft water (Block 2). So a total of 24 dyed samples were prepared for each red dye. The dyeing was started at room temperature with the dye bath containing the required amount of dye. The dye bath temperature was raised to the final value in 20 minutes and then the electrolyte was added to the dye bath. The dyeing continued at the final temperature for the required amount of time (*Table I and II).* At the end of the dyeing, the samples were thoroughly rinsed in water (40 ˚C) and finally dried.

TABLE I. Taguchi experimental design table for the red dyes.

	Coded factor values			
Run No.	**X_1**	**X_2**	**X_3**	**X_4**
1	1	1	1	1
2	1	2	2	1
3	1	1	1	1
4	1	1	2	2
5	1	2	2	2
6	1	2	1	2
7	2	1	2	1
8	2	2	1	1
9	2	2	2	1
10	2	1	2	2
11	2	2	1	2
12	2	1	1	2

X_1 = Direct dye concentration (% on weight of fiber);
X_2 = Electrolyte (sodium chloride) concentration (% on weight of fiber);
X_3 = Dye bath temperature (˚C);
X_4 = Dyeing time (Min)

TABLE II. The amount of coded factor values in Taguchi experimental design stated in *Table I.*

Factors	**X_1**		**X_2**		**X_3**		**X_4**	
Levels **Direct dyes**	**Level 1**	**Level 2**	**Level 1**	**Level 2**	**Level 1**	**Level 2**	**Level 1**	**Level 2**
C.I. Direct Blue 67	0.5 %	2 %	15 %	35 %	60	70	30	60
C.I. Direct Red 31	0.5 %	2.5 %	15 %	35 %	40	60	30	50
C.I. Direct Blue 1	0.5 %	2.5 %	15 %	35 %	50	60	50	70
C.I. Direct Red 224	0.5 %	2.5 %	15 %	35 %	70	80	30	50
C.I. Direct Blue 2	0.5 %	2.5 %	15 %	35 %	50	60	30	50
C.I. Direct Red 23	0.5 %	2.5 %	15 %	35 %	70	80	50	70

The color yield of the samples (F_k) was measured by the Tex flash spectrophotometer (Datacolor), from which *K/S* (Kubelka-Munk theory) was calculated as shown in Eq. (5):

$$\left(K/S\right)_{\lambda} = \frac{\left(1-R_{\lambda}\right)^{2}}{2R_{\lambda}} \qquad (5)$$

R_λ was the minimum reflectance of light with a given wavelength (predominant wavelength) from a sample of infinite thickness, expressed in fractional form. The F_k function considered *K/S* in different wavelengths of the visible light as well as color matching functions Eq. (6).

$$F_{k} = \sum_{400}^{700} \left(K/S\right)_{\lambda} \left(\bar{x}_{10,\lambda} + \bar{y}_{10,\lambda} + \bar{z}_{10,\lambda}\right) \qquad (6)$$

Where $\bar{x}_{10,\lambda}, \bar{y}_{10,\lambda}\ and\ \bar{z}_{10,\lambda}$ were the color matching functions for the 10° standard observer at each wavelength measured (ISO 7724/1-1984) [19].

After identifying the significant factors in the Taguchi approach, the level of significant factors was increased from 2 to 3 and the supplementary experiments were carried out using the full factorial design. *Table III* and *Table IV* show the full factorial experimental design for C.I. Direct Red 23 and C.I. Direct Red 224 respectively. The factors and their levels in the full factorial design are shown in *Table V*. It is worth mentioning that the non-significant factors in the Taguchi method were kept constant at their lowest level in the full factorial design.

Moreover, it must be pointed out that all the runs listed in the full factorial tables were performed separately. The randomization of experiments was carried out by using Minitab software.

TABLE III. Full factorial experimental design table for C.I. Direct Red 23.

	Coded factor values		
Run no.	**X_1**	**X_2**	**X_3**
1	-1	-1	0
2	1	0	1
3	0	1	0
4	1	1	0
5	0	-1	1
6	0	0	-1
7	1	0	-1
8	1	-1	-1
9	-1	0	-1
10	-1	1	-1
11	0	0	0
12	0	1	1
13	1	1	-1
14	1	-1	0
15	-1	0	0
16	-1	1	1
17	0	-1	-1
18	-1	-1	-1
19	0	1	-1
20	1	-1	1
21	1	1	1
22	-1	1	0
23	-1	0	1
24	0	-1	0
25	-1	-1	1
26	0	0	1
27	1	0	0

TABLE IV. Full factorial experimental design table for C.I. Direct Red 224.

	Coded factor values	
Run no.	**X_1**	**X_2**
1	1	-1
2	-1	-1
3	1	0
4	-1	0
5	0	-1
6	1	1
7	0	0
8	0	1
9	-1	1

TABLE V. The amount of coded factor values in full factorial experimental design stated in *Table III* and *Table IV*.

Factors	**X_1**			**X_2**			**X_3**			**X_4**
Levels **Direct dyes**	**Level -1**	**Level 0**	**Level 1**	**Level -1**	**Level 0**	**Level 1**	**Level -1**	**Level 0**	**Level 1**	
C.I. Direct Blue 67	0.5 %	1.25 %	2 %	15 %	25 %	35 %	60			30
C.I. Direct Red 31	0.5 %	1.5 %	2.5 %	15 %			40	50	60	30
C.I. Direct Blue 1	0.5 %	1.5 %	2.5 %	15 %	25 %	35 %	50			50
C.I. Direct Red 224	0.5 %	1.5 %	2.5 %	15 %	25 %	35 %	70			30
C.I. Direct Blue 2	0.5 %	1.5 %	2.5 %	15 %	25 %	35 %	50			30
C.I. Direct Red 23	0.5 %	1.5 %	2.5 %	15 %	25 %	35 %	70	80	90	50

RESULTS AND DISCUSSION

Due to a lack of space, the complete methodology to obtain the final model for C.I. Direct Red 23 and C.I. Direct Red 224 are reported here. For the rest of the dyes, only the final models will be presented.

C.I. Direct Red 23

The data *(F_k)* obtained from the Taguchi design was analyzed by ANOVA and the significant factors at the 5% level (*Table VI*), namely, X_1 (dye concentration), X_2 (electrolyte concentration) and X_3 (dye bath temperature), were determined. In the next stage, the level of the three significant factors was increased to 3 and the supplementary tests were carried out using the full factorial design. Again, the obtained data was analyzed using ANOVA and the significant factors at the 5% level (*Table VII*), i.e., X_1, X_2 and X_3 were determined.

To produce a model, the response surface regression method was applied employing significant factors in the full factorial design with the help of a statistical software package (MINITAB 14). Eq. (7) shows the initial model.

$$F_k = 108.773 + 48.965 (X_1) + 7.039 (X_2) - 1.995 (X_3) - 11.453 (X_1X_1) \tag{7}$$

To verify the validity of the initial model, Box-Cox transformation was implemented.

Figure 1 shows the Box-Cox transformation for the initial model. As seen in *Figure 1*, the suggested value for λ is equal to 1. In other words, the Box-Cox does not propose a modification and the initial model accepted as stands. The validity of the initial model was also evaluated using residual graphs. *Figure 2* shows the normal plot of the residual and the residual versus fits for C.I. Direct Red 23. These results were obtained with the help of MINITAB software. As can be seen, the initial model enjoyed a good fit. *Table VIII* shows the R^2, R^2_{adj} and RMSE of the model. The plotting of the main effects for C.I. Direct Red 23 (for *S/N* with "larger is better" mode) are shown in *Figure 3*.

TABLE VI. ANOVA for C. I. Direct Red 23 while using Taguchi design for *S/N*.

Source	**Degree of freedom**	**Sum of squares**	**Mean Squares**	**F**	**P-Value**
X_1	1	252.035	192.501	4204.980	0.000
X_2	1	0.890	0.913	19.940	0.037
X_3	1	0.848	0.662	14.470	0.049
X_4	1	0.910	0.349	7.620	0.110
X_1X_4	1	0.060	0.040	0.870	0.449
X_1X_2	1	0.065	0.053	1.160	0.394
X_2X_4	1	0.084	0.094	2.050	0.289
X_3X_4	1	0.020	0.020	0.440	0.574
Error	2	0.092	0.046		
Total	10	255.004			

Signal to noise (*S/N*): Larger is better

TABLE VII. ANOVA for C.I. Direct Red 23 while using full factorial design.

Source	Degree of freedom	Sum of squares	Mean squares	F	P-Value
Block	1	2.000	2.000	0.070	0.792
X_1	2	87887.800	43943.900	1583.690	0.000
X_2	2	1784.300	892.200	32.150	0.000
X_3	2	236.500	118.200	4.260	0.025
X_1X_2	4	255.200	63.8	2.30	0.086
X_1X_3	4	67.200	16.8	0.610	0.662
X_2X_3	4	268.300	67.1	2.420	0.074
$X_1X_2X_3$	8	196.600	24.6	0.890	0.542
Error	26	721.400	27.7		
Total	53	91419.300			

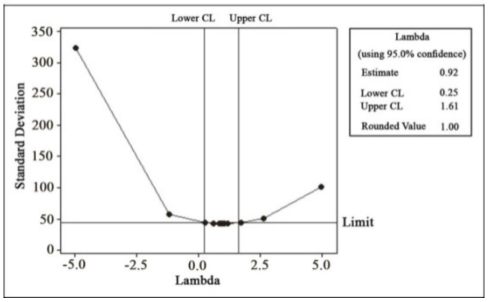
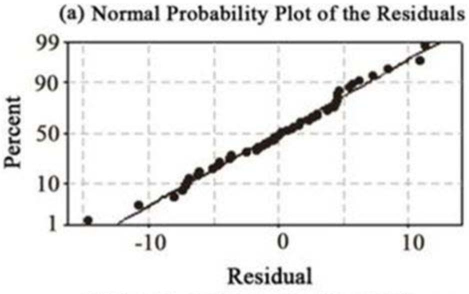


FIGURE 1. Box-Cox transformation for C.I. Direct Red 23.



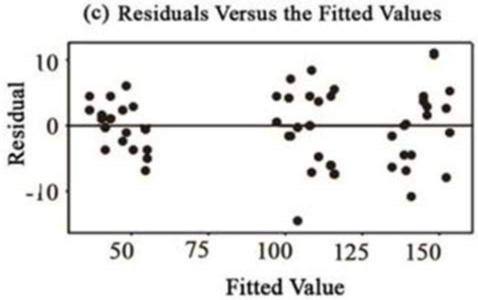


FIGURE 2. Normal plot of residual and residual versus fits for C.I. Direct Red 23.

TABLE VIII. Descriptive indices of the final model for C.I. Direct Red 23.

R^2	98.30 %
R^2_{adj}	98.10 %
MSE	32.1

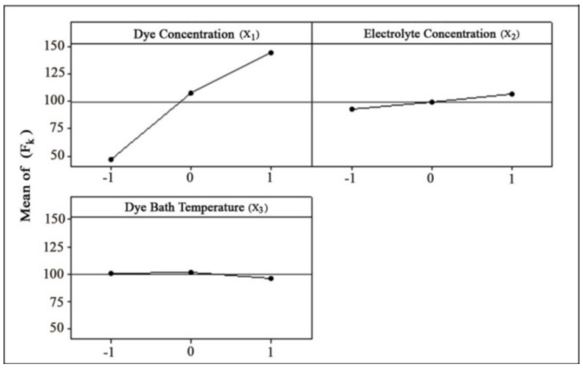


FIGURE 3. Main effects plot for C.I. Direct Red 23.

C.I. Direct Red 224

Similar to the C.I. Direct Red 23, the data *(F_k)* obtained from the Taguchi design was analyzed by ANOVA and significant factors at the 5% level were determined (*Table IX*). *Table IX* shows that X_1 (dye concentration) and X_2 (electrolyte concentration) were significant at a 5% level. The level of these significant factors was raised to 3 and a series of tests were carried out using the full factorial experimental design. *Table X* shows the result of ANOVA for the obtained results. As can be seen, in this case only X_1 was significant at the 5% level.

TABLE IX. ANOVA for C.I. Direct Red 224 while using Taguchi design for *S/N*.

Source	Degree of freedom	Sum of squares	Mean Squares	F	P-Value
X_1	1	120.626	85.190	59.160	0.016
X_2	1	27.739	27.965	19.420	0.048
X_3	1	5.441	0.591	0.410	0.587
X_4	1	12.010	10.188	7.080	0.117
X_1X_4	1	5.115	1.199	0.830	0.458
X_1X_2	1	8.557	8.129	5.650	0.141
X_2X_4	1	6.350	6.732	4.680	0.163
X_3X_4	1	0.530	0.530	0.370	0.606
Error	2	2.880	1.440		
Total	10	189.248			

Signal to noise (*S/N*): Larger is better

TABLE X. ANOVA for C.I. Direct Red 224 while using full factorial design.

Source	Degree of freedom	Sum of squares	Mean squares	F	P-Value
Block	1	3588.500	3588.500	23.850	0.001
X_1	2	6255.800	3127.900	20.790	0.001
X_2	2	983.000	491.500	3.270	0.092
X_1X_2	4	863.100	215.800	1.430	0.307
Error	8	1203.800	150.500		
Total	17	12894.100			

The initial model obtained for the C.I. Direct Red 224, using response surface regression method, is shown in Eq. (8).

$$F_k=58.420-14.119\,(B)+22.325\,(X_1) \qquad (8)$$

Figure 4 shows the Box-Cox transformation for the model Eq. (8) suggesting the value zero for λ. After applying the modification, the ANOVA shows that X_1, X_2 and X_1X_1 were significant at the 5% level. The final model is presented in Eq. (9).

$$(F_k) = 3.949-0.275\,(B)+0.476\,(X_1) + 0.121\,(X_2)-0.238\,(X_1X_1) \qquad (9)$$

B in Eq. (9) shows the effect of the Block (using soft or hard water) in the model. *Figure 5* shows the normal plot of the residual and the residual versus fits for C.I. Direct Red 224. As can be seen, the modified model is a good fit. *Table XI* shows the R^2, R^2_{adj} and the MSE of the initial and modified model. *Table XI* shows that the descriptive indicators have improved after the modification. The plotting of the main effect for C.I. Direct Red 224 (for *S/N* with "larger is better" mode) is shown in *Figure 6*. *Table XII* shows the final models obtained for all the six diazo dyes employed in this research. Finally, the models obtained in this research were compared with those obtained by Zavare, et al. [14], who employed Central Composite Design for the same direct dyes. A comparison of the descriptive indicators (R^2 and R^2_{adj}) shows that the models obtained through the Taguchi and full factorial designs, have improved relative to the central composite design.

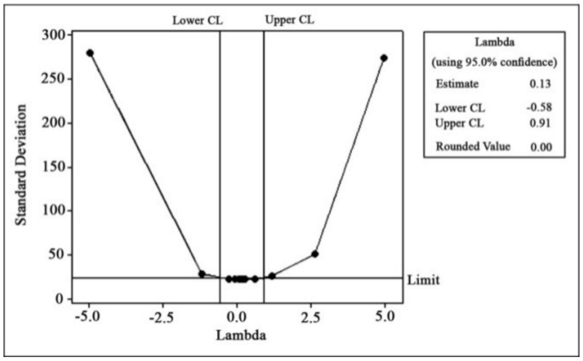
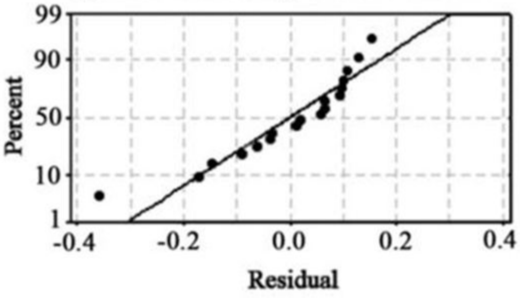


FIGURE 4. Box-Cox transformation for C.I. Direct Red 224.

(a) Normal Probability Plot of the Residuals



(c) Residuals Versus the Fitted Values

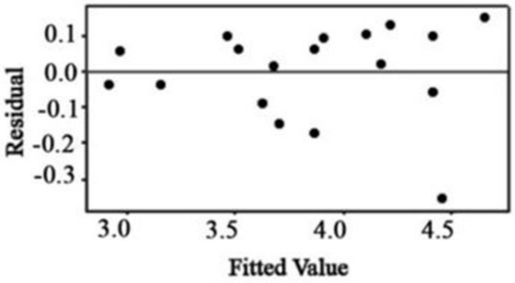


FIGURE 5. Normal plot of residual and residual versus fits for C.I. Direct Red 224.

TABLE XI. Descriptive indicators of the final model for C.I. Direct Red 224.

Descriptive indices	Initial Model	Modified Model
R^2	76.3 %	94.00 %
R^2_{adj}	71.3 %	91.50 %
MSE	217.85	0.024

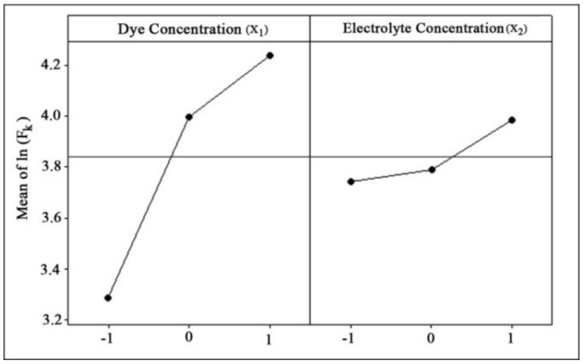


FIGURE 6. Main effects plot for C.I. Direct Red 224.

TABLE XII. The final models and related descriptive indices for the dyes.

Dye		R^2	R^2_{adj}	MSE	Final model
Class A	C.I Direct Blue 67	% 98.2	% 97.4	34.70	$F_k = 117.441 + 3.192 (B) + 41.251 (X_1) + 9.014 (X_2) - 11.701 (X_1X_1)$
	C.I Direct Red 31	% 98.0	% 97.2	0.005	$\ln (F_k) = 4.741 + 0.424 (X_1) + 0.226 (X_3) – 0.228 (X_1X_1) - 0.103 (X_3X_3) + 0.133 (X_1X_3)$
Class B	C.I Direct Blue 1	% 99.1	% 98.9	0.035	$(F_k)^{0.5} = 6.843 + 0.145 (B) + 2.040 (X_1) – 0.474 (X_1X_1)$
	C.I Direct Red 224	% 94.0	% 91.5	0.024	$\ln (F_k) = 3.949 – 0.275 (B) + 0.476 (X_1) + 0.121 (X_2) – 0.238 (X_1X_1)$
Class C	C.I Direct Blue 2	% 98.2	% 97.7	16.20	$F_k = 60.683 + 30.690 (X_1) + 5.630 (X_2) – 5.312 (X_1X_1)$
	C.I Direct Red 23	% 98.3	% 98.1	32.10	$F_k = 108.773 + 48.965 (X_1) + 7.039 (X_2) – 1.995 (X_3) – 11.453 (X_1X_1)$

CONCLUSION

The color yield of 100% cotton fabrics dyed with the six selected direct dyes can be modeled using the Taguchi and full factorial design, as well as the response surface regression method. The value of R^2 and R^2_{adj} of the obtained models show that the models fit all the cases. However, neither the models of the dye classes A, B and C show any similarity, nor the models of the dyes belonging to each of the classes of A, B and C. For the six direct dyes selected in this research, the electrolyte concentration and dyeing temperature are the most important factors on the color yield. The time of dyeing, in the range selected in this research, did not affect the color yield.

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